Development and rheological evaluation of hydrophilic cosmetic gel

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Abstract

Background: Among the topical systems to delivery cosmetic actives, the gel form is very desired, because it offers non-oily sensory and also allows the application of the formulation associated with techniques for skin rejuvenation, such as phonophoresis. The purpose of the study was to develop and evaluate rheological parameters of a hydrophilic cosmetic gel.

Methods: The rheological analysis consisted of steady-state flow measurements with shear rates ranging from 5 to 1000 s^{-1}, amplitude sweep test conducted at an angular frequency of 10 rad s^{-1} to obtain the linear viscoelastic region, and the frequency sweep performed in the range 0.1–100 rad s^{-1}. The three-interval thixotropy test (3ITT) was also performed.

Results: The sample was found to have a stable rheological state over the 16 weeks. She also exhibited "solid-like" (gel) behavior. Furthermore, it was characterized as a strong gel and shear-thinning pseudoplastic non-Newtonian fluid, with no thixotropy. The developed formulation presented physical stability, and therefore we suggest the formula to be used in cosmetic formulations.

Conclusion: The vehicle used can impact the stability and effectiveness of cosmetic products. The rheological study of cosmetic vehicles can predict the physical stability of formulations and help formulators in the development of more stable formulas.

Keywords: Rheology, gel formation, carbomer.

Introduction

Hydrophilic thickeners are raw materials of natural or synthetic origin, capable of swelling in the presence of water, giving viscosity to the medium in such a way that the resulting product can take the appearance of gel [1]. Hydrophilic thickeners can be of natural origin, of synthetic or semi-synthetic origin. They can be classified as gums, alginates, cellulose derivatives, clays, gelatin, synthetic resins, among many others that have been emerging in recent times [1]. Among synthetic resins, there are several types of carbomers (carboxyvinyl polymer), among which one can mention the carbomers 934, 940, 960, NF, among others [2].

Carbomers are made up of high molecular weight, cross-linked acrylic acid polymers. Carbomers can be dispersed in water to form low viscosity acidic colloidal solutions [1]. When dispersed in water, its thickening power is quite limited. One way to develop the complete viscosity potential of these polymers is by adding an organic or inorganic base, such as triethanolamine or sodium hydroxide, to the Aqueous Dispersion of the polymer [2]. This converts the acid groups of the polymer chain into its salt form, causing the chain to unroll and forming the extended structure that provides maximum efficiency as a thickener [2]. Aqueous gels should be added with humectants (such as propylene glycol, glycerin) because they tend to dry out over time, that is, evaporation of the water present in the gel can occur, causing drying of the product.

These materials can be applied, for example, in drug and cosmetic delivery systems [3]. In cosmetology, the gel cosmetic form is quite widely used. The gel form is presented as a stable suspension, being quite suitable for topical formulations. Gels are indicated to convey water-soluble active substances and liposomes. They are most often used on oily and Combination Skin [2].

Rheological analysis is one of the techniques used to study carbomers as to their mechanical and flow behavior [3]. This technique allows analyzing the flow of a fluid and the deformations that are exerted in it, enabling the determination of system stability parameters [3].

There are two main categories of rheological assays: rotational rheological assays and oscillatory rheological assays. While rotational tests study the flow behavior of the product, oscillatory tests evaluate the viscoelastic properties of the sample and energy conservation, being used in predictive stability studies [4]–[6].

In oscillatory rheological assays, the shear stress varies like a sine wave. The relationship between the applied stress waves and the resulting deformation provide important data on the structure of the formulations through the values referring to loss or conservation of energy by the system, given by the values in elastic modulus (G') and values in viscous modulus (G") [7].

The G' modulus response values, also known as the storage modulus, represent the energy stored during deformation to increasing stress, being released when the stress is relaxed. As its name says, this module represents recoverable deformations and is associated with system stability [4], [7].

The response values of Modulus G", or loss, represent the applied stress that is dissipated as irreversible deformation, this modulus indicates the liquid character, and therefore unstable, of the material [4], [7].

A parameter widely used in rheological analysis of fluids is viscosity (CF). Viscosity is the resistance of a fluid to flow [8]. According to the viscosity behavior, a material (or fluid) can be classified as Newtonian and non-Newtonian. The Newtonian fluid has a constant viscosity as a function of the shear rate applied. Dilating fluids are characterized by an increase in viscosity with an increase in shear rate. This behavior is observed in some suspensions. Finally, pseudoplastic fluids are characterized by a decrease in viscosity as a function of shear rate. This is the typical behavior of polymeric solutions, associated with their intertwined network of molecules [8].

The objective of this study was to develop and evaluate the Rheology of a hydrophilic gel.

Materials and Methods

The flow properties, viscoelastic behavior and thixotropy of the formulations were evaluated as stability parameters of the formulation over 16 weeks at room temperature.

Rheological measurements were carried out on the Anton Paar MCR-102 modular Compact rheometer with plate cone geometry (50 mm diameter, cone angle of 0.9815 and truncation of 0.97 RMB) at a temperature of 32.6 RMB C.

Steady-state flow measurements were obtained, with shear rates ranging from 5 to 1000 s^{-1}. The Ostwald-De-Waele model (Eq) was used. 1) to adjust the viscosity curves of the semi-solid formulation, which are given by Equation 1.

$$\eta(\dot{\gamma}) = m\dot{\gamma}^{\,n-1} \qquad \text{(Eq. 1)}$$

where $\dot{\gamma}$ is the shear rate m is the consistency index associated with the viscosity magnitude and n is a dimensionless parameter indicating the degree of dependence of viscosity on the shear rate, where n = 1 indicates Newtonian behavior, less than one (n<1) indicates shear-thinning behavior and more than one (n>1) indicates shear-thickenning behavior.

Small amplitude oscillatory test (Osas) was performed to analyze viscoelastic properties. The amplitude scan test was conducted at an angular frequency of 10 rad s^{-1} to obtain the linear viscoelastic region (LVE = 1% for all samples). Then frequency scanning test was performed in the range of 0.1-100 rad s^{-1}.

The three-interval thixotropy test (3ITT). In this, three shear rates are used to analyze viscosity recovery under shear. In the first, a low shear rate of 1 s^{-1} it is applied for 25 s, followed by a sudden increase to 100 s^{-1} for 50s and back to 1s^{-1} for 250 s [9]. Rheological measurements were performed in triplicate at 0, 8 and 16 weeks.

Results and discussion

Steady-stade shear viscosity

In order to characterize the rheological behavior of the samples, rheological tests were carried out at skin temperature (32.6 CF.c). The viscosity in a permanent regime as a function of the shear rate was obtained for the base and the formulation, as shown in Figure 3. It has been observed that the apparent viscosity decreases rapidly with increasing shear rate of 10 s^{-1} to 1000 s^{-1}, suggesting that the shear-thinning sample pseudoplastic non-Newtonian fluid. At the beginning of the test, when the net shear rate is high, the polymer chains are entangled. As the shear rate increases, the chains will undo and follow the flow, which may justify the decrease in apparent viscosity, since it depends on the chain structure.

It was also observed that the apparent viscosity value does not differ with respect to time (p=0.1167), indicating a stable rheological state over 16 weeks.

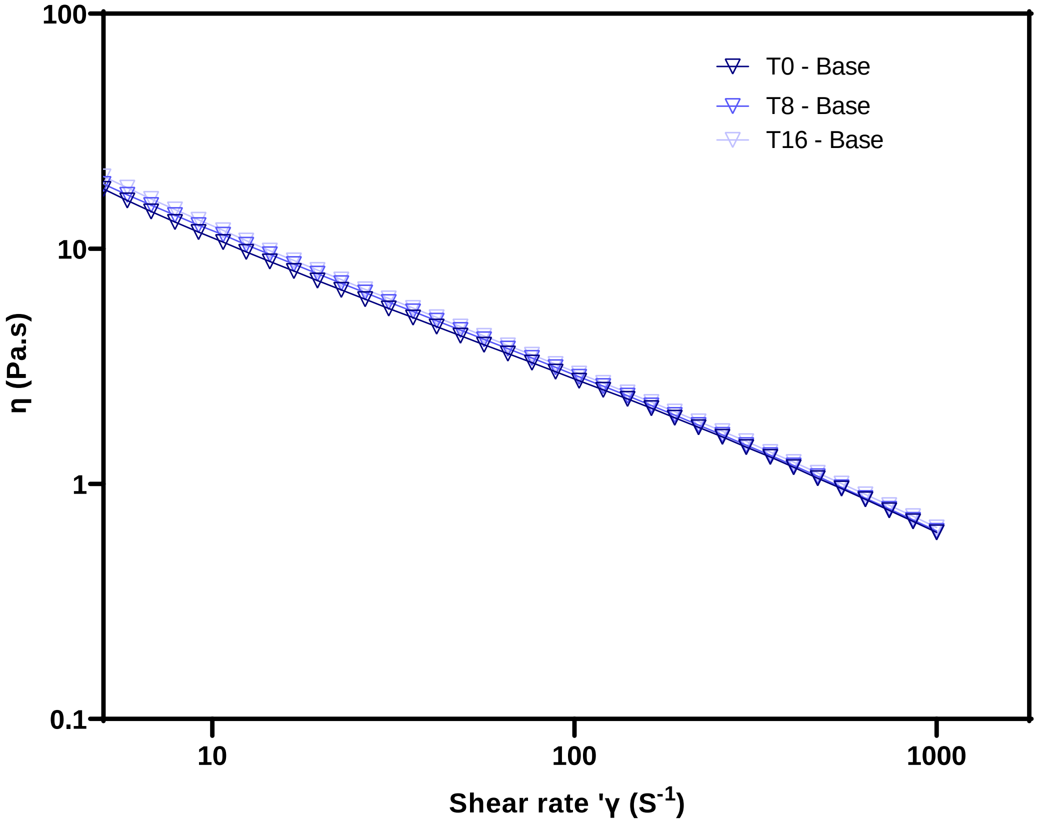

Figure 3. Steady shear deformation test.

Oscillatory deformation (frequency sweep)

To determine the linear viscoelastic behavior of the formed gel, a 1% frequency scan was performed (within the LVR) and the results obtained are shown in Figure 5. In the entire frequency range, the storage module, G', showed values much higher than the loss module, G", showing “solid-like” (gel)behavior [10]. There is no crossing of G’ and G’, so there is no transition region of behavior. This trend was maintained even after 16 weeks of storage, which may be related to sample stability.

In addition, the behavior of the sample is almost independent of the angular frequency, indicating that it is a strong gel [11].

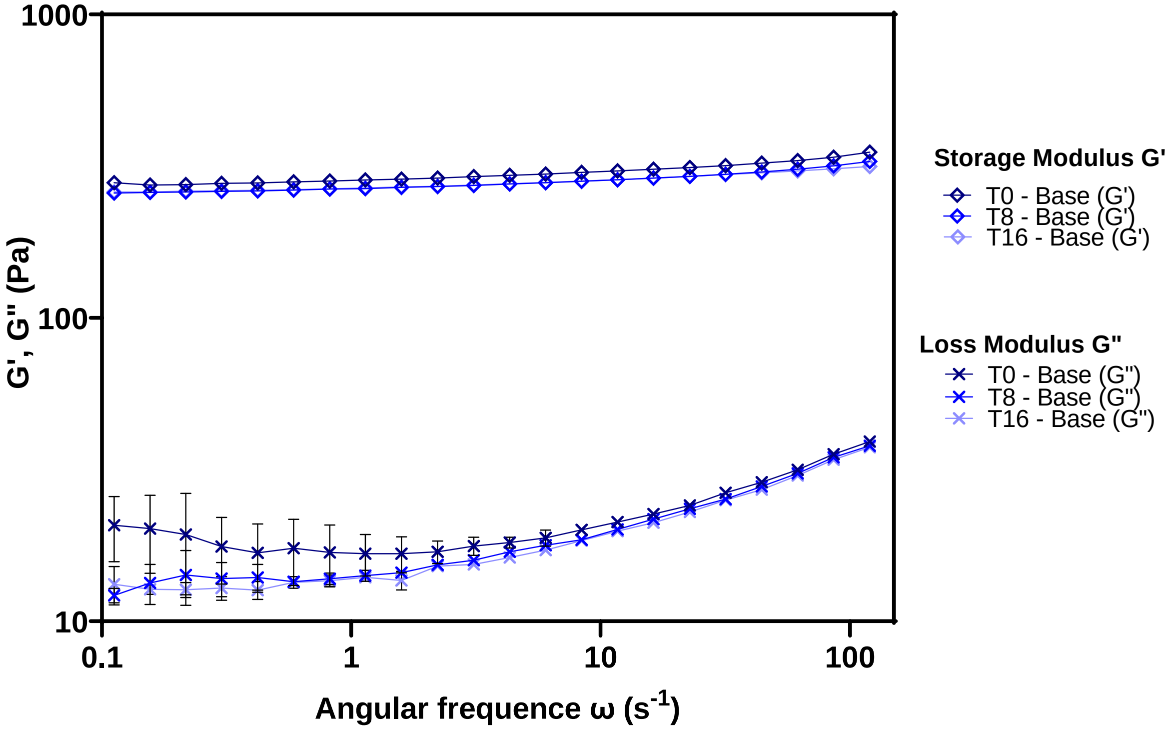

Figure 5. Frequence sweep test.

Thixotropic behavior

The 3-interval thixotropy test (3itt) describes viscosity recovery when a stress is applied and then removed from the material. This parameter is important for hydrogel formulations for topical use [12]. The test simulates the application of the product to the skin, since a shear rate is applied with the fingers when spreading the product.

Fig. 2a shows viscosity changes over time as a function of shear rate. As noted in Fig. 2a and 2b, the gel base does not depend on the time for viscosity recovery (short recovery time) and a recovery greater than 90%. This short reconstruction time and the percentage of viscosity recovery indicates that the sample is not thixotropic. The values of$_{rec}$ from the base in relation to time does not differ from each other.

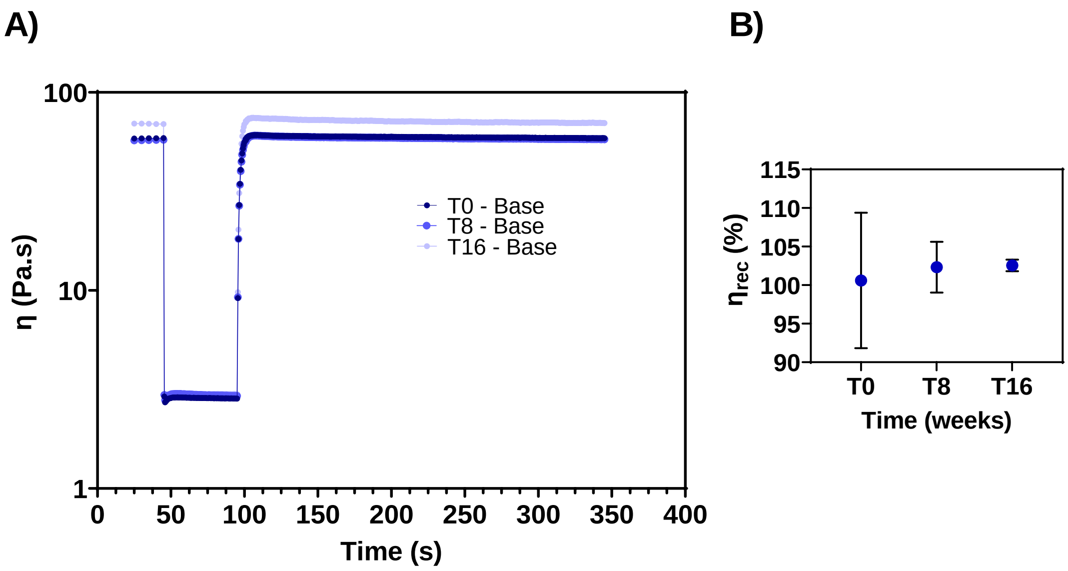

Figure 2. A) viscosity curves as a function of time for three interval thixotropy test (3itt) for formulation; B) percentage of viscosity recovery as a function of time, showing structural regeneration after 3itt.

Conclusion

The vehicle used can impact the stability and effectiveness of cosmetic products. Rheological study can predict the physical stability of formulations and help formulators in the development of more stable formulas, therefore more effective and with better acceptance by consumers. Gel consisting of carbomer, glycerin, phenoxyethanol and Ethylhexylglycerin, disodic EDTA, sodium hydroxide and water presented a stable rheological state over 16 weeks, in addition to showing "solid-like" behavior (gel). In addition, it was characterized as a strong gel and shear-thinning pseudoplastic non-Newtonian fluid, as well as absence of thixotropy. It is concluded that the gel developed is a promising formulation for future topical administration of cosmetic assets.

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Conflict of Interest Statement

None.

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