Meadowfoam seed oil as a natural dispersing agent for colorants in lipstick

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Abstract
Objective: Green and sustainable trends are growing and with that the demand for naturally derived ingredients is rising. Dispersing agents are essential components of lipsticks due to their ability to wet pigment particles, reduce agglomerates and prevent re-agglomeration by stabilizing pigment particles. In this study, meadowfoam seed oil was evaluated as a pigment-dispersing agent for lipsticks and compared with castor oil and octyldodecanol.

Methods: Dispersions of Red 7 Lake were formulated with 20, 30 and 40% solid content using castor oil, octyldodecanol or meadowfoam seed oil. Particle size, viscosity, spreadability, wetting, oil absorption and colour were measured. Four of the nine dispersions were then formulated into lipsticks, including all the 30% pigment dispersions and the 40% dispersion with meadowfoam seed oil. Lipsticks were tested for hardness, pay-off, friction, rheology, colour and stability for 4 weeks.

Results: Average particle size was between 6 and 9 µm across the dispersions. The castor oil dispersions were more viscous, stickier and harder to spread than the other dispersions. The wetting contact angle was very low for all three dispersing agents, indicating that all of the oils wet the pigment well. The lipsticks varied in hardness, as expected, based on differences in the viscosity of the dispersing agents, and oil absorption of the powder. Red 7 Lake absorbed the highest amount of castor oil, which contributed to higher stick hardness. The castor oil lipstick and the meadowfoam seed oil lipstick containing 40% pigment were the hardest and most elastic. The octyldodecanol lipstick was the softest. Friction was the lowest for the meadowfoam seed oil lipstick containing 40% pigment, while pay-off was the highest for the octyldodecanol lipstick. The colour of the lipsticks as a stick and after being spread on paper was very similar.

Conclusion: While the chemical composition and physicochemical properties of the dispersing agents were different, all three dispersing agents studied formed dispersions and lipsticks with appropriate characteristics. Meadowfoam seed oil's performance was qualitatively and quantitatively similar to castor oil and octyldodecanol. By modifying the amount of pigment and dispersing agent used, lipsticks that have similar characteristics to commercial products can be formulated.
INTRODUCTION

Lipstick has been an iconic makeup product for centuries. Pigments are essential to lipsticks as they provide colour to the product and consumers’ lips. Cosmetic pigments are typically supplied as powders, which typically consists of aggregates and agglomerates with a smaller amount of primary particles. Most commonly, formulators use pigment dispersion instead of powder pigments due to multiple advantages, including full development of colour during the dispersion process, and a smooth consistency in which the aggregates are broken down into smaller units and/or primary particles [1]. Lipstick pigment dispersions consist of powder pigments homogenously dispersed in a carrier liquid (also referred
MEADOWFOAM SEED OIL AS A DISPERSING AGENT

Since pigments are insoluble in the dispersing agent, stabilization of the particles is essential [2]. Dispersing agents play a critical part in the stability and quality of the pigment dispersion by creating a barrier between the particles and, therefore, dispersion by improving the separation of particles in the dispersion and reducing settling and/or agglomeration [3–5]. The three stages of the particle dispersion process consist of wetting, particle separation/dispersion (particle size reduction) and suspension stabilization [6–7]. Most typically, dispersing agents for lipstick pigments include oils [8]. When selecting carrier oils, key considerations usually include viscosity, refractive index and price [9].

Castor oil is a natural vegetable oil derived from the seeds of the castor plant and is very commonly used as a pigment-dispersing agent for lipsticks [10]. Castor oil consists of triglycerides of different fatty acids (Figure 1): 90% ricinoleic acid (C18), 3%–4% oleic acid (C18) and 3%–4% linoleic acid (C18). Castor oil is a clear, slightly yellowish, viscous liquid [11] and is used in commercially available dispersions [12]. Ricinoleic acid, the main component of castor oil, has surfactant-like properties, which can help to stabilize pigments and contribute to castor oil's excellent dispersing properties.

Octyldodecanol is a branched fatty alcohol (C20, Figure 1) produced from natural fats and oils by reduction in the fatty acid grouping to the hydroxyl function [13–14]. Octyldodecanol is a clear, colourless, low-viscosity liquid, and it is also used commercially as a dispersing agent for makeup pigments [15]. In certain dispersions, octyldodecanol is mixed with surfactants, such as sorbitan oleate, to help wet the pigment and stabilize the dispersion.

Meadowfoam seed oil is a triglyceride composed of approximately 95% fatty acids with chain lengths of 20 carbons or more (Figure 1). Meadowfoam seed oil is a clear, light coloured, low-viscosity liquid with a high level of oxidative stability [16].

Most cosmetic fatty acids have a chain length of 18 or less carbons with unsaturated double bonds at the Δ9 and Δ12 position—for example the fatty acids in castor oil. The chemistry of octyldodecanol is different because it is a branched fatty alcohol. Meadowfoam seed oil's chemistry is also different from the previous two emollients as meadowfoam seed oil consist of C20 and C22 fatty acids, and the unsaturated double bonds are at the Δ5 and Δ13 position. This chemistry provides meadowfoam seed oil with a higher oxidative stability than castor oil [17,18], which is helpful in hot processing, such as lipstick moulding.

In this study, three dispersing agents, that is castor oil, octyldodecanol and meadowfoam seed oil, were evaluated for their ability to disperse and stabilize Red 7 Lake powder for lipsticks. First, dispersions with three different pigment contents were created and evaluated; then, four different lipsticks were formulated and tested qualitatively and quantitatively.

MATERIAL AND METHODS

Materials

The pigment was Red 7 Lake powder (INCI name: Red 7 Lake, Making Cosmetics, Redmond, WA). The pigment was a calcium lake with a dye content of 50%–55% [19].
The dispersing agents were castor oil (INCI name: Ricinus Communis (Castor) Seed Oil, Making Cosmetics, Redmond, WA), octyldodecanol (INCI name: Octyldodecanol, Making Cosmetics, Redmond, WA) and meadowfoam seed oil (INCI name: Limnanthes Alba (Meadowfoam) Seed Oil, Natural Plant Products, Salem, OR).

Microcrystalline wax, ozokerite, candelilla wax, ethylhexyl palmitate, caprylic/capric triglyceride, shea butter, castor oil, octyldodecanol and vitamin E were purchased from Making Cosmetics (Redmond, WA). All ingredients were of cosmetic grade. Three commercial lipsticks were tested in this study as controls, including Wet and Wild Silk Finish Lipstick in Dark Wine colour (‘C1’) purchased at a local store (Dollar General, Toledo, OH); L’Oréal Colour Riche Comfortable Creamy Matte Lipstick in Matte-ly in Love colour (‘C2’) purchased in online (Amazon.com); and Yves Saint Laurent Rouge Volupte Shine Lipstick Balm (‘C3’) purchased in online (Sephora.com).

Methods—Pigment dispersions

Formulation of dispersions

A total of nine dispersion (50 g each) were formulated using the three-roll mill (EXAKT Technologies, Inc., Oklahoma City, OK, Table 1). The appropriate amount of Red 7 lake powder and the oil was added to a stainless-steel bowl and mixed using a silicone spatula until the powder was wet. Then, this mixture was transferred to the three-roll mill. Each dispersion was milled in five cycles. Red 7 Lake has to be processed between four and five passes to reach maximum colour strength [20]. Cycles one and two had a ratio of the infeed to scraper gap ratios at 6:3 and 4:2, respectively. The third, fourth and fifth cycles had a ratio of 2:1. The first three cycles were run at speed 1, and the rest were run at speed 3.

| Dispersing agent     | Sample name | Solid content (w/w %) |
|----------------------|-------------|-----------------------|
| Castor oil           | C20         | 20                    |
|                      | C30         | 30                    |
|                      | C40         | 40                    |
| Octyldodecanol       | O20         | 20                    |
|                      | O30         | 30                    |
|                      | O40         | 40                    |
| Meadowfoam seed oil  | M20         | 20                    |
|                      | M30         | 30                    |
|                      | M40         | 40                    |

Particle size

Particle size was analysed using a Hegman gauge. About 3 g of each dispersion was placed at the top of the Hegman gauge and using a metal scraper the dispersion was pulled down at a constant speed. Streaks were then observed, and particle size was recorded. All nine dispersions were tested in triplicate.

On the same day, the dispersions were formulated, and particle size distribution was tested using a light microscope (AmScope T490 3MT, Irvine, California). The dispersion was diluted with the corresponding dispersing agent on a glass plate. Then, a pinhead amount of the diluted dispersion was placed on a microscope slide and covered with a cover slide. Images were taken at 10x magnifications (IS Capture software, Irvine, CA). The same steps were repeated for all nine dispersions. Particle size was analysed manually on 35 particles for each dispersion.

Rheology

On the same day, the dispersions were formulated, and the dispersions’ and neat oils’ rheology was tested using a dynamic hybrid rheometer (DHR-3, TA Instruments, New Castle, DE) with a 40 mm parallel plate geometry. A small amount (<3 g) of the dispersion was placed on the rheometer, and a gap of 500 μm was used. After trimming the sample, amplitude sweep, frequency sweep and flow sweep were run for all dispersions.

Firmness and stickiness

Firmness and stickiness were tested using the TA texture analyser (Texture Technologies Corp., Hamilton, MA) and

| TABLE 2 Lipstick formula | % (w/w) |
|--------------------------|---------|
| Ingredients              | C30, O30, M30 | M40 |
| Phase A                  |          |      |
| Microcrystalline wax     | 2.0      | 2    |
| Ozokerite                | 4.5      | 4.5  |
| Candelilla wax           | 15.0     | 15   |
| Ethylhexyl Palmitate     | 25.3     | 15.3 |
| Caprylic/Capric Triglyceride | 20.5     | 20.5 |
| Shea butter              | 2.5      | 2.5  |
| Phase B                  |          |      |
| Dispersion—oil varies    | 30.0     | 40.0 |
| Phase C                  |          |      |
| Vitamin E                | 0.2      | 0.2  |
a spreadability fixture 90° male cone probe. Test mode was set to compression. Pre-test, test and post-test speeds were set at 3 mm/s. Target mode was distance, and distance was 23 mm. Trigger type was set at pretravel, and trigger distance was set at 5 mm. Approximately 8 g of the dispersion was placed in the sample holder. Then, the sample was placed underneath the male cone, assigned a pre-set location at 5 mm. Each dispersion was tested three times.

Oil absorption

The oil absorption of Red 7 Lake for each dispersing agent was measured using the spatula rub-out method (ASTM D281-12 method). First, 1.00 g of the pigment was weighed and placed on a glass plate. Then, oil was added dropwise to the pigment and rubbed firmly with a spatula until a well-mixed paste with no particles is formed. The weight of the bottles was recorded before and after each test, and the difference was calculated to measure how much oil was used. Each oil was tested three times.

Contact angle

In order to measure how the dispersing oils wet Red 7 Lake powder, flat tablets were created from the Red 7 Lake powder using a Korsch EK0 eccentric tablet machine (Korsch GmbH, Berlin, Germany) and standard concave punches and die 0.9525 cm in diameter. The three dispersing agents, including castor oil, octyldodecanol and meadowfoam seed oil, were used in the test and a CAM-PLUS MICRO contact angle micrometre (Tantec, Inc., Schaumburg, IL, USA). The tablet was placed on a flat surface. Droplets of 80 μl of the emollient were pipetted onto the surface of the tablet, and images were taken 10 s after dropping the oil using a Keyence VHX-6000 Optical Microscope (Amscope, Irvine, California). The instrument software was used to measure the contact angle. Five tablets were used for each emollient.

Colour

The colour of all nine dispersions was tested using a CM-5 spectrophotometer (Konica Minolta, Chiyoda City, Tokyo, Japan). Measurement type was set to reflectance.

Stability

All nine dispersions werelabelled and kept in the laboratory at room temperature in plastic jars for 5 months and observed visually once a month for any signs of physical change.

Methods—Lipsticks

Formulation of lipsticks

The lipstick ingredients and their amounts were kept constant, and only the dispersion was changed between the different lipstick batches (Table 2). First, a new batch of each dispersion was made using the same steps mentioned above. Lipsticks were formulated by first heating phase A to 80°C. Then, phase B was added, and the mixture was taken off the heat. Phase C was then added and while still hot the mixture was poured into a metal lipstick mould. After the mixture settled, the sticks were topped off, and the mould was placed in a freezer (−18°C) for 15 min. Once the lipsticks were taken out of the freezer, they were inserted into plastic lipstick cases. Lipsticks were kept in stability cabinets (25 and 40°C) to be tested on day 1, week 2 and week 4.

|        | Firmness (g) | Stickiness (g) | Viscosity (Pa. s) at 100 s⁻¹ | Colour |        |
|--------|--------------|----------------|-----------------------------|--------|--------|
|        |              |                |                             | L*     | a*     | b*     |
| C20    | 515 ± 7      | −421 ± 1       | 1.8                         | 31     | 25     | 11     |
| C30    | 1255 ± 18    | −964 ± 16      | 1.7                         | 32     | 21     | 9      |
| C40    | 3226 ± 65    | −2477 ± 36     | 12.0                        | 28     | 28     | 12     |
| O20    | 40 ± 2       | −22 ± 2        | 0.5                         | 28     | 27     | 10     |
| O30    | 129 ± 5      | −84 ± 4        | 1.0                         | 32     | 24     | 9      |
| O40    | 472 ± 5      | −336 ± 2       | 2.3                         | 30     | 26     | 10     |
| M20    | 51 ± 0       | −29 ± 0        | 0.3                         | 30     | 24     | 9      |
| M30    | 151 ± 2      | −96 ± 3        | 0.7                         | 28     | 26     | 9      |
| M40    | 385 ± 5      | −270 ± 4       | 3.7                         | 32     | 21     | 9      |

Abbreviations: C, castor oil; O, octyldodecanol; M, meadowfoam seed oil, the numbers after the letter refer to the solid content (w/w %).
 Needle penetration test

The method described in our previous paper was followed [21].

Pay-off and friction

The pay-off to fabric and friction method described in our previous paper was followed here [21].

Rheology

Twenty-four hours after the lipsticks were formulated, their complex rheology was tested using the rheometer mentioned above. The lipsticks were cut into discs with a height of 1–2 mm. Gap size was set to 500 µm, and using an 8 mm plate geometry, amplitude and frequency sweeps were run at temperatures from 25 to 45°C in triplicate.

Colour

A small amount of each stick was melted in a 50 g glass beaker and allowed to solidify and form a disc. Colour was also measured after each lipstick was applied to printer paper in four layers (moving the lipstick up–down two times) and spread evenly using a fingertip. The colour of the printer paper pieces covered with the lipstick was analysed using the colorimeter mentioned above.

Stability

The stability of lipsticks was monitored at three temperatures, including room temperature (25°C) and two elevated temperatures (40 and 45°C) in stability cabinets for 4 weeks. Samples were checked visually and tested instrumentally for hardness at day 1, week 2 and week 4.

Data analysis

Differences in the dispersion firmness, stickiness, particle size, and oil absorption and lipstick hardness, friction, pay-off and rheology were evaluated using one-way ANOVA followed by Tukey's multiple comparison test using SPSS Statistics 21 software (IBM Armonk, NY). A $p$ value less than 0.05 was taken as the minimal degree of statistical significance.

Results—Pigment dispersions

Particle size

All three castor oil dispersions spread evenly on the plate, and each castor oil dispersion's particle size was below 10 µm. These dispersions were very viscous compared with the other dispersions. O20 and M20 dispersions did not spread well on the plate (Figure S1). The wetting contact angle measurements showed that all three oils wet the pigments very well. Therefore, we believe the reason for the poor spreading of O20 and M20 was the very low viscosity of the dispersions. The particle size of all other O and M dispersions was below 10 µm when analysed with the Hegman gauge, which means that all dispersing agents were effective at de-agglomerating the pigment.

The average particle size based on the microscopic images was between 6.2 and 8.6 µm, which was consistent with the values found using the Hegman gauge (Figure S2). The C30 particle size was significantly higher than O30 and M40 ($p < 0.05$). The particle size distribution was homogenous, which proves that the dispersing agents were effective at de-agglomerating the pigments during the milling process.

Firmness and stickiness

With the texture analysis, the firmness and stickiness of the samples were compared, which mainly depended on the dispersing oil. Given the higher viscosity and well-known sticky nature of castor oil, the castor oil samples were by far the firmest and stickiest at all three solid contents compared with the other two dispersing agents (Table 3). C30 was significantly firmer and stickier than O30, M30 and M40 ($p < 0.05$). O and M at the same solid contents were similar in terms of both of these properties.

Rheology

The viscosity of castor oil was the highest, while the viscosity of octyldodecanol and meadowfoam oil was 7–14 times

| Dispersing agent     | Viscosity (Pa. s) at 10 s⁻¹ | Oil absorption (g oil/1 g of pigment) | Contact angle (°) |
|----------------------|-----------------------------|--------------------------------------|-------------------|
| Castor oil           | 0.69 ± 0.005                | 1.54 ± 0.10                          | 14 ± 2            |
| Octyldodecanol       | 0.05 ± 0.003                | 1.11 ± 0.10                          | 9 ± 1             |
| Meadowfoam seed oil  | 0.08 ± 0.002                | 1.05 ± 0.10                          | 7 ± 1             |
lower (Table 4). As a result, the castor oil dispersions had the highest viscosity, while the viscosity of the octyldodecanol and meadowfoam seed oil dispersions was similar (Table 3). While octyldodecanol and meadowfoam seed oil had lower viscosities, no issues were observed during the milling process. The octyldodecanol and meadowfoam seed oil dispersions adhered to the rolls and did not drip between the rolls onto the collection pan. The storage modulus (G’) was higher than the loss modulus (G”) for all nine dispersions.

Oil absorption

Red 7 Lake pigment absorbed a greater amount of castor oil compared with the rest of the oils (Table 4). Octyldodecanol and meadowfoam seed oil had comparable averages, but overall, the pigment absorbed less meadowfoam seed oil.

Contact angle

All three oils wet the surface of the Red 7 Lake tablets very well, with meadowfoam seed oil having the lowest contact angle of the three dispersing agents (Table 4). The lower the contact angle, the better the surface wetting. Thus, meadowfoam seed oil wet the surface better than the other two oils.

Colour

All nine dispersions looked very similar by naked eye, and even with the colorimeter, the colours were very similar

| Colour of lipsticks in beaker | Colour of lipsticks on printer paper |
|------------------------------|-------------------------------------|
| L*    a*    b*                | L*    a*    b*                        |
| C30   40.81 54.88 16.27      | C30   20.70 37.45 21.50               |
| O30   40.29 55.89 15.06      | O30   18.53 40.94 27.25               |
| M30   42.50 57.36 8.67       | M30   17.50 40.16 26.06               |
| M40   40.73 55.84 13.84      | M40   18.00 41.58 27.62               |

FIGURE 2  Hardness of lipsticks on day 1 measured with the needle penetration method (average and SD, n = 5). Abbreviations: C1: commercial 1, C2: commercial 2, C3: commercial 3; C: castor oil, O: octyldodecanol, M: meadowfoam seed oil, the numbers after the letter refer to the solid content (w/w %)

FIGURE 3  Friction of lipsticks on day 1 at 25°C

FIGURE 4  A. Lipsticks formulated in this study. B. Lipstick applied to printer paper

TABLE 5  Colour of our lipsticks in beaker and on printer paper
The Red 7 Lake powder had about the same lightness, but it was redder and yellower ($L^* = 32$, $a^* = 45$ and $b^* = 19$). Some differences for Red 7 Lake were expected when comparing a powder with dispersions.

**Stability**

None of the 9 dispersions showed separation, that is had any oil layer on the surface. All the 20% solid content dispersions remained thin and did not change over time. Dispersions with 30% and 40% solid content became slightly thicker (qualitatively), but could be re-dispersed with mixing. During microscopic evaluation, the particle size of the dispersions slightly increased over time (Figure S3). The average particle size based on the microscopic images was between 7.7 and 13.9 µm. It should be noted that no wetting agents were used in this study, which, however, are known to aid in stabilizing the pigment particles and preventing agglomeration [22]. The effect of wetting agents on these dispersions could be explored in the future.

**Results—Part II: Lipsticks**

**Needle penetration test**

The hardness of the lipsticks varied between 80 and 150 g among the lipsticks formulated, which were harder than the commercial lipsticks tested (Figure 2). Our goal was not to match the hardness of our lipsticks to commercial products, but to see how our lipsticks compared with commercial products. C30 was significantly harder than all other three lipsticks on day 1, week 2 and week 4 at 25°C ($p < 0.05$), which could be related to the viscosity and firmness of the castor oil dispersion. O30 was the softest, while M30 and M40 were similar, which means that the 10% increase in pigment content did not influence the hardness significantly. The hardness of the lipsticks remained consistent throughout the testing period at 25°C (Figure S4). At 40°C, the hardness of the lipsticks decreased by about 30%, while at 45°C, the hardness decreased by about 40% during the 4-week testing period.

**Pay-off and friction**

The average friction of the commercial lipsticks was smaller than the lipsticks formulated. The order of average friction was the following: C30 = O30 = M30 > M40 (Figure 3). Friction during the second cycle was lower for all lipsticks. Thus, the lipsticks became easier to spread after the first cycle. One explanation is that during cycle 1, the lipstick moves across the fabric, while in cycle 2, the fabric is already coated with one layer of the lipstick, making it glide easier.

As shown in the hardness results, O30 was the softest lipstick. The pay-off results were in correlation with this observation as O30 had the highest amount of pay-off to fabric (70 ± 10 mg). C30, M30 and M40 had a similar amount transferred to fabric, 55 ± 5; 55 ± 5; and 50 ± 1 mg, respectively, at 25°C.

**Colour**

All lipsticks seemed very similar by naked eyes (Figure 4a). The same conclusion can be drawn for the colour on printer paper (Figure 4b). When tested with the colorimeter, the numbers were similar as well. C30 was the lightest, less red and less yellow on paper compared with the other lipsticks; however, it should be noted that these differences were insignificant (Table 5).

**Rheology**

The commercial lipsticks and our O30, M30 and M40 lipsticks had similar elastic moduli (Figure 5). At 25°C, the moduli were between $10^6$ and $10^7$ Pa. C30 and M40 had a higher storage modulus than other lipsticks tested at all temperatures. As discussed above, C30 had the highest hardness value, while M40 had an additional 10% pigment. These characteristics made the lipsticks harder and more elastic—these characteristics could be optimized if needed by adjusting the wax/oil ratio.
CONCLUSION

Meadowfoam seed oil was used as a pigment dispersant for lipsticks and was compared with common dispersing agents, castor oil (C) and octyldodecanol (O).

All three dispersing agents were effective at de-agglomerating the Red 7 Lake powder and yielded stable dispersions. The castor oil dispersions were the most viscous and stickiest at all three solid contents, while the octyldodecanol and meadowfoam seed oil dispersions were very similar in the characteristics measured.

Overall, the castor oil lipstick was found to be the hardest. The high hardness was a result of the viscosity of castor oil and the oil absorption of the pigment. Red 7 Lake absorbed more castor oil than octyldodecanol or meadowfoam seed oil, and therefore, more oil was used to wet and stabilize the pigment than in the case of the other two dispersing agents. The octyldodecanol lipstick was the softest and had the highest pay-off. The 30% meadowfoam seed oil lipstick were in between the other two lipsticks, softer than the castor oil lipsticks, but harder than the octyldodecanol lipsticks. While the 40% meadowfoam seed oil lipsticks were softer than the castor oil lipsticks, they had the lowest friction and very similar pay-off and rheological properties as the castor oil lipsticks. Having lower friction translates to better glide, which can enhance the product application experience. All lipsticks were stable at 25°C, and moderate sweating was observed at 40 and 45°C.

The findings of this research study indicate that it is possible to create lipsticks with meadowfoam seed oil that resemble castor oil lipsticks or commercial lipsticks—with proper selection of the amount and type of ingredients, very similar products can be created. Specifically, meadowfoam seed oil proved to be a viable dispersing agent, forming dispersions and lipsticks qualitatively and quantitatively similar to castor oil and octyldodecanol.

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SUPPORTING INFORMATION
Additional supporting information may be found online in the Supporting Information section.

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