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

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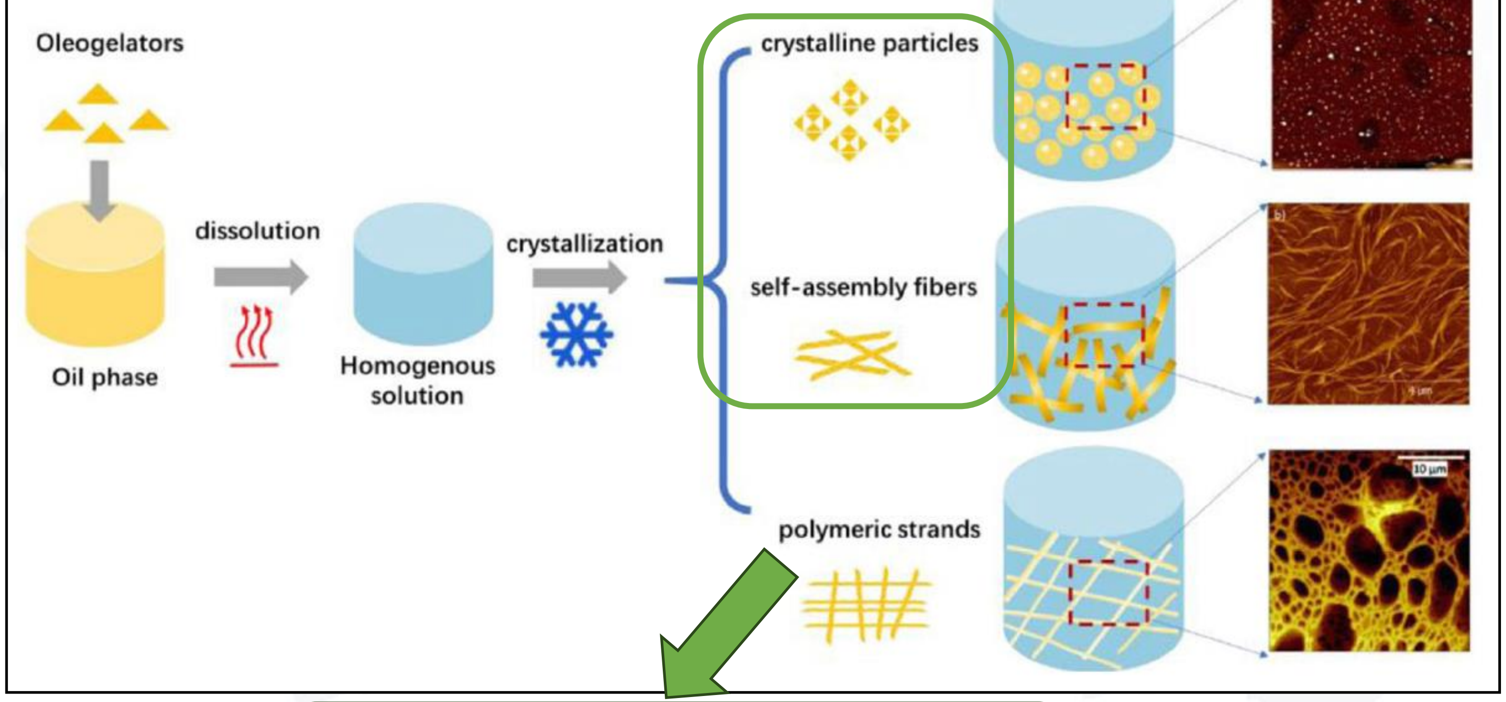
Introduction – Oleogels

- LMWO oleogels are highly sensitive to temperature and shear forces.
- HMWO oleogels are easier to manipulate, as they strongly depend on the properties and concentration of polymers (Davidovich-Pinhas et al., 2019)

Low molecular weight oleogelators

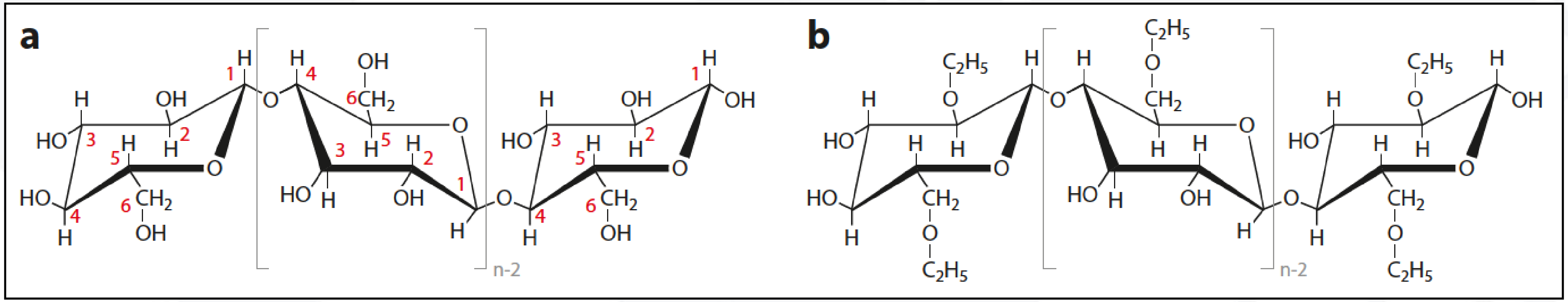
Adapted from Li, *et al.* (2022).



High molecular weight oleogelators

Davidovich-Pinhas, M. (2019) 'Oil structuring using polysaccharides', Current Opinion in Food Science, 27, pp. 29–35. doi: 10.1016/j.cofs.2019.04.006.
Li, L., Liu, G., Bogojevic, O., Pedersen, J.N. and Guo, Z. (2022) 'Edible oleogels as solid fat alternatives: Composition and oleogelation mechanism implications', Comprehensive Reviews in Food Science and Food Safety, 21(3), pp. 2077–2104. doi: 10.1111/1541-4337.12928.

Introduction – Ethylcellulose



- EC (semicrystalline polymer) forms **physical**, **thermo-reversible** oleogels via polymer-polymer **hydrogen bonding**.
- EC is biocompatible and an approved food ingredient (E462) – **assumed to be safe for lip applications**.
- The physical properties of EC oleogels can be changed using different oils and surfactants – formulation versatility.

Davidovich-Pinhas, M., Barbut, S. and Marangoni, A.G. (2016) 'Development, Characterization, and Utilization of Food-Grade Polymer Oleogels', *Annual Review of Food Science and Technology,* 7, pp. 4.1–4.27. doi: 10.1146/annurev-food-041715-033225.

Introduction – Ethylcellulose

- T_g (~120 – 140º C)
  - M_W
  - [EC]
- T_{gel}
  - M_W
  - [EC]
- CGC Hardness
  - M_W
  - [EC] Oil Type
- Pore Diameter
  - M_W
  - [EC] Oil Type
- T_{set}
  - M_W
  - [EC]

EC oleogel hardness is dependent on [EC] and M_W.

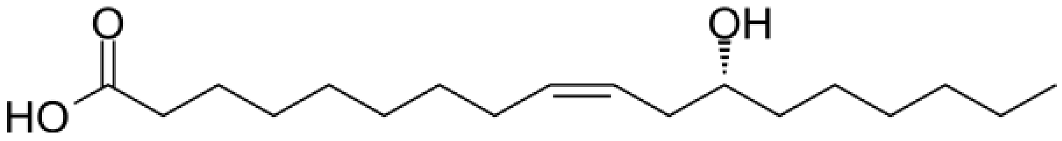
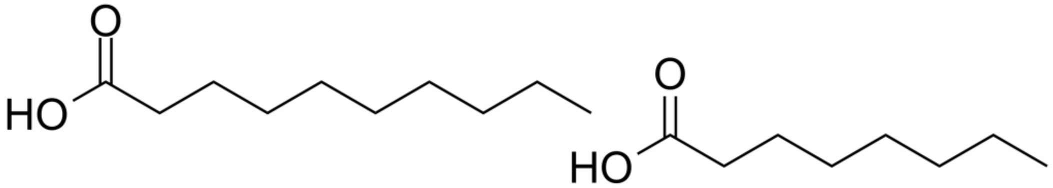
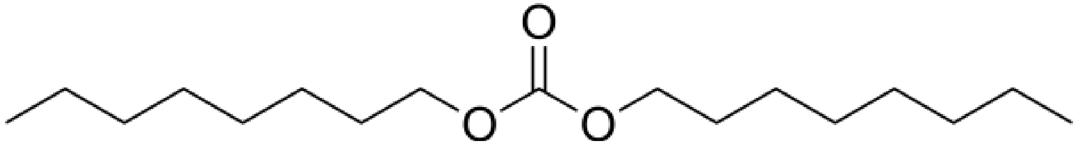
If using temperatures below T_m (~170 – 195º C), T_{set} could be critical.

Introduction – Lip Glosses

- Commonly blends of film-formers and volatiles of silicone- or petroleum-derived ingredients.
- Products should be thick liquids / easily sheared soft solids and **minimise pigment sedimentation**.
- **Castor Oil** is a common pigment wetting oil in lipsticks.

Vegetable-derived alternatives are desirable

Materials - Oils

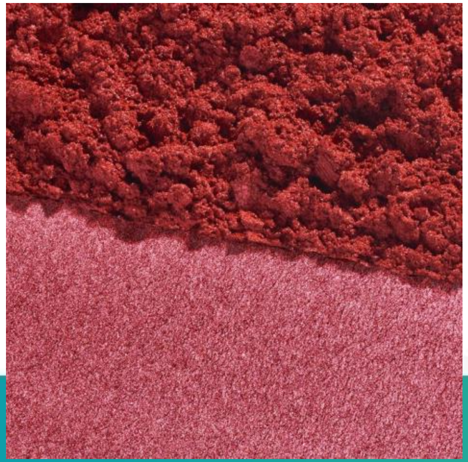
Oil INCI	Abbreviation	Chemical Structure	Density
Ricinus communis Oil	**CO** (Castor Oil)		~0.96 g cm^{-3}
Caprylic/Capric Triglycerides	**CTT**		~0.95 g cm^{-3}
Dicaprylyl Carbonate	**DC**		~0.89 g cm^{-3}

Castor Oil:

- Known for its good pigment wetting properties.
- Its unique chemistry allows hydrogen bonding with EC.

Merck, KGaA (2025) https://surface-portal.merckgroup.com/GB/en

Materials - Pigments

Red Iron Oxide / CI 77491 (and) Silica 5 – 50 µm	Mica (and) Titanium Dioxide 5 – 25 µm	Calcium Aluminium Borosilicate (and) Titanium Dioxide (and) Silica (and) Tin Oxide 20 – 200 µm
RP (Red Pigment)	**SP** (Small Particle Size Pearl)	**LP** (Large Particle Size Pearl)
		

Methods – Oleogel Preparation

Oleogel	CO	CO/CCT	CO/DC	CO/CTT/DC
INCI	% (w/w)			
Ethylcellulose (100 cP)	5.0	5.0	5.0	5.0
Ricinus communis Oil	95.0	47.5	47.5	31.6
Caprylic/Capric Triglycerides	-	47.5	-	31.6
Dicaprylyl Carbonate	-	-	47.5	31.6

All ingredients heated to 150º C and slowly cooled to 27º C whilst continuously stirred at 250 rpm.

Methods – Lip Gloss Preparation

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

Individual pigments added to respective oleogels by manually stirring until homogenous.

Methods – Product Characterisation

- Rheology
  - 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}) to measure **viscosity**.
  - Amplitude sweep (1–1400 Pa, 1 Hz) to measure **complex modulus (G*)** and **phase angle (δ)**.
- Texture Analysis
  - **Firmness**, **spreadability** and **stickiness** tested with a TA.XTplus Texture Analyser (Stable Micro Systems, UK), with a TTC Spreadability Rig and Heavy Duty Platform at 22 ± 1º C.

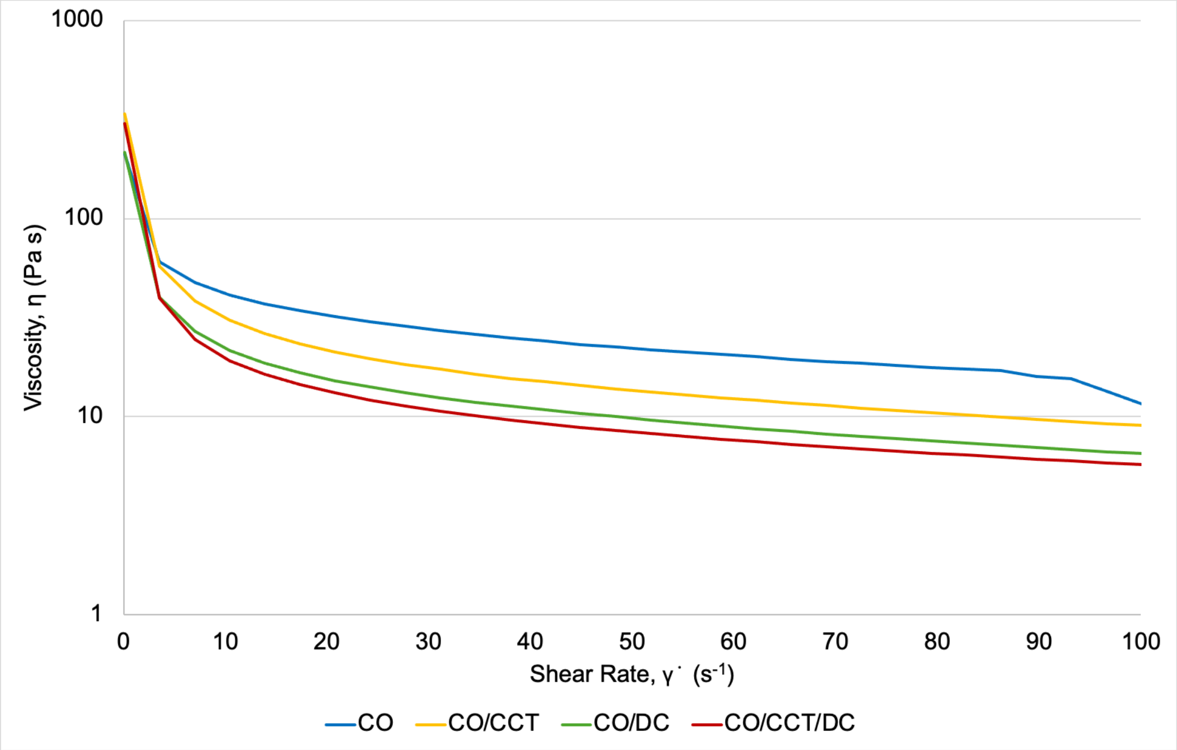
Results – Thickened Oils & Stability

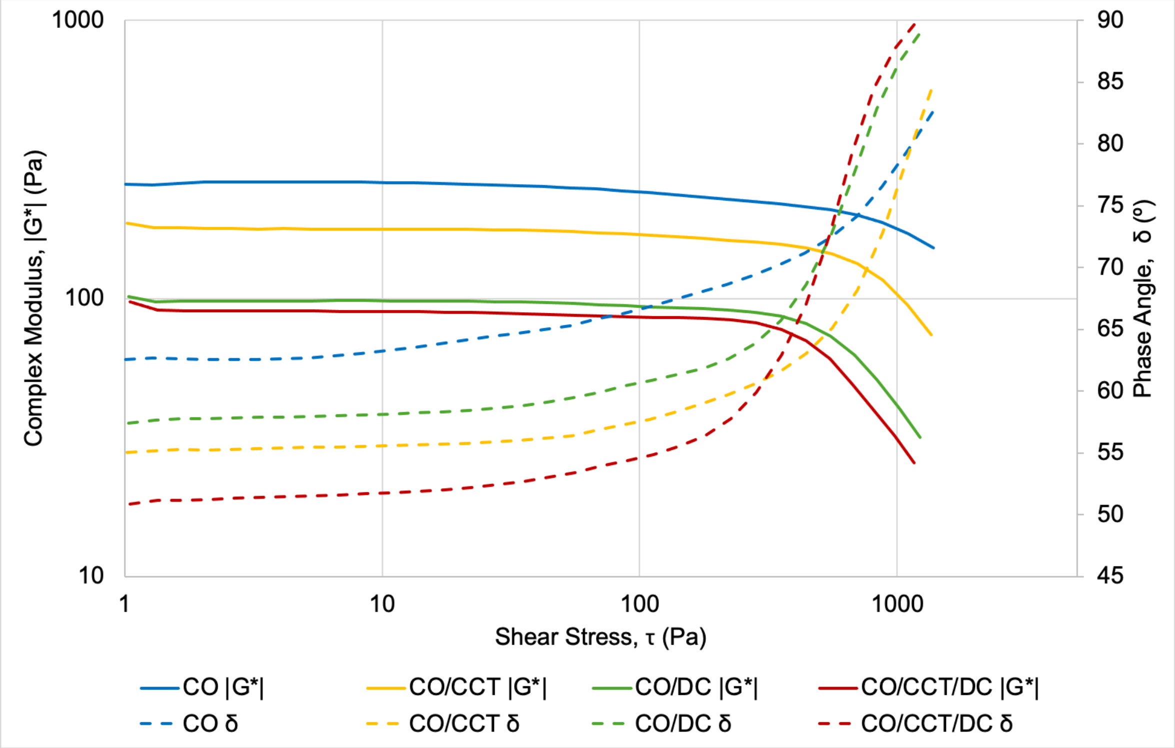
- A commercial lip gloss base (**LGB**) was used to select the EC grade providing visual and tactile properties like those of a typical lip gloss:
  - **EC 100 cP**

- Only systems with CO including oil blends were stable.
- Other single-oil systems presented some oil syneresis.
- All reported samples were stable in accelerated (40º C) conditions after 28 days.

LGB – Hydrogenated Polyisobutene (and) Ethylene/Propylene/Styrene Copolymer (and) Butylene/Ethylene/Styrene Copolymer (and) BHT

Results – Rheology of Thickened Oils

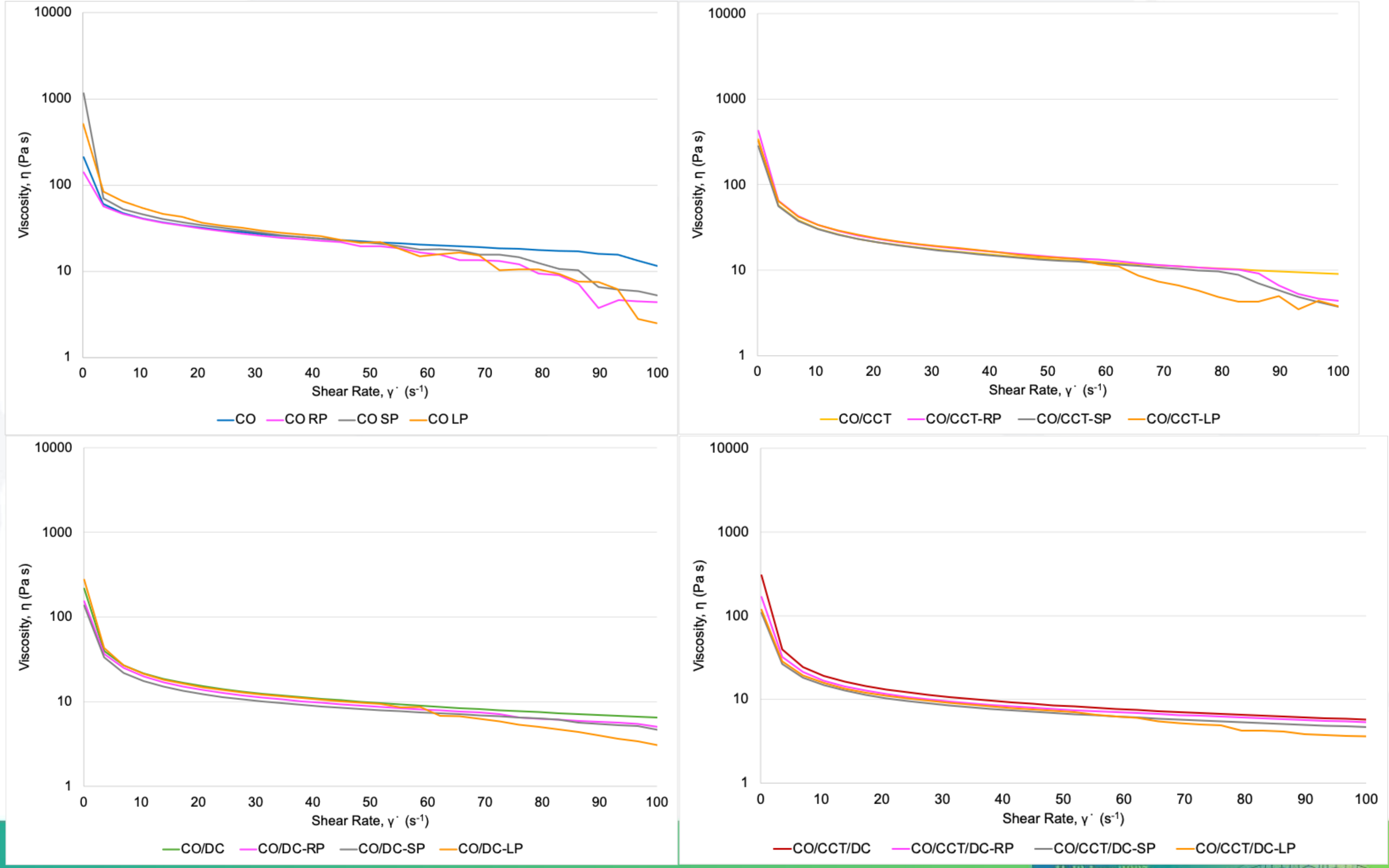




- All systems were shear thinning.
- Reducing CO resulted in lower viscosity and stiffness, but higher elasticity.

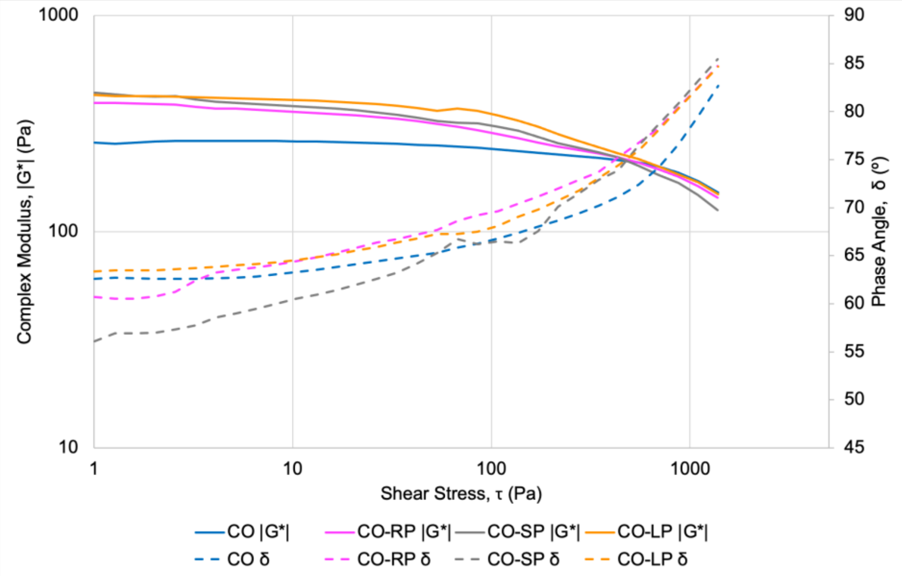
Results – Lip Gloss Viscosity

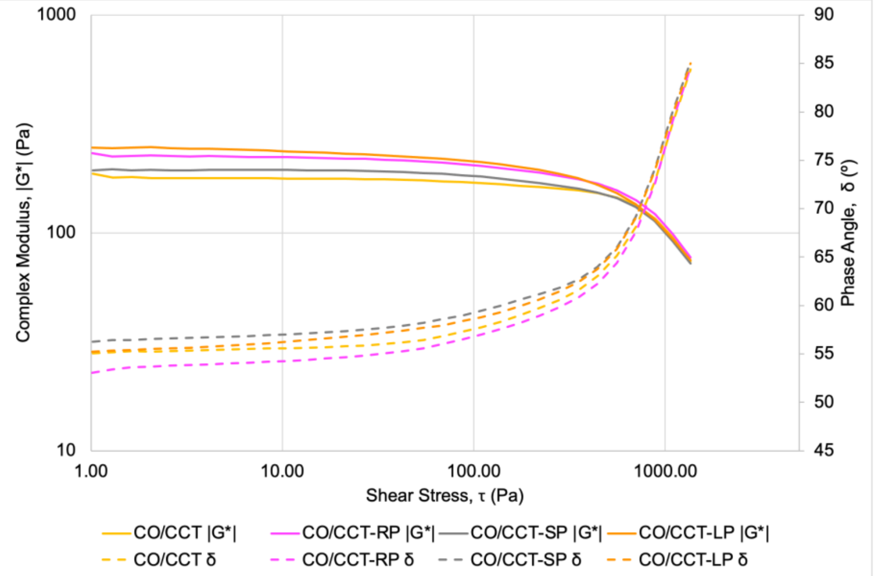
- Pigments did not change viscosity of original systems.
- Collision and aggregation of particles may have resulted in inconsistent curves at high shear, especially LP.

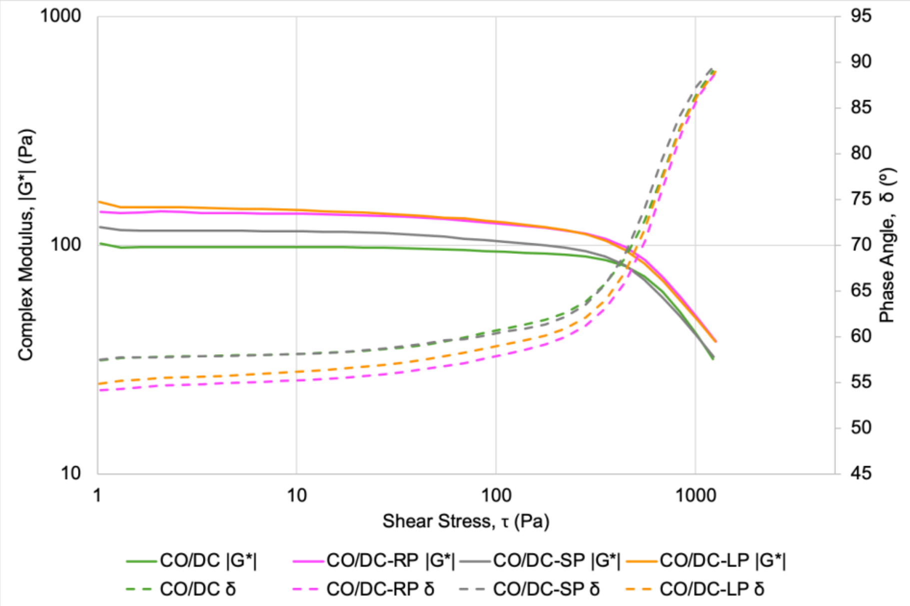


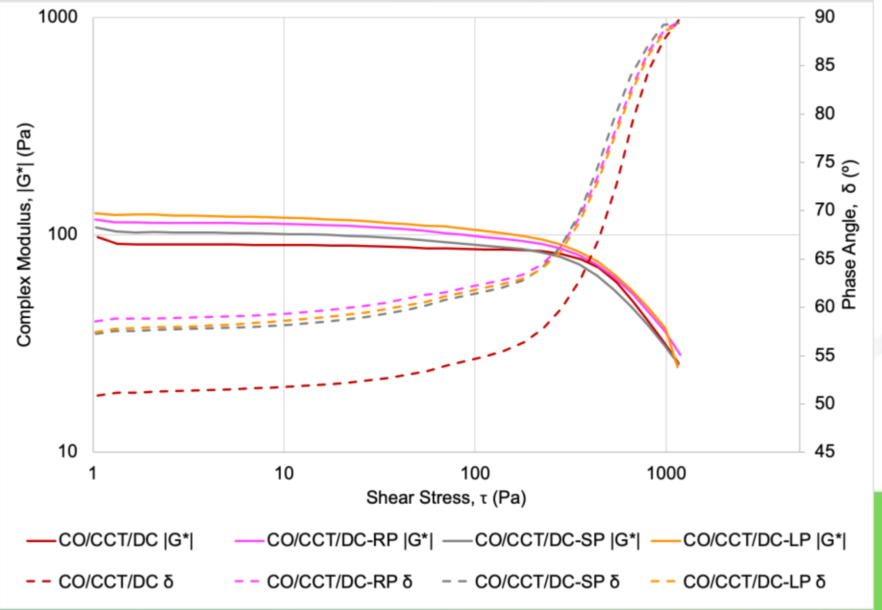
Results – Lip Gloss Viscoelasticity

- Pigments increased stiffness.
- Elasticity remained similar, except in the sample with the least CO, where pigments may have disrupted polymer entanglement.









Results – Lip Gloss Viscoelasticity

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

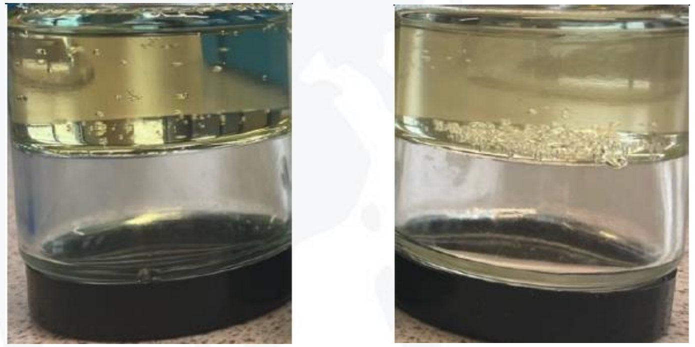
- The firmness, stickiness and work of shear of CO, CO/CTT and CO/DC were higher than LGB.
- Reducing CO lowered all textural parameters.
- Pigments did not change textural parameters, except CO, whose firmness and work of shear decreased.
- CO/CCT/DC was similar to LGB.

Conclusions

- **EC-CO systems have shown considerable potential as bases for lip gloss applications.**
- CO determines stability, viscosity, stiffness and viscoelasticity of EC-oil systems – **increases EC solubility**.
- CCT and DC reduced viscosity and rigidity, and increased elasticity, which could benefit storage stability.
- Pigments did not change viscosity, but slightly increased stiffness.
- Mixing CO with other oils is key for obtaining lip gloss bases with acceptable textural properties.

Future Perspectives

- EC oleogels (solid at rest) can be prepared by **static cooling**. Cooling rate may be important (Davidovich-Pinhas et al., 2015a).
- The structure of EC oleogels can be restored by **annealing** (Davidovich-Pinhas et al., 2015b).
- Ingredients to be tested for lip gloss applications:
  - Film-formers.
  - Volatile ingredients.



Static cooling
CO (left) and CCT (right) oleogels

Davidovich-Pinhas, M., Barbut, S. and Marangoni, A.G. (2015a) 'The gelation of oil using ethyl cellulose', *Carbohydrate Polymers,* 117, pp. 869–878. doi: 10.1016/j.carbpol.2014.10.035.

Davidovich-Pinhas, M., Gravelle, A.J., Barbut, S. and Marangoni, A.G. (2015b) 'Temperature effects on the gelation of ethylcellulose oleogels', Food Hydrocolloids, 46, pp. 76–83. doi: 10.1016/j.foodhyd.2014.12.030.

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Thank you

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