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Ethylcellulose oleogel lip glosses: the effects of different emollients and pigments on rheology and textural properties

Introduction

Lip glosses enhance the appearance of the lips by imparting shine and a hint of colour. For a smooth and homogenous application of colour on the lips, pigments must be wetted by the oils, for which Ricinus communis (Castor) oil (CO) is commonly used [1]. **Oleogels** are systems of oils entrapped within oleogelator networks, which **have versatile application potential in cosmetics** [2]. **Ethylcellulose (EC) is a biocompatible oleogelator that structures oils** via polymer-polymer and polymer-solvent hydrogen bonding mechanisms [3].

The **aim of this study** was to investigate the effects that different emollients and pigments have on the physical properties of EC oleogel lip glosses.

Materials & Methods

Castor oil (CO) was selected as the main oil for its good pigment wetting properties [1]. A **red iron oxide pigment (RP)**, a **small particle size pearl (SP)** and a **large particle size pearl (LP)** were used [1]. The composition of **oleogels (without pigment)** and **lip glosses (oleogels with pigment)** is detailed in **Tables 1 and 2**, respectively. Oleogels were manufactured by heating all ingredients to 150º C and cooling whilst continuously stirred at 250 rpm. Lip glosses were obtained by adding pigments to oleogels via manual stirring until homogenous. Rheological characterisation was performed with a HAAKE™ MARS™ iQ Air Modular rheometer with a parallel plate geometry with a 1.0 mm gap (Thermo Fisher Scientific, USA) at 20º C. Shear rate sweep (0.1 s^{-1}–100 s^{-1}) was used to measure viscosity. Oscillatory stress sweep (1–1400 Pa, 1 Hz) was used to measure complex modulus (G*) and phase angle (δ). Firmness, spreadability and stickiness were tested using a TA.XTplus Texture Analyser (Stable Micro Systems, UK), with a TTC Spreadability Rig and Heavy Duty Platform at 22 ± 1º C.

Table 1. Composition of EC oleogels with different emollient mixtures.

Oleogel	CO	CO/CCT	CO/DC	CO/CTT/DC
Oil (INCI)	% (w/w)			
Ethylcellulose (EC)	5.0	5.0	5.0	5.0
Ricinus communis Oil (CO)	95.0	47.5	47.5	31.6
Caprylic/Capric Triglycerides (CCT)	-	47.5	-	31.6
Dicaprylyl Carbonate (DC)	-	-	47.5	31.6

Table 2. Composition of lip glosses. **SP** (Small Particle Size Pearl, particle size 5–25 μm, INCI: Mica (and) Titanium Dioxide), **LP** (Large Particle Size Pearl, particle size 20–200 μm, INCI: Calcium Aluminium Borosilicate (and) Titanium Dioxide (and) Silica (and) Tin Oxide) and **RP** (Red Pigment, particle size 5–50 μm, INCI: Iron Oxide/CI 77491 (and) Silica).

% (w/w)		Oleogel Base											
		CO			CO/CCT			CO/DC			CO/CCT/DC		
EC Oleogel		90	90	90	90	90	90	90	90	90	90	90	90
Pigment	RP	10	-	-	10	-	-	10	-	-	10	-	-
	SP	-	10	-	-	10	-	-	10	-	-	10	-
	LP	-	-	10	-	-	10	-	-	10	-	-	10

Results – Viscosity

- **Figure 1**: all oleogels and lip glosses were shear-thinning, as expected.
- **Figure 1A**: CO alone resulted in the most viscous oleogels; at the lowest CO concentration (CO/CCT/DC) the resulting oleogel was the least viscous. CO's unique hydroxyl group in its alkyl chain [4] is responsible for its ability to form hydrogen bonds with EC [5].
- **Figures 1B–E**: pigments had little to no effect on the viscosity of oleogels, suggesting that the application of EC oleogels on the lips may be similar when pigments are included. This is probably due to EC's gelation mechanism [3], where CO plays an important role in maintaining EC's network structure [4,5], which appears to be independent of the presence of solid particles.

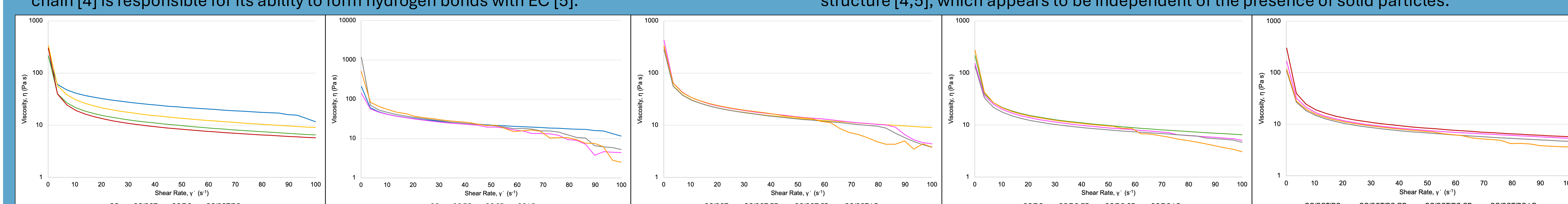

Figure 1. Viscosity curves of oleogels and lip glosses. **A**: CO, CO/CCT, CO/DC and CO/CCT/DC. **B**: oleogel CO and lip glosses CO-RP, CO-SP and CO-LP. **C**: oleogel CO/CCT and lip glosses CO/CCT-RP, CO/CCT-SP and CO/CCT-LP. **D**: oleogel CO/DC and lip glosses CO/DC-RP, CO/DC-SP and CO/DC-LP. **E**: oleogel CO/CCT/DC and lip glosses CO/CCT/DC-RP, CO/CCT/DC-SP and CO/CCT/DC-LP.

Results – Viscoelastic Properties

- **Figure 2**: all oleogels and lip glosses presented phase angles above 45º, describing a predominantly viscous (liquid) behaviour, confirming visual observations (all formulas were free-flowing).
- **Figure 2A**: in line with Figure 1, the CO oleogel was the most rigid, followed by CO/CCT, then CO/DC and finally CO/CCT/DC, confirming that increased hydrogen bonding, oil polarity and unsaturation levels result in stronger EC oleogels [3,5]. The CO oleogel was the least elastic, followed by CO/CCT, then CO/DC and finally CO/CCT/DC as the most elastic oleogel. This suggests that CO contributes to reducing the brittleness of EC oleogels.
- **Figures 2B–E**: pigments increased the rigidity of oleogels, as seen by the higher complex moduli of all lip glosses when compared to their original EC oleogels; pigments had little to no effect on the phase angles of EC oleogels.

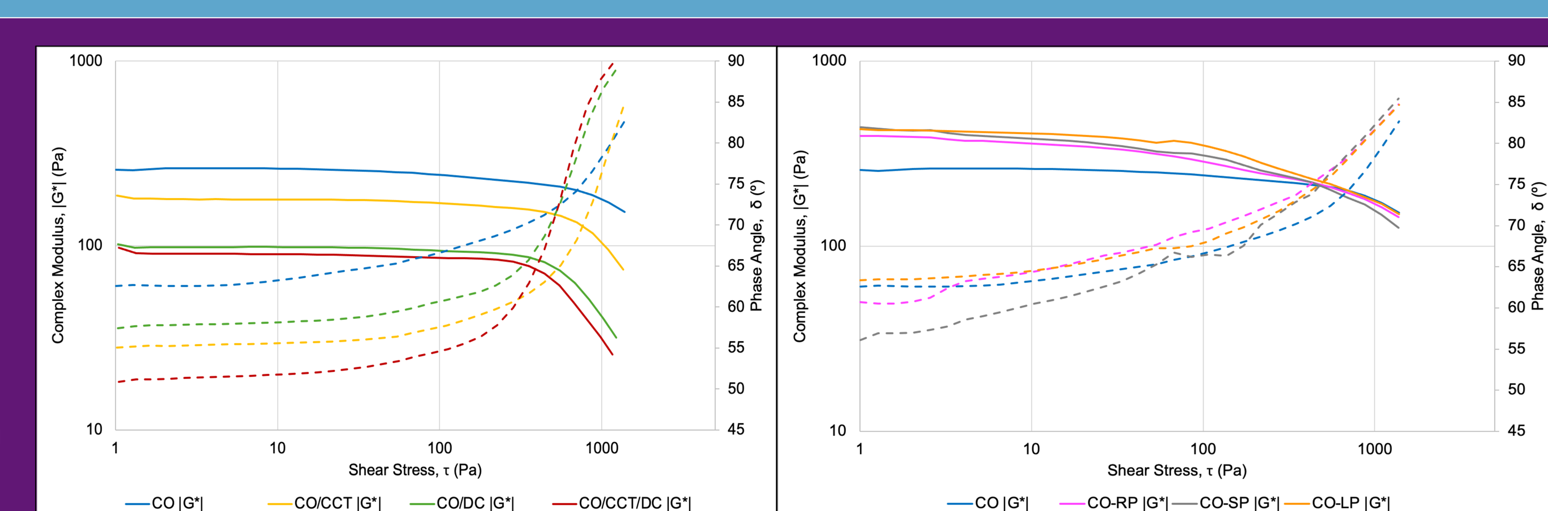
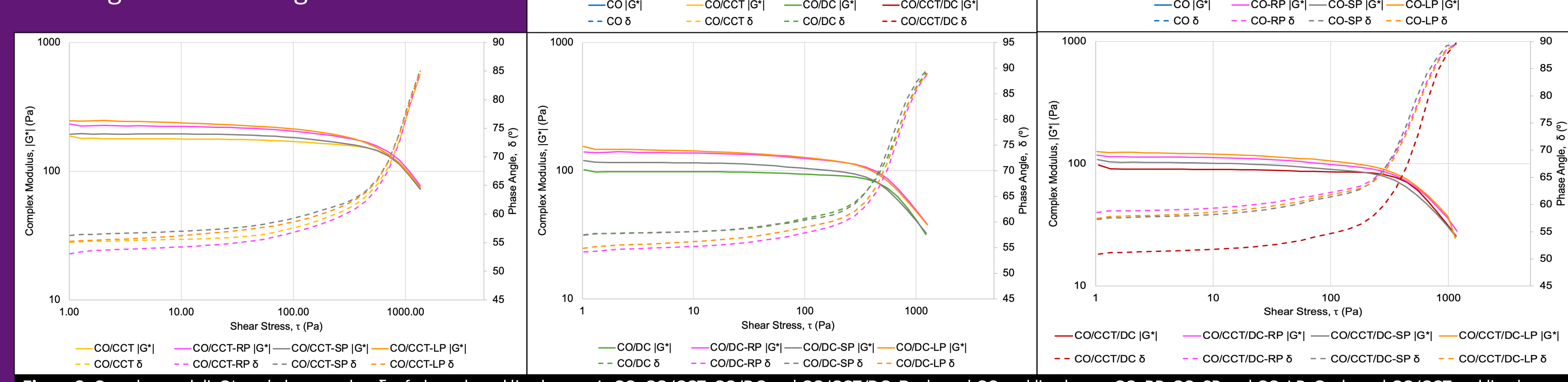

Figure 2. Complex moduli, G*, and phase angles, δ, of oleogels and lip glosses. **A**: CO, CO/CCT, CO/DC and CO/CCT/DC. **B**: oleogel CO and lip glosses CO-RP, CO-SP and CO-LP. **C**: oleogel CO/CCT and lip glosses CO/CCT-RP, CO/CCT-SP and CO/CCT-LP. **D**: oleogel CO/DC and lip glosses CO/DC-RP, CO/DC-SP and CO/DC-LP. **E**: oleogel CO/CCT/DC and lip glosses CO/CCT/DC-RP, CO/CCT/DC-SP and CO/CCT/DC-LP.

Table 3. Firmness (g), work of shear, WoS (g s), and stickiness (g) of oleogels, lip glosses and **LGB – lip gloss base commercial benchmark**, INCI: Hydrogenated Polyisobutene (and) Ethylene/Propylene/Styrene Copolymer (and) Butylene/Ethylene/Styrene Copolymer (and) BHT. (mean ± standard deviation).

Formulation	Firmness (g)	WoS (g s)	Stickiness (g)
Lip Gloss Base	540.00 ± 4.19	361.52 ± 4.36	-875.98 ± 7.11
LGB – RP	656.58 ± 8.09	449.31 ± 3.85	-964.35 ± 17.96
LGB – SP	605.31 ± 6.96	421.64 ± 5.41	-898.32 ± 0.59
LGB – LP	601.42 ± 11.14	432.31 ± 9.77	-883.13 ± 21.47
CO	2151.89 ± 12.45	4194.63 ± 22.56	-2108.24 ± 4.37
CO – RP	1635.36 ± 10.51	1007.85 ± 12.81	-1956.40 ± 3.15
CO – SP	1584.65 ± 16.05	945.42 ± 29.57	-1947.47 ± 8.99
CO – LP	1937.69 ± 15.24	1242.02 ± 16.45	-2042.71 ± 5.98
CO/CCT	1525.26 ± 30.16	1052.58 ± 3.73	-1775.94 ± 14.7
CO/CCT – RP	1614.47 ± 37.86	1134.28 ± 72.66	-1846.04 ± 7.06
CO/CCT – SP	1453.81 ± 42.46	972.36 ±80.13	-1802.77 ± 2.08
CO/CCT – LP	1530.19 ± 20.83	1087.49 ± 55.41	-1808.19 ± 8.29
CO/DC	1121.58 ± 16.23	759.81 ± 39.63	-1385.68 ± 13.46
CO/DC – RP	1095.95 ± 43.81	754.11 ± 75.81	-1308.25 ± 12.34
CO/DC – SP	1065.03 ± 18.92	711.85 ± 44.84	-1343.98 ± 10.45
CO/DC – LP	1217.11 ± 40.95	848.75 ± 68.89	-1500.39 ± 10.60
CO/CCT/DC	524.85 ± 16.23	418.80 ± 39.63	-471.24 ± 13.46
CO/CCT/DC – RP	493.59 ± 43.81	385.62± 75.81	-476.37 ± 12.34
CO/CCT/DC – SP	493.65 ± 18.92	380.07 ± 44.84	-453.97 ± 10.45
CO/CCT/DC – LP	519.09 ± 40.95	403.02 ± 68.89	-500.47 ± 10.60

Results – Texture Analysis

- **Table 3**: all oleogels and lip glosses were substantially firmer, stickier and less spreadable compared to LGB, probably due to CO's ability to interact strongly with EC via hydrogen bonding [4,5], whereas LGB is hydrocarbon-based. In line with Figures 1 and 2, CO oleogels were the firmest, the stickiest and the least spreadable. Pigments had no relevant effect in the textural parameters of EC oleogels.

Conclusion

Results have shown that **CO determines EC oleogel stability, viscosity, rigidity and viscoelasticity**. CCT and DC reduced the viscosity and rigidity of EC/CO oleogels without affecting their stability, however, they increased elasticity, which could benefit storage stability. Pigments did not change the viscosity of EC oleogels, but slightly increased their rigidity. Texture analysis showed that mixing CO with other oils is key in obtaining lip glosses of acceptable textural properties.
Overall, **EC/CO oleogels have shown considerable potential as bases for lip gloss applications**.

References: [1] Faulkner EB, Hollenberg JC (2021) Coloring the Cosmetic World. 2nd ed. USA: Wiley. [2] Martinez RM, Rosado C, Velasco MVR, Lannes SCS, Baby AR (2019) Main features and applications of organogels in cosmetics. Int J Cosmet Sci, 41(2):109-117. [3] Laredo T, Barbut S, Marangoni AG (2011) Molecular interactions of polymer oleogelation. Soft Matter 7(6):2734-2743. [4] Patel VR, Dumancas GG, Kasi Viswanath LC, Maples R, Subong BJJ (2016) Castor Oil: properties, uses, and optimization of processing parameters in commercial production. Lipid Insights, 9:1-12. [5] Davidovich-Pinhas M (2017) Thermo-gelation of ethylcellulose oleogels, in: Patel AR (ed.) Edible Oil Structuring: concepts, methods and applications. The Royal Society of Chemistry .