Investigation of lipstick properties using statistical methods

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Abstract: *The rheological non-destructive oscillation testing is conducted in order to predict and quantify the samples' properties of the commercial brand lipstick. The influence of the variation of the process parameters temperature, phase angle and strain are investigated. Regression models for the dependencies of the rheological and thermal properties (elastic modulus and shear stress) from the described process parameters are estimated, based on 681 experimental data in the temperature region from 26.9 °C to 74.7 °C.*

KEYWORDS*: LIPSTICK, OSCILLCTORY SHEAR RHEOLOGY, ELASTIC MODULUS, SHEAR STRESS, REGRESSION MODELS*

1. Introduction

The aspiration to look physically attractive is a universal desire. All kinds of cosmetics including lipstick, although originally developed by the ancient Egyptians as ointments and ritual oils for the dead, eventually end up being used to soothe, adorn, accentuate and treat the skin of the modern, living humans. In recent time statistics show that these products are used by over 90 % of the female population. The face is the part of the human body that receives the most attention compared to the rest of it. Lipsticks play a significant role as a part of the cosmetic products created for facial use, regardless of gender and/or age. The common understanding that lipsticks or lip balms are created only for the female population is false [1 - 4].

All the lipsticks appearing on the market must meet specific standards, connected with their physical-mechanical properties. Some of them are: stability against temperature fluctuations (55 °C - 75 °C) and humidity; long shelf life; dermatological safety; pleasant taste and smell; softening at physiological lip temperature around 32 °C; good durability and no defects such as air bubbles; no cracks or sweating, should be free of physical changes, etc. [3, 4].

The viscosity of the lipstick should be high enough to keep its structure in the matrix. In addition, it should gradually decrease as the mechanical stress increases during physiological application [3, 4]. From a tribological point of view, lipsticks require varying degrees of friction to glide effortlessly on the skin [3, 5].

In this paper the rheological non-destructive oscillation testing was conducted in order to predict and quantify the properties (threshold flow stress, elastic modulus) of the lipstick samples. Regression models, describing the dependencies of the lipsticks characteristics: y_1 - elastic modulus [Pa] and y_2 - shear stress [Pa] on the variation of the process parameters: temperature, phase angle and strain are estimated, based on 681 experimental data in the temperature region from 26.9 °C to 74.7 °C.

2. Response surface methodology

Response surface methodology (RSM) is a group of mathematical and statistical procedures used in fitting an empirical model to the experimental data obtained in relation to an experimental design [6]. Regression models (polynomial models of some order), giving an adequate functional relationship between a response of interest y (performance characteristic) and a number of associated control (or input) variables (process parameters) x_1, x_2, … x_m, are estimated:

$$\hat{y}(\vec{x}) = \sum_{i=1}^{k} \hat{\theta}_i f_i(\vec{x}) \quad (1)$$

where $\hat{\theta}_i$ are the estimates of the coefficients in the regression model.

The natural values of the factors (z_i) in the regression models are coded in the region [-1 ÷ 1] and the relation between the coded (x_i) and the natural values (z_i) is given by:

$$x_i = (2z_i - z_{i,max} - z_{i,min})/(z_{i,max} - z_{i,min}), \quad (2)$$

where $z_{i,min}$ and $z_{i,max}$ are the corresponding values of the minimum and the maximum of the process parameters during the experiment.

3. Experimental conditions

The rheological method - oscillatory shear rheology to characterize the rheological behavior of the studied brand of lipstick was applied.

There are three different types of stresses (tension, pressure and shear) that can occur when a material is exposed to a load. If the material is stretched or compressed in one dimension, this leads to tension or pressure, respectively. However, if opposite forces are applied to opposite surfaces of the material, shear stresses are obtained. The modules that result from these stresses can provide information about the properties of the material.

Shear stresses can be obtained using rheological techniques which provide information on the elastic behavior of a material. Shear stress can be calculated when dividing the force required to cause deformation by the cross-sectional area of the material. The strain is the distance moved divided by the original length of the material.

For elastic solids, the energy generated (modulus of elasticity) during deformation of the material is stored, as a result of which the material returns to its original shape (is recovered) after removing the force causing deformation (Hooke's law) - an important feature for lipsticks.

The modulus of elasticity is a measure of the strain energy stored in the sample during shear, thus showing the elastic (solid-like) behavior.

An experiment was performed with samples of commercial brand lipstick to test its rheological and thermal properties like threshold flow stress, elastic modulus, etc. Minimum quantity (7 - 10 mg) of the lipstick is scratched on the surface of a closed aluminum capsule (diameter: 6 mm). The sample was heated at a constant rate of 5 °C for 1 min. The experiments were conducted during the cooling of the lipstick at an initial starting temperature 74.7 °C and the measurements were stopped when reaching 26.9 °C. As a result of the experiment 681 measurements were done.

The rheological behavior of the studied brand of lipstick characterized by the following performance parameters: y_1 [Pa] - elastic modulus and y_2 [Pa] - shear stress. The variation regions [z_{min} - z_{max}] of the process parameters were: for temperature (z_1) – [26.9 – 74.7 °C]; the phase angle (z_2) – [0.0024 – 90 deg] and the strain (z_3) – [1.37×10^{-5} – 1.483×10^{-2}]. The angular frequency for these experiments was constant and is 0.2 Hz.

4. Regression models estimation

The dependencies of the lipstick characteristics: y_1 – elastic modulus [Pa] and y_2 – shear stress [Pa] on the variation of the process parameters: z_1 – temperature [°C], z_2 – phase angle [deg] and z_3 – strain are estimated.

They are presented for coded in the region [-1 ÷ 1] values of the process parameters in Table 1, together with the square of the multiple correlation coefficient R^2 and the square of the adjusted multiple correlation coefficient R^2_{adj}. The values of both coefficients are high (close to 1 or 100 %) and consequently the estimated models for the elastic modulus $\hat{y}_1(\vec{x})$ and shear stress $\hat{y}_2(\vec{x})$ can be considered as good enough for prediction and investigation.

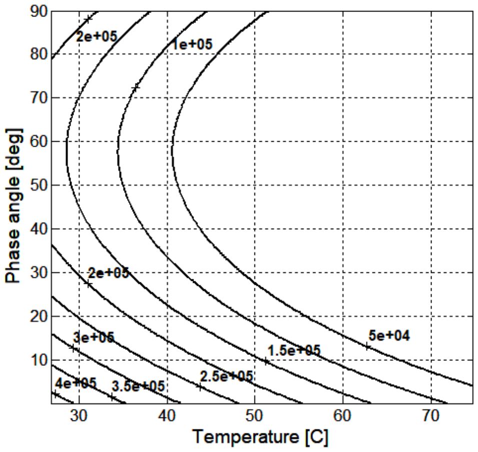
Table 1: *Regression models for the lipstick characteristics*

	Regression models	*R^2 [%]*	*R^2_{adj} [%]*
$\hat{y}_1(\vec{x})$	$106094 - 168532x_1 + 37062x_2 + 22270x_1^2 + 156319x_2^2 - 119409x_3^2 + 125116x_2x_3$	85.73	85.26
$\hat{y}_2(\vec{x})$	$795.8 - 992x_1 + 243.6x_2 + 266.8x_3 + 479.1x_1^2 + 368x_2^2 - 704.7x_3^2 - 1283.1x_1x_3 + 275.5x_2x_3$	82.72	82.52

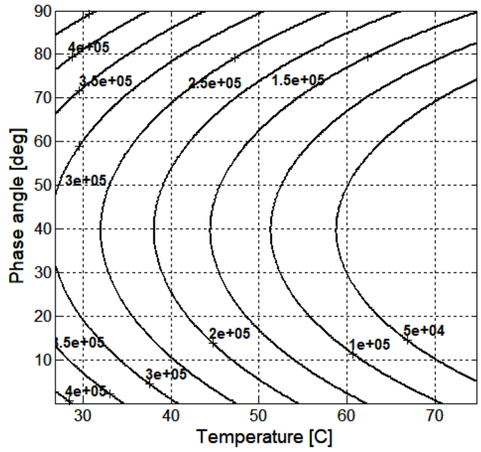
In Fig. 1 contour plots of the elastic modulus – y_1, depending on the variation of temperature (z_1) and the phase angle (z_2) for different values for strain – z_3 are presented.

On Fig. 1a it can be seen that when the temperature (z_1) is around 32 °C, which is the normal temperature for the human lips and phase angle (z_2) is minimum, the elastic modulus $\hat{y}_1(\vec{x})$ has high values. Fig 1b shows that in order to obtain a maximum of $\hat{y}_1(\vec{x})$, z_2 must have very large or small values within the experimental region, and in Fig. 1c it can be seen that maximum of the investigated lipstick characteristic is reached when the phase angle z_2 has smallest values (0.0024 deg).

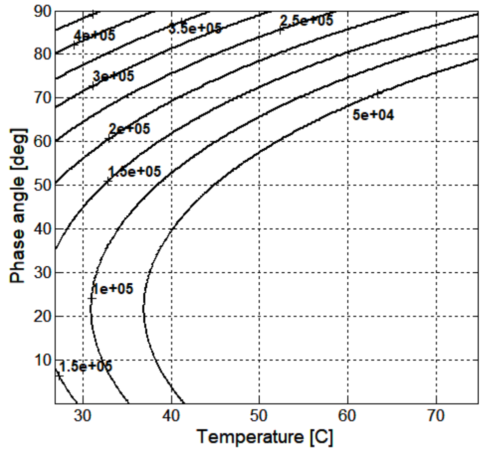
Contour plots of the shear stress – $\hat{y}_2(\vec{x})$ of the investigated commercial brand lipstick, depending on the variation of temperature (z_1) and the phase angle (z_2) for different values for strain – z_3 are shown in Fig. 2. In the first case (Fig. 2a) it can be seen that around the normal human lip's temperature (32 °C) $\hat{y}_2(\vec{x})$ and phase angle is between 30 and 60 deg. the shear stress is less than 5 Pa. On other two contour plots graphics (Fig 2b and Fig 2c) $\hat{y}_2(\vec{x})$ has a maximal value, if the z_2 is maximum too.



a) $z_3 = 1.37\times10^{-5}$

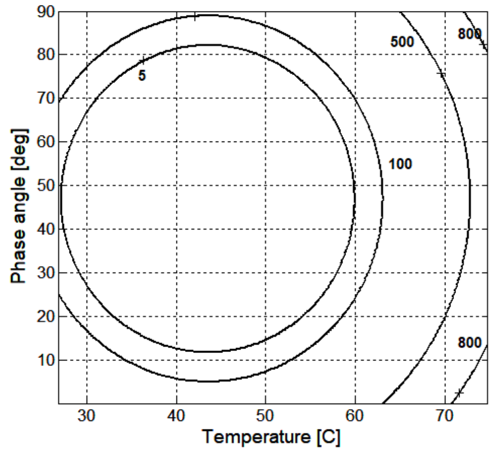


b) $z_3 = 7.42185\times10^{-3}$

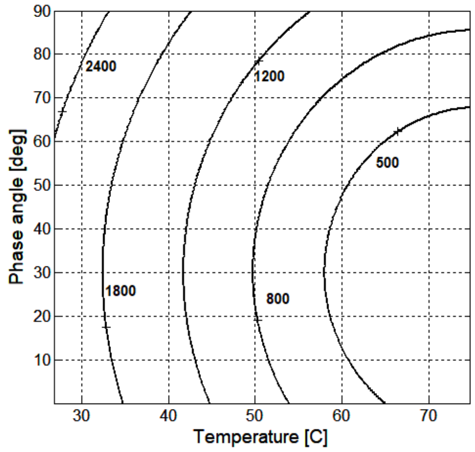


c) $z_3 = 1.37\times10^{-5}$

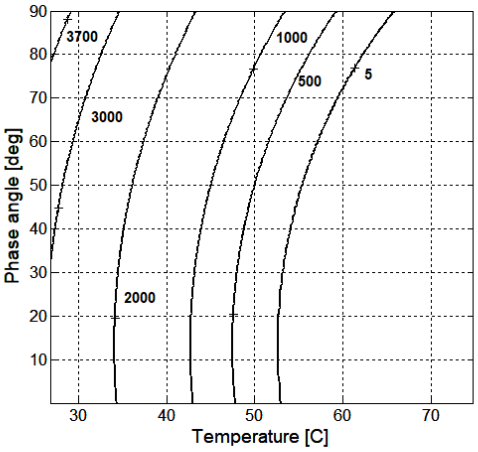
Fig. 1 *Contour plots of the elastic modulus – $\hat{y}_1(\vec{x})$, depending on the variation of Temperature (z_1) and the Phase angle (z_2) for different values for strain – z_3: a) strain – $z_3 = 1.37\times10^{-5}$; b) strain – $z_3 = 7.42185\times10^{-3}$ and c) strain – $z_3 = 1.483\times10^{-2}$.*



a) $z_3 = 1.37\times10^{-5}$



b) $z_3 = 7.42185\times10^{-3}$



c) $z_3 = 1.483\times10^{-2}$

Fig. 2 *Contour plots of the shear stress – y_2, depending on the variation of Temperature (z_1) and the Phase angle (z_2) for different values for strain – z_3: a) strain – $z_3 = 1.37\times10^{-5}$; b) strain – $z_3 = 7.42185\times10^{-3}$ and c) strain – $z_3 = 1.483\times10^{-2}$.*

5. Conclusions

Investigation of the commercial brand lipstick using oscillatory shear rheology in order to tested its rheological and thermal properties is performed.

Regression models for the elastic modulus and the shear stress of the investigated product depending on the variations of the process parameters were estimated.

The obtained regression models can be used for future investigation and optimization of the rheological and thermal properties of the investigated commercial brad lipstick.

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