

## Development of a continuous lab process for the production of emulsion based lipstick

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The industrial production of lipstick is currently limited to batch processes. The introduction of a continuous process, such as a scraped surface heat exchanger (SSHE) is a necessity to improve production efficiency. A SSHE has been used for years in the food industry in combination with a pin stirrer (PS) to produce, for example, margarine. SSHE allows a fine control of the heat transfer resulting in good control of crystal formation, while the use of a PS allows good control over structuring the wax crystal network.

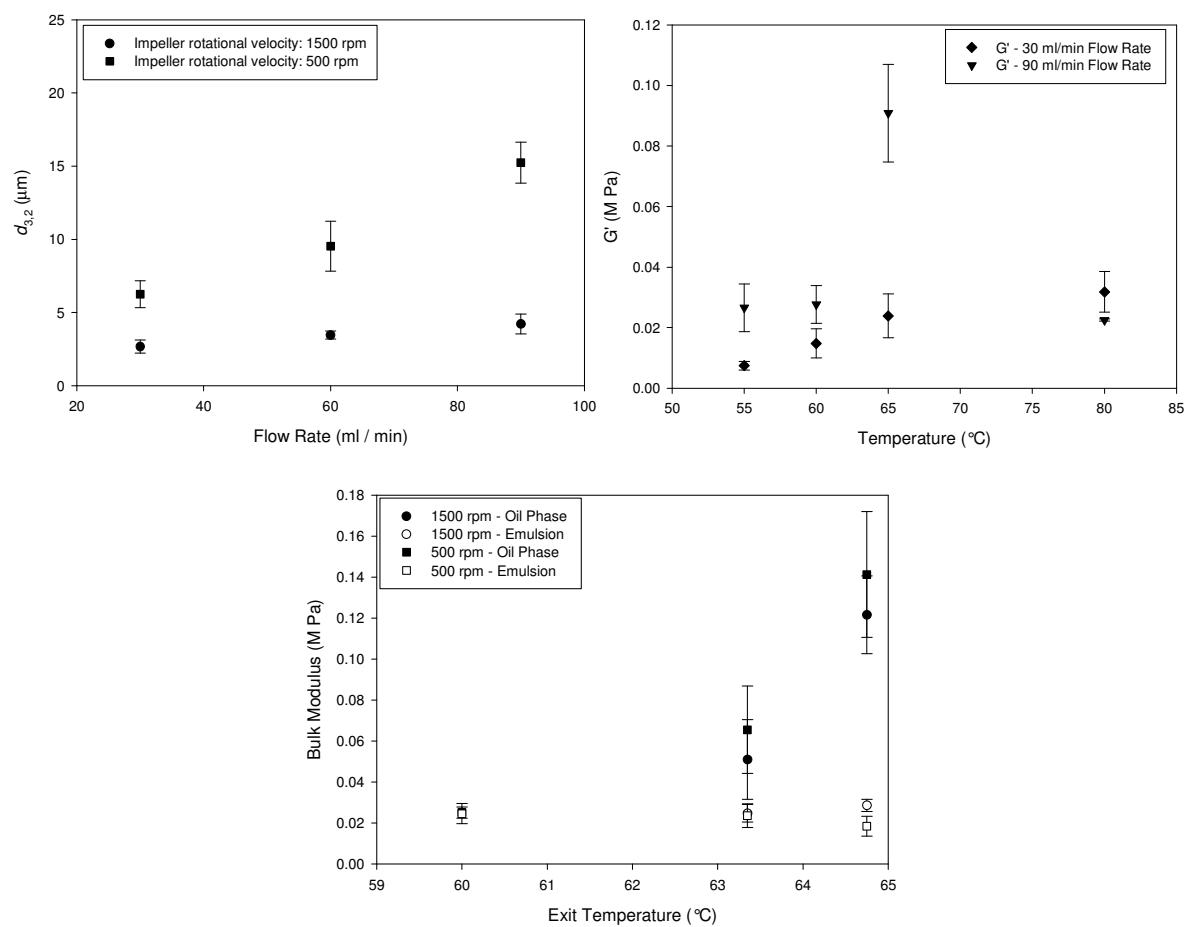
The aim of this work was to investigate the use of a SSHE/PS process in producing water-in-oil emulsion-based lipsticks. The effects of both units, SSHE and PS, on the emulsion-based lipstick physical properties (droplet size and rheology) and material properties (elastic and bulk modulus) were studied. A coarse emulsion was initially passed through the SSHE at various flow rates (30, 60, 90 ml/min), jacket temperatures (55, 60, 65, 80°C, chosen below and above the wax crystallisation temperature  $T_c$ ) and impeller speeds (500 and 1500 rpm) in order to determine the optimum SSHE parameters. Then, the emulsion formed in the SSHE unit (at 60 ml/min with either impeller speeds of 500 and 1500 rpm) was passed through a PS at various jacket temperatures (55, 60, 65, 80°C) and different impeller speeds (500, 1000, 1500 rpm) in order to investigate how the PS unit affects the emulsion-based lipstick structure (droplet size, elastic modulus and bulk modulus).

The results obtained show that the emulsion droplet size increases by increasing the flow rate and the jacket temperature through the SSHE unit and decreasing impeller velocity, in the range from 3 $\mu$ m to 17 $\mu$ m (**Figure 1a**). The residence time within the SSHE chamber is reduced by increasing the flow rate which gives less time for the impeller to disrupt the water droplets (regardless of the temperature and impeller speed). The presence of crystals at the interface as well as in the bulk was found to be crucial for reducing droplet size; increasing the temperature above  $T_c$ , fewer to no crystals are formed in the bulk resulting in the formation of bigger droplets. Rheological analysis (**Figure 1b**) shows that the highest elastic modulus is achieved at a jacket temperature of 65 °C and the highest flow rate. At this temperature, crystallisation occurs in the SSHE chamber and small crystals are formed due to the impeller. However, because 65°C is slightly higher than  $T_c$ , it is likely that the number of crystals formed is not optimum, some crystals having being melted in the chamber. Then, during post-production cooling, bigger crystals are also formed. The combination of both small and large crystals in the bulk phase allows greater connections, resulting in a stronger wax network. In comparison, at low temperatures and high temperature, either crystallisation occurs in the SSHE or post production, respectively, allowing either small or large crystals to be formed resulting in a weaker network being formed. This effect can also be seen in compression testing, where the bulk modulus was calculated. In **Figure 1c**, the jacket temperature was maintained at 65 °C, thus allowing crystallisation to occur in the SSHE. At high flow rates, wax crystals are formed in both the SSHE and post-production, whereas at low flow rates the majority of crystals will be formed in the SSHE. It can be seen in **Figure 1c** that a combination of crystals results in the highest bulk modulus, as a stronger network has been formed. It is also showed that introduction of water droplets (10%) rapidly reduces the bulk modulus as defects have been introduced to

the microstructure. It is also interesting to note that the impeller velocity, i.e. the shear induced within the SSHE chamber does not affect the bulk modulus.

The addition of PS to the process has no (or only little) effect on the droplet size if a high rotational velocity is used for the SSHE unit. However the elastic modulus decreases (from 0.2 M Pa – 0.1 M Pa) as the rotational velocity of the impeller in the PS increases (for  $T \leq 65^\circ\text{C}$ ). This is due to network disruption caused by the pin stirrer. It can also be observed that the elastic modulus increases (0.2 M Pa – 0.4 M Pa) with the jacket temperature. At higher temperatures the crystal network that was formed in the SSHE chamber tends to melt within the PS unit and then re-crystallise during post-production, resulting in a stronger network being formed.

In conclusion, emulsion-based lipstick properties such as droplet size and hardness can be controlled by adjusting the processing parameters of a SSHE. The introduction of a PS into the process results in either an increase or decrease of the elastic modulus of the emulsion, depending on the processing conditions. The great control achieved in using a SSHE/PS continuous process has the potential to have a great impact of the products produced in the cosmetic industry.



**Figure 1 – Effect of SSHE on emulsion properties. (a) Average droplet size ( $d_{3,2}$ ) as a function of flow with varying impeller rotational velocity when jacket temperature is  $60^\circ\text{C}$ , (b) Elastic and viscous modulus as a function of temperature with varying flow rates when impeller rotational speed is 1500 rpm and (c) bulk modulus as a function of flow rate with varying impeller speeds when jacket temperature is  $65^\circ\text{C}$ .**