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"A Holistic Approach to Maximize Pigment Performance in Color Cosmetics and Sunscreens: from Wetting, Dispersing, to Stabilization"

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1. Introduction

Inorganic pigments are a vital raw material category in color cosmetics and sunscreens. The end-performance of color cosmetics and inorganic UV filter-based sunscreens is mainly determined by the performance of pigments in the formulations. To maximize the performance of pigments, it is important to ensure a good dispersing environment, where raw materials and process are involved. This is necessary to produce good appearance on application and fulfill the functions as a makeup or sunscreen product.

Metal oxides-based pigments (yellow, red, and titanium dioxide) were studied. The process of pigment dispersion preparation involves three stages: wetting, dispersing, and stabilizing [1-2]. Ingredients to facilitate the pigment dispersion at each stage were investigated separately. High shear mixers were used to optimize dispersing. In the wetting stage, Daniel wet & flow point methods were used to assess the performance of emollient esters as carrier for pigments [3-4]. The particle size and fineness of the dispersions were determined by the fineness gauge and digital microscope. Colorimeter was used to gauge the color strength. The overall performance of pigments in medium was thus defined.

The Daniel wet & flow points for different color pigments are different depending on the pigment type and emollient esters. The wetting of pigments can be greatly improved with esters that can displace air from pigment surfaces and separate the pigment flocculates. Methyl Glucose Dioleate can effectively reduce the interfacial tension and improve initial formation of the liquid-solid interfaces. High shear forces applied can efficiently break down the particle size and further separate the agglomerates. Surfactants like hyperdispersant polymer with anchoring groups can absorb on the particle and impart steric hindrance among particles, preventing settling and re-agglomeration of pigments.

From wetting to stabilization, with each stage optimized, better color uniformity and greater color intensity can be observed. Hence, better performance with more economical solutions can be achieved. This study serves as a guidance on optimizing pigment dispersion to accomplish high performance in color and sunscreen applications.

2. Materials and Methods

Materials

Pigments: CI77491 (and) Triethoxycaprylysilane (Red), CI 77891 (and) Triethoxycaprylysilane (White), CI 77492 (and) Triethoxycaprylysilane (Yellow), Titanium dioxide (and) Triethoxycaprylylsilane (GZ0987), Titanium Dioxide (and) Aluminum Hydroxide (and) Stearic Acid (Al-TiO2) were used as supplied.

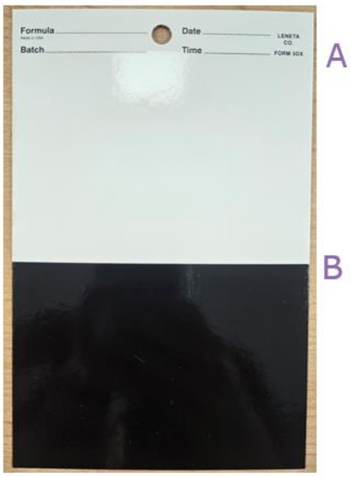
Emollients: Cyclopentasiloxane & Cyclohexasiloxane (PMX345), Cocoyl Adipic Acid/Trimethylolpropane Copolymer (CATC), Neopentyl Glycol Diethylhexanoate (NGDO), Isostearyl Isostearate (1818), Glyceryl Isostearate (GMIS), Diisostearyl Malate (DISM), Isopropyl Isostearate (318), Propylene Glycol Laurate (PGML), Lauryl Lactate (LL) were used as supplied.

Surfactants: Methyl Glucose Dioleate (DO), Polyhydroxystearic Acid (and) Neopentyl Glycol Diethylhexanoate (MS-1), were used as supplied.

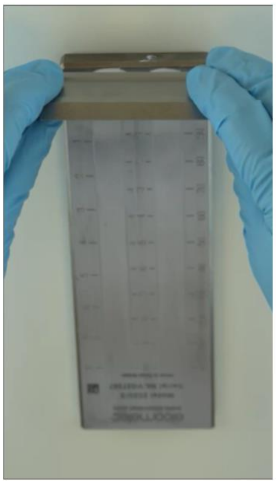
Methods

Daniel's wet point: 5 g of pigment is added into a 50 ml beaker. Emollient is slowly added and mixed with the pigment to form a solid ball-like structure.

Daniel's flow point: More emollient is slowly added and mixed with the pigment till the ball-like structure transfers into a liquid. 1 g of the sample (dispersed pigment in emollient) is then placed onto a paper which will be tilted at a 90-degree angle to let the sample flow from point A to B (see **Figure 1A**). The duration of sample flow from point A to B will be measured. For the flow point, the duration should be 20 ± 2 seconds. If the sample takes less than 18 seconds or longer than 22 seconds, it means that the flow point is missed or not reached.



(A)



(B)

Figure 1. (A) Test of Daniel's flow point on the paper card. (B) Pigment dispersion on fineness gauge.

Dispersion test (fineness gauge): Sample (30% pigment and 70% emollient) is homogenized at 10,000 rpm for 2 min with a total of 50 g, except for 318 as due to the low viscosity nature it is homogenized at 5,000 rpm for 3 min. Sample is added onto one end of the fineness gauge, using the scrape sample is pulled down from top to bottom using a scraper in one smooth motion (see **Figure 1B**).

Dispersion test (microscope): Sample (30% pigment with 70% emollient) after homogenization is added onto a glass slide and checked with digital microscope at 200x, 500x, 1000x magnification at room temperature at day 0.

Color intensity test: Pigment dispersion sample (30% pigment with 70% emollient) after homogenization is added into a plastic cuvette, using the colorimeter to test 4 sides of the cuvette to get an average color intensity value.

Stability test: Pigment dispersion samples are added into glass vials and put into centrifuge to test at different rpm and time.

Viscosity measurement: viscosity of the pigment dispersions is determined by rotational viscosity tested at room temperature, 20 rpm.

3. Results

Daniel's wet & flow point for different emollients. The bar charts plotted with wet point and flow point of each emollient for each pigment showed the amount of emollient needed to wet 1 g pigment and to make 1 g pigment a flowable dispersion (see **Figure 2A, 2B,** and **2C**). It required the least amount of 1818 to disperse red, GMIS for yellow, and CATC for white pigments.

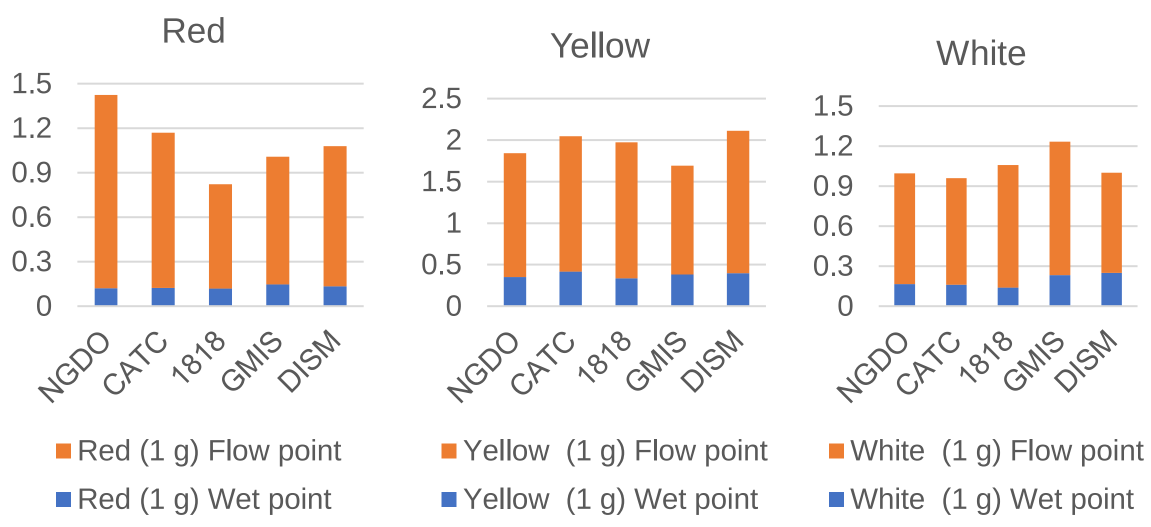
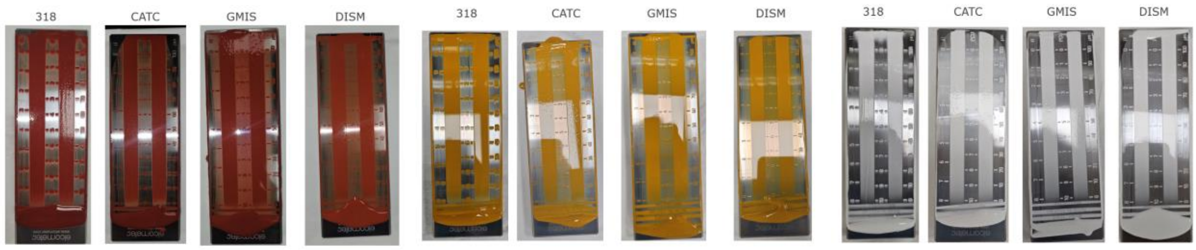


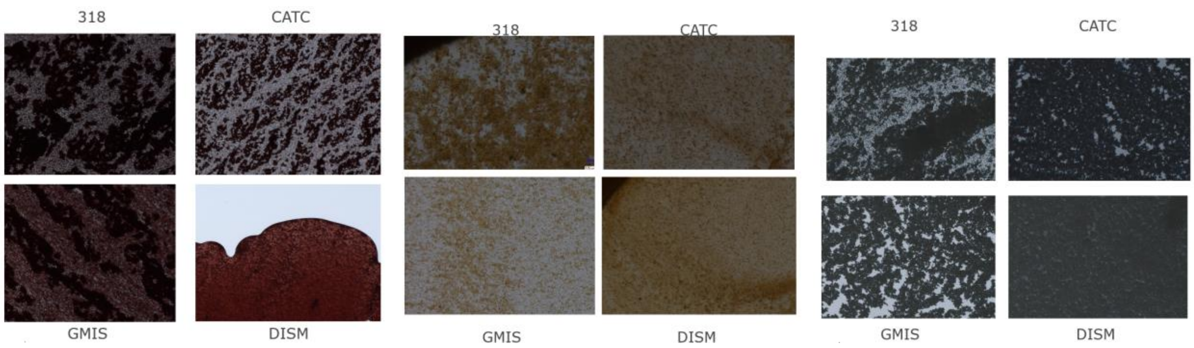
Figure 2. Daniel's wet & flow point of emollients for (A) red, (B) yellow, and (C) white.

Dispersion tests. The dispersion samples tested with fineness gauge and microscope showed similar results in terms of performance (see **Figure 3** & **4**). Emollients used to disperse red, yellow, and white pigment at 30% loading level all showed a homogenous dispersion after preparation. The particle size and size distribution are apparently different depending on the emollients used. DISM appears to be the best emollients in all three pigments.



(A) (B) (C)

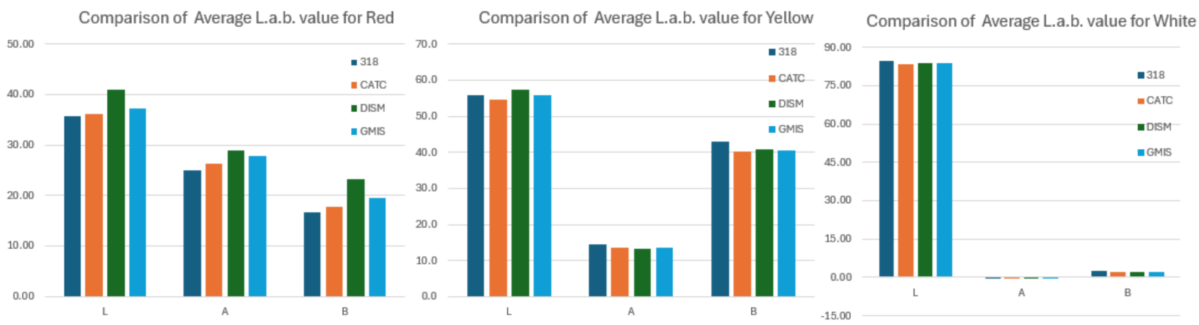
Figure 3. Dispersion tests using fineness gauge for (A) red, (B) yellow, and (C) white.



(A) (B) (C)

Figure 4. Dispersion tests using microscope for (A) red, (B) yellow, and (C) white.

Color intensity. The color intensity of the pigment dispersion depends on the dispersing emollients. As seen in **Figure 5**, for red, "a" value is the highest with DISM followed by GMIS, CATC, and 318. DISM based dispersion is the brightest, too. For yellow, the highest "b" value is given by 318, followed by DISM, GMIS, and CATC, whereas the DISM gave the brightest color. For white, 318 yielded the highest "L" value followed by GMIS, DISM, and CATC. However, 318 also provided the yellowest hue of the dispersion.



(A) (B) (C)

Figure 5. Color intensity measured by colorimeter. (A) red, (B) yellow, and (C) white.

Viscosity change. The viscosity of the pigment dispersions changed the most with 318 for all three types of color pigments.

Dispersion stability. When tested with Al-TiO2, 318 was the least stable, which only passed 1,000 rpm for 10 min. CATC, GMIS, and DISM based Al-TiO2 dispersions can pass 3,000 rpm for 30 min without visible sedimentation or phase separation.

Surfactants. Adding DO can significantly reduced the viscosity of dispersion (see **Figure 6**). The addition of MS-1 even lowered the viscosity and improved the dispersion (see **Figure 7**).

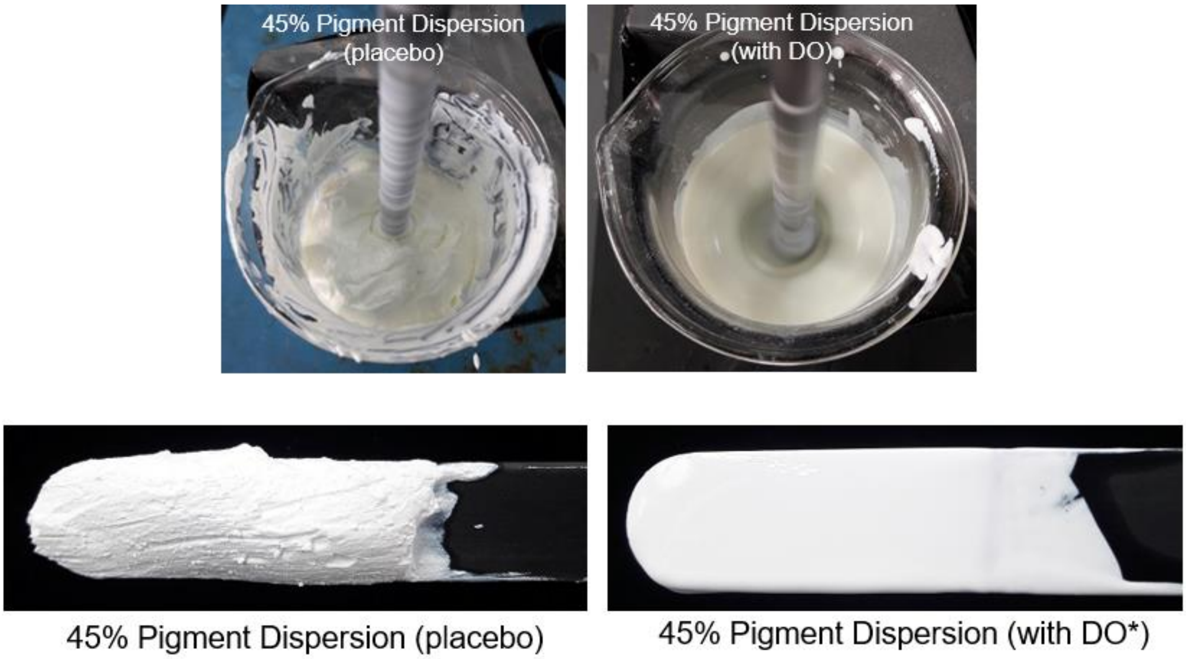


Figure 6. Pigment dispersions with and without DO.

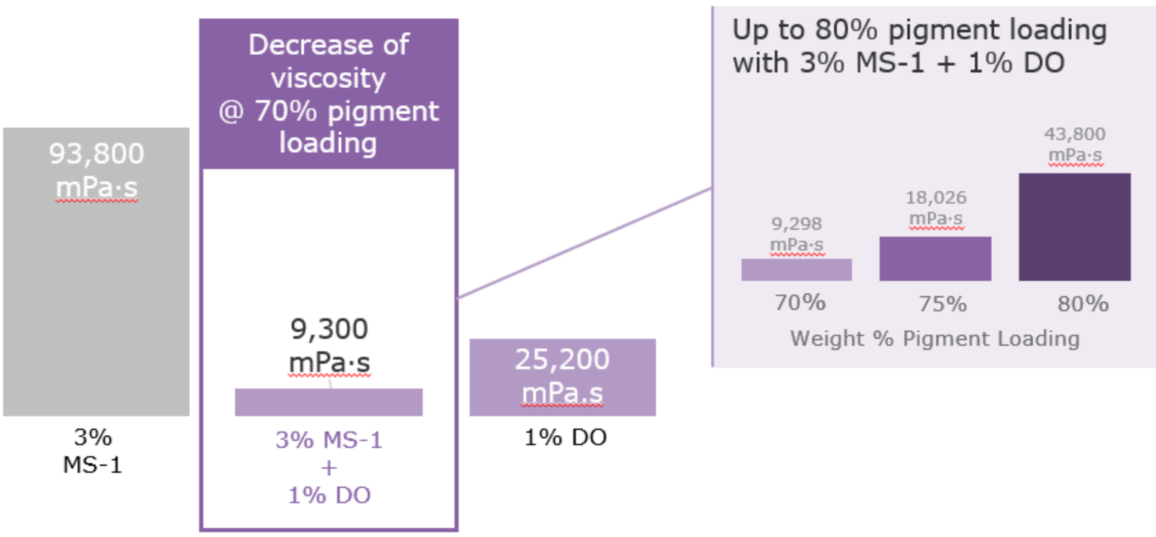


Figure 7. Impact of MS-1 and DO on white pigment dispersion.

4. Discussion

Dispersing pigment particles starts with dispersing medium, in this study, the emollients. The emollients work as dispersing agents to replace the air/solid interface with liquid/solid interface for further formulation process. The quality of the dispersion largely depends on the quality of the medium, the better the medium, the easier the formulation, the better the end-performance.

To evaluate the impact on the interface, wet points are critical to consider. The lower the wet point is, the minimum effort it needs to replace the air/solid interface. However, the wet point is often subjective and may not be significant to notice the difference. Hence, flow point is another parameter to assess the quality of a dispersing agent. For red pigment, 1818 yielded a relatively low wet point and flow point. For yellow, 318 is the best. CATC gave the lowest flow point for white, but the lowest wet point is from 1818.

Although DISM didn't outperform in the wet and flow point tests, it showed a very good dispersing property when tested with fineness gauge and color intensity. DISM is a relatively heavy emollient compared with the other emollients tested. The intrinsic viscosity of DISM is higher than the other emollients which may make the dispersion prepared take longer to flow.

DO is an effective wetting agent as surfactants can help lower the surface tension, making it easier for the liquid medium to flow into the air/solid spaces. On the other hand, MS-1 is designed to be a pigment dispersant, which can help disperse and stabilize pigment particles in emollients. The pigment anchoring and steric stabilization of MS-1 for pigment are illustrated in **Figure 8**.

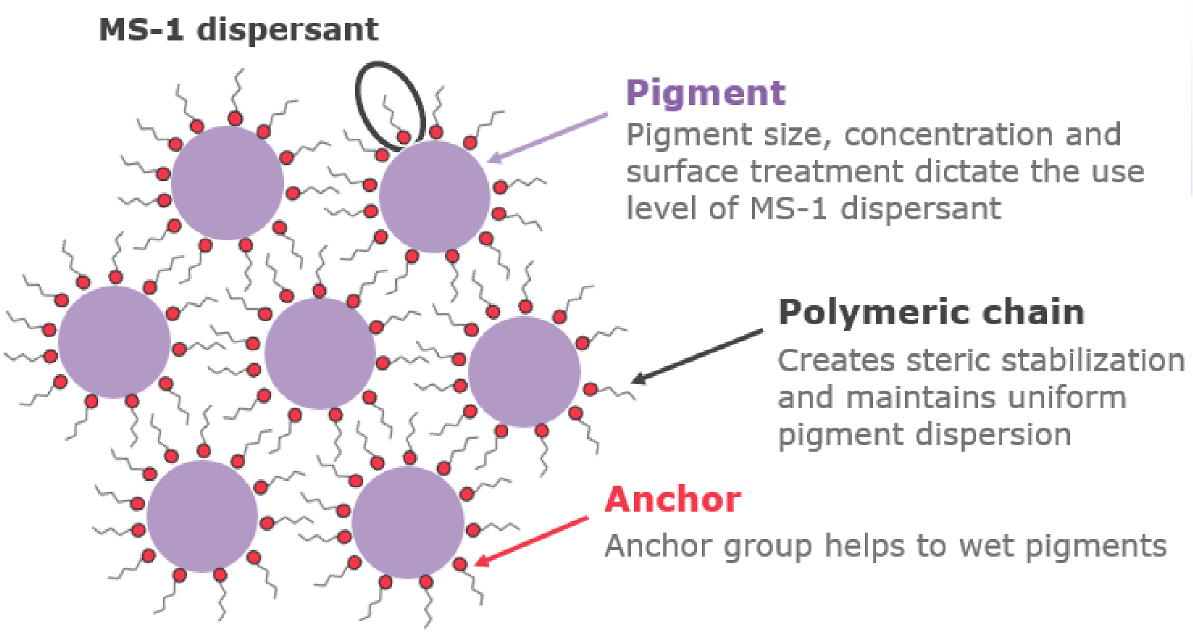


Figure 8. Mode of action of MS-1 for pigment dispersion.

5. Conclusion

The emollients tested for red, yellow, and white all showed different wet and flow points, suggesting optimization for each pigment may be necessary. Emollients can work efficiently to replace the air/solid interface with liquid/solid. High shear mixing is necessary to facilitate the dispersing process. Addition of surfactants can greatly impact on pigment dispersion is obvious, with great reduction in viscosity.

6. Acknowledgments

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7. Conflict of Interest Statement

NONE.

8. Reference

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