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“Lipsticks: Understanding Structure-Property Relationships and Material Breakdown Mechanisms through Oscillatory Rheology”

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Abstract

Lipsticks are heterogeneous, fat-based cosmetics historically made from a combination of wax, oil, and pigments. As consumers have become increasingly demanding about product performance, formulations have evolved substantially, now incorporating a variety of advanced components such as polymers, silicones, and fillers. This evolution has made the mechanisms underlying the macroscopic properties of lipsticks increasingly complex. Understanding the mechanical properties of lipsticks and their behavior under loading has therefore become of paramount importance.

Oscillatory rheology is a valuable technique for investigating the viscoelastic properties of materials under strain, offering insight into their structural behavior at the microscale. In this study, the linear and non-linear viscoelastic properties of three grades of commercially available lipsticks are explored by means of oscillatory rheological experiments, respectively Small Amplitude Oscillatory Shear (SAOS) and Large Amplitude Oscillatory Shear (LAOS).

The large deformation oscillatory rheology provided valuable insights into the microstructural mechanisms leading to material failure: the Lissajous-Bowditch plots revealed that all lipsticks demonstrate intracycle strain-stiffening and shear-thinning non-linearities prior to structural breakdown. These non-linear behaviors indicate an elastic stiffening as well as a decrease in viscosity under shear during individual oscillation cycles.

This work shows that each lipstick exhibits distinct linear rheological properties in the small deformation regime, corresponding to different material textures and sensory qualities. Moreover, significant differences in their behavior emerge in the large deformation regime, where non-linear stress responses are observed. These observations deepen our understanding of the mechanisms behind material breakdown, which is essential for optimizing formulation design and improving product performance.

1. Introduction

Lipsticks are emblematic makeup products that have mirrored cultural and societal transformations over time, with evidence of their use dating back to the Bronze Age [1,2]. Nowadays, lipsticks play a central role in the beauty industry and represent a major share of global cosmetics sales [3]. As market trends evolve, emphasis is placed on the sensory qualities and product finish on the lips, leading to continuous innovation in formulation. Indeed, customers expect lipsticks that are both flawless in appearance and refined in texture, particularly in the luxury cosmetics sector. Originally, lipsticks relied on simple blends of oils, waxes, butters and pigments, but recent formulations incorporate a wide range of additional ingredients such as polymers, silicones, and fillers. As a result, modern lipstick formulas are complex and often contain more than thirty ingredients [4].

The desired sensory attributes of a lipstick are closely linked to its rheological properties: Achieving a soft texture with a melting effect upon application can lead to a reduced rigidity of the finished product and weaken the structural integrity of the stick. The delicate balance between sensory qualities and mechanical stability becomes even more complex in the context of the “Clean Beauty” movement, which pushes for the replacement of long-established synthetic ingredients with natural alternatives, adding further complexity to the formula stability [5–7].

While some research projects have investigated the sensory properties of lipsticks [3,8,9], as well as their crystallization kinetics [10] and thermal stability [7,11–13], the rheological response of commercial lipsticks at room temperature has received limited attention and remains poorly understood. This is, however, of major importance for understanding the performance in service – as lipsticks must withstand important shear stresses and shear rates during application while maintaining structural integrity and performance under these conditions.

The present study aims to provide a comprehensive understanding of the structural properties of lipsticks and of their breakdown mechanisms under shear loading, by investigating the rheological behavior of three distinct commercially available lipsticks and characterizing their response to large amplitude shear.

2. Materials and Methods

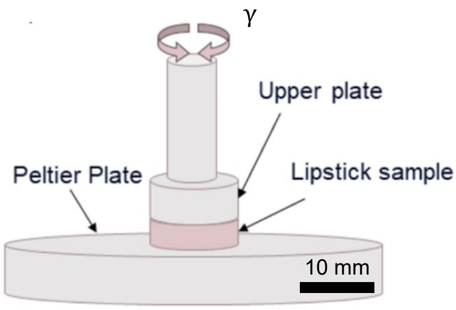
2.1. Materials

The study was conducted on samples of three commercially available lipsticks, each formulated with different ingredient compositions and varying lipid content, labelled as L1, L2, and L3. These formulations were chosen to capture a wide range of textures and formulations, with the aim of exploring the interplay between their composition and rheological properties. L1 is characterized by its softness and smoothness on the lips. In contrast, L3 offers a matte appearance and a firm texture. L2 provides a more traditional lipstick experience, balancing both sensorial qualities and stick aspect.

2.2. Methods – Oscillatory Shear Rheology

Oscillatory rheological measurements were performed using a stress-controlled rheometer (TA Instruments, USA) equipped with a fixed Peltier plate for precise temperature control. Data acquisition was carried out using the TRIOS 5.1 software. A 12 mm diameter plate was used as the upper mobile geometry, as shown in Figure 1.b. To prevent the sample from slipping during deformation, the top and bottom plates were lined with abrasive paper.

Prior to testing, the lipstick samples were stored in a proofer at 20 °C for a minimum of 12 hours. During isothermal measurements, the Peltier plate was kept at 20 °C. Thin slices about 1.5 mm high were cut from the sticks and placed on the Peltier stage. The top geometry was then lowered to create a 1 mm gap, and excess material was carefully removed with a spatula. Amplitude sweeps were conducted at fixed angular frequencies of ω = 10 Hz over a stress amplitude range of 0.001 % < γ < 100%.



(**a**) (**b**)

Figure 1. a) Representation of the mechanical loading applied on the lipstick during application on lips; b) Schematic of the oscillatory rheology set up used to perform small amplitude and large amplitude oscillatory shear measurements.

3. Results

3.1. Linear rheological behavior

Figure 2 shows the storage (G') and loss (G'') moduli as a function of oscillatory shear stress, obtained from oscillatory strain amplitude sweeps at 10 Hz over the strain range 0.001 % < γ < 100 % for the three lipstick samples. In the linear region, the rheological properties remain constant, indicating that the material structure is not altered by the applied shear loading. Table 1 summarizes the values of G', G'' and the loss factor tan(δ) for each formulation within the linear regime at an oscillation frequency of 10 Hz. Among the samples, L2 and L3 have the highest G' values, suggesting greater rigidity, comparing to L1. Moreover, L1 and L2 exhibit larger tan(δ) values than L3, indicating more prononced damping behavior. These rheological features are consistent with the perceived texture of the lipsticks. For all samples, G’ is larger than G” throughout the linear range, confirming the predominance of elastic behavior in this regime.

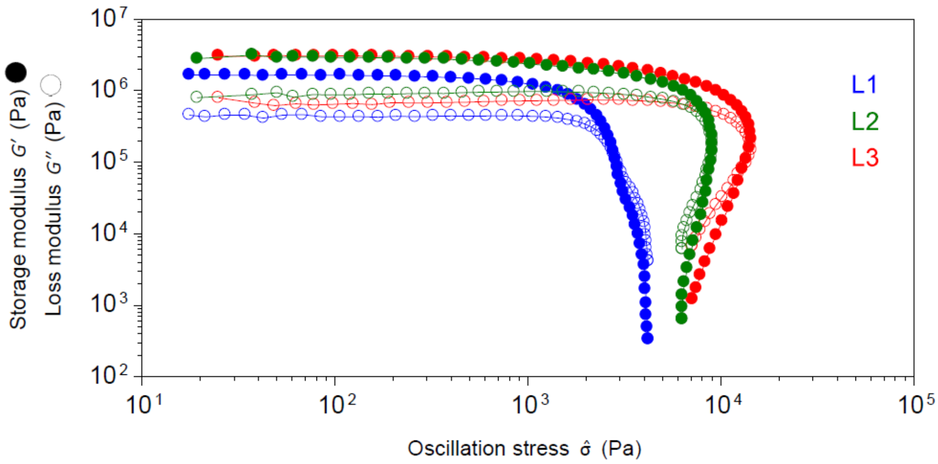


Figure 2. Storage (G’) and loss (G’’) moduli as a function of oscillatory shear stress, obtained from oscillatory strain amplitude sweeps at 10 Hz over the strain range 0.001 % < γ < 100 %

Table 1. Storage modulus G’, loss modulus G’’ and loss factor tan(δ) measured in the linear viscoelastic region during amplitude sweep oscillatory rheological tests conducted at 10 Hz and at 20 °C.

Lipstick sample	G’ (Pa)	G’’ (Pa)	tan(δ)
L1	1636790	440653	0.27
L2	2883500	886191	0.31
L3	3034690	669597	0.22

3.2 . Material response to large amplitude shear

The extent of the linear region on Figure 2 indicates the maximum shear stress amplitude that each material can withstand before deviating from its linear behavior and entering the plastic deformation regime. Table 2 displays the maximum shear stress limit σ_f before failure for the three lipsticks, corresponding to the onset of structural failure of the material and to the G’ and G’’ crossover visible on Figure 2. Notably, L3 exhibits a failure stress value almost seven times larger than that of L1 value, indicating a significantly greater resistance to stress before collapsing. L2 exhibits an intermediate stress value, falling in between.

At large shear amplitudes, Figure 3 reveals differences in failure behavior. Both L2 and L3 curves display a maximum in stress before structural collapse, whereas in L1, stress continues to slighly rise even after irreversible damage has occured. This contrast highlights different stress dissipation mechanisms across the formulations.

Table 2. Maximum shear stress σ_f before failure and collapse of the elastic network and corresponding failure strain γ_f measured during amplitude sweep oscillatory rheological tests conducted at 10 Hz and at 20 °C.

Lipstick sample	σ_f (Pa)	γ_f
L1	2842	1.8 %
L2	9088	7.1 %
L3	14327	5.6 %

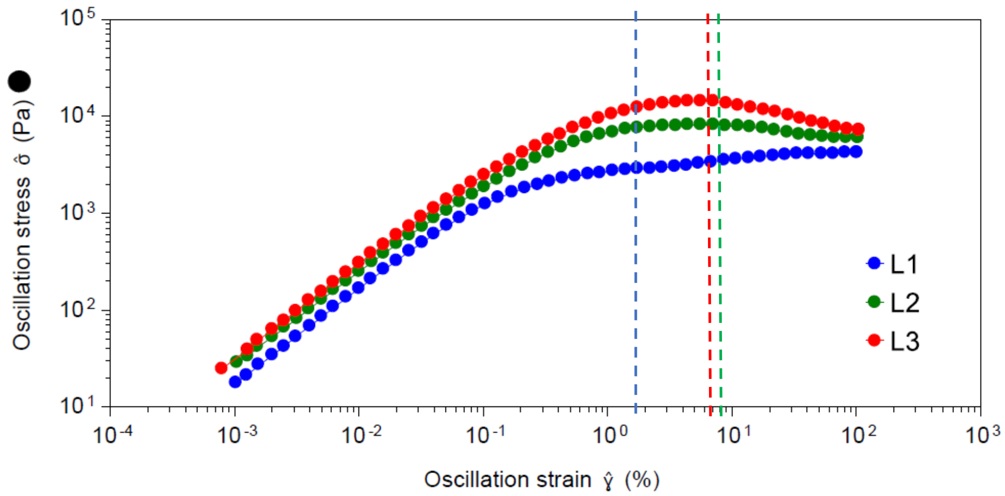


Figure 3. Oscillatory strain-stress curves obtained from strain amplitude sweeps at 10 Hz over the strain range 0.001 % < γ < 100 %. The dashed vertical lines correspond to the maximum shear strain values before the structural collapse and breakdown of the material (G' and G'' crossover point visible on Figure 2)

The elastic and viscous Lissajous-Bowditch curves shown in Figure 4 provide qualitative insights into the structural breakdown of the materials. These curves were plotted for the three lipstick samples at an oscillation frequency of 10 Hz and at two shear strain amplitudes: 0.1 % and 1.7 %. At 0.1 %, the curves appear as perfect ellipses, indicating that the behavior of the lipsticks is still linear. However, we observe a distortion of the ellipses when the shear strain is increased to 1.7 %, reflecting the onset of non-linearities appearing in the stress response of the samples.

In the elastic plot, an upward distortion of the ellipse is observed, which is indicative of intracycle strain stiffening of the material [8,14]. In the viscous plot, the ellipse shows a downwards distortion, revealing intracycle shear thinning behavior: as the shear rate is increased, the viscosity of the materials decreases. This property is particularly relevant in cosmetic applications, as it is closely linked to sensory qualities. As previsouly demonstrated, the L1 material is less resistant to shear strain than the two others; therefore, the more pronounced non-linear response observed under the same loading amplitude is consistent with its weaker structure.

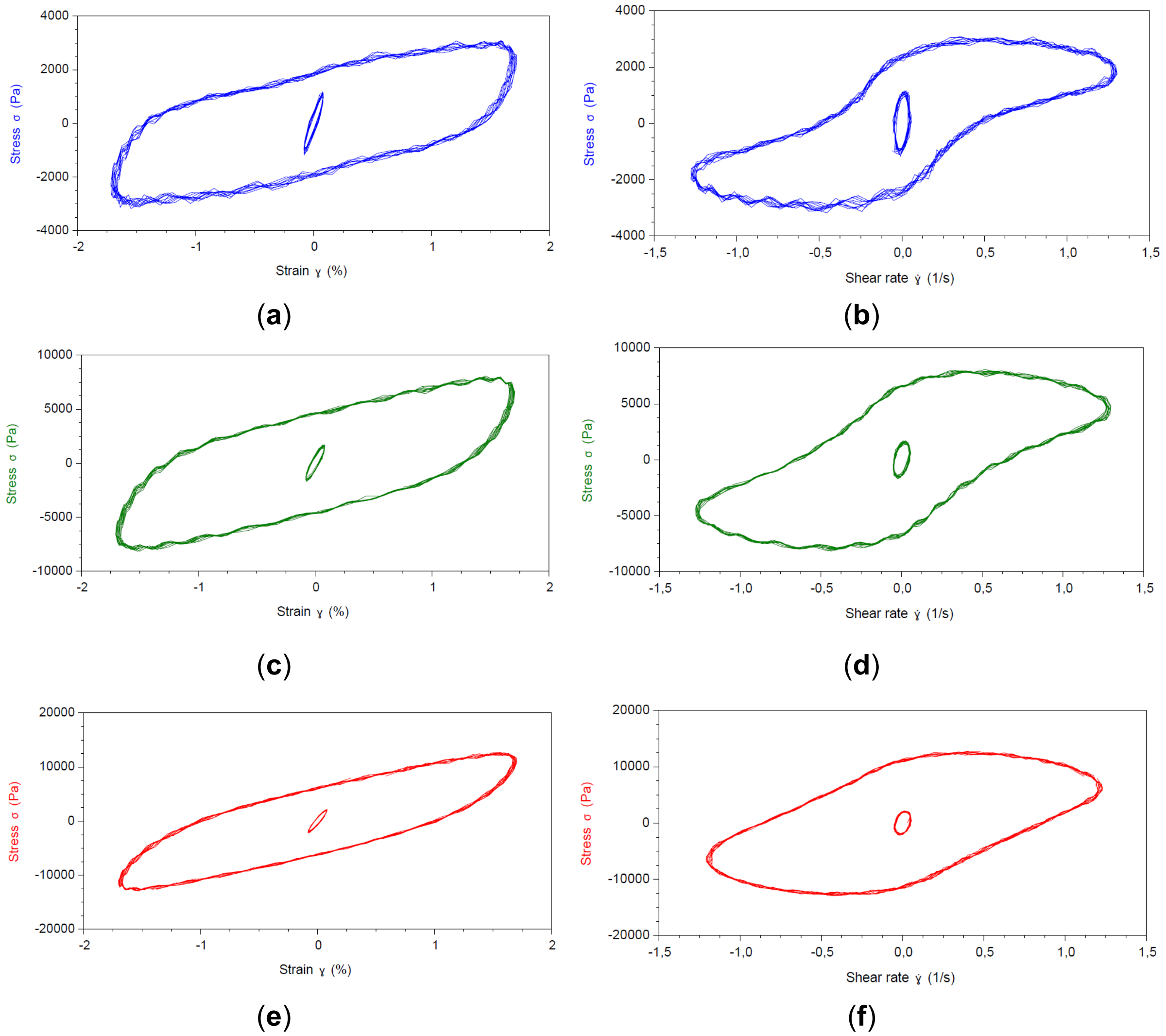


Figure 4. Elastic Lissajous-Bowditch curves of the lipstick samples L1 (a), L2 (c) and L3 (e) and viscous Lissajous-Bowditch curves of the lipstick samples L1 (b), L2 (d) and L3 (f) at an oscillation frequency of 10 Hz and at the strain amplitudes of 0.1 % and 1.7 %

4. Discussion

Fat-based materials are widely used in the food industry and have been the subject of extensive rheological studies aimed at understanding their texture and mechanical stability [15–18]. Cosmetics materials, and particularly lipsticks, are also predominantely fat-based, and their rheological properties are strongly formulation-dependent. While several studies have investigated wax-oil binary systems and simplified lipstick systems in order to improve the general understanding of lipstick microstructure and macroscopic behavior [3,7,8,10,19], research on the mechanical integrity of full-formula lipsticks at room temperature remains limited. In this study, the rheological behavior of the three lipstick formulations revealed differences in structure and stability under deformation, which can be directly related to their functional performance and sensory qualities.

In the linear viscoelastic regime, all samples exhibit a predominance of elastic behavior, indicating solid-like properties at small strain amplitudes. Among the three, L3 displayed the largest storage modulus G′, suggesting a more rigid internal structure, followed closely by L2. L1 softer texture was reflected by its significantly smaller G′ value. Moreover, L1 and L2 exhibited higher loss factor values, indicating a more pronounced viscous response compared to L3, which maintained a more elastic profile. These observations are consistent with the perceived firmness of the lipsticks, suggesting that L1 and L2 may appear smoother during application on lips while L3 may offer more hold.

As strain amplitude increased beyond the linear region, stronger differences appeared in the structural resilience of the materials. The maximum stress measured before failure (Table 2) revealed the superior mechanical strength of L3 while L2 demonstrated intermediate resistance. Moreover, L2 and L3 displayed a distinct stress peak followed by structural collapse (Figure 3), typical of materials that fail through cohesive network breakdown. Conversely, L1 did not exhibit a clear yield stress point after structural failure, indicating a less defined elastic network. Further qualitative insights were obtained through Lissajous–Bowditch analysis (Figure 4). Nonlinear intracycle responses such as strain-stiffening and shear-thinning were observed. Shear-thinning is particularly desirable in cosmetic formulations, as it enhances spreadability. These observations are consistent with the one previously made on research about lipstick sensoriality properties at lip temperature (32 °C) [8,20]. L1 exhibited the most pronounced distortions, consistent with its weaker structure and earlier departure from the linear regime, which reinforces the conclusion that L1 is less mechanically stable under large deformations, while L2 and L3 maintain more cohesive networks.

5. Conclusion

This study examined the rheological properties of three lipstick formulations, enabling clear discrimination between the materials and revealing interesting differences in mechanical integrity closely linked with the texture of the product. In the linear viscoelastic regime, the softest product (L1) exhibited a smaller elastic modulus and a larger damping factor, indicating a more pronounced viscous response to applied stress and strain. In contrast, the stiffest and matte material (L3) showed a predominantly elastic response in the linear regime with reduced damping properties. Beyond the linear regime – where the materials began to undergo plastic deformation prior to collapse – distinct mechanisms emerged between the materials. Despite its lower stress tolerance compared to L2 and L3, the soft product L1 was the only one not exhibiting a stress maximum in the stress-strain curves, suggesting a less cohesive and less defined elastic network.

Moreover, the large amplitude oscillatory rheological profiles provided a rich qualitative and quantitative framework for understanding the microstructure of the materials and their mechanical stability under shear stress. Intracycle strain-stiffening and shear-thinning non-linearities were observed before material collapse. As we increased the strain amplitude to 100 %, the material breakdown was marked by a sharp decrease in both shear moduli, leading to a crossover between the storage modulus and the loss modulus.

These findings offer valuable insights for optimizing product performance and for tailoring formulations to achieve mechanical integrity of the stick material, combined with optimal sensory attributes in cosmetic applications.

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