Stability of Non-Synthetic Pigments in Fully Formulated Film Coating Systems

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Introduction

Film coatings impart mechanical integrity, gloss, light and moisture protection to tablets. Inclusion of pigments enhances appearance, provides brand differentiation and improves acceptability of coated tablets to consumers. With the increase in consumer preferences for clean label ingredients in dietary supplements and nutritional products, this move is driving the need for titanium dioxide (TiO_2) free and non-synthetic (natural or naturally derived) pigments. However, coating formulations that include naturally derived pigments may create color stability challenges not typically encountered with synthetic pigments. The objective of this study was to evaluate on-tablet stability of ten naturally derived pigments included in Nutrafinish® TiO_2 Free Film Coatings.

Methods

To determine the in-use color stability of naturally derived pigments, an aqueous based Nutrafinish TiO_2 free film coating containing pigment, polymer (hypromellose), opacifier (calcium carbonate) and plasticizer was used. In this study the following 10 pigments were evaluated:

- Carmine
- Caramel
- Riboflavin
- Radish anthocyanins
- Spirulina extract
- Red beet powder
- Copper chlorophyll
- Safflower
- Curcumin
- Carbon black

The pigments were individually included in the film coating formulation at a consistent level. The film coating dispersions were prepared at 20% w/w solids content in water and applied onto 1.3 kg of placebo tablets (10 mm, round, biconvex) using a fully perforated 12″ side-vented coating pan (Labcoat II, O'Hara Technologies Inc., Canada). Tablets were packaged in HDPE bottles (100 cc without desiccant) for stability testing at 25°C/60% RH and 30°C/65% RH for 12 months. Additionally, open-dish stability studies at 25°C/60% RH, 30°C/65% RH and 40°C/75% RH were carried out for coatings containing carmine, radish anthocyanins, spirulina extract, red beet powder, copper chlorophyll and curcumin.

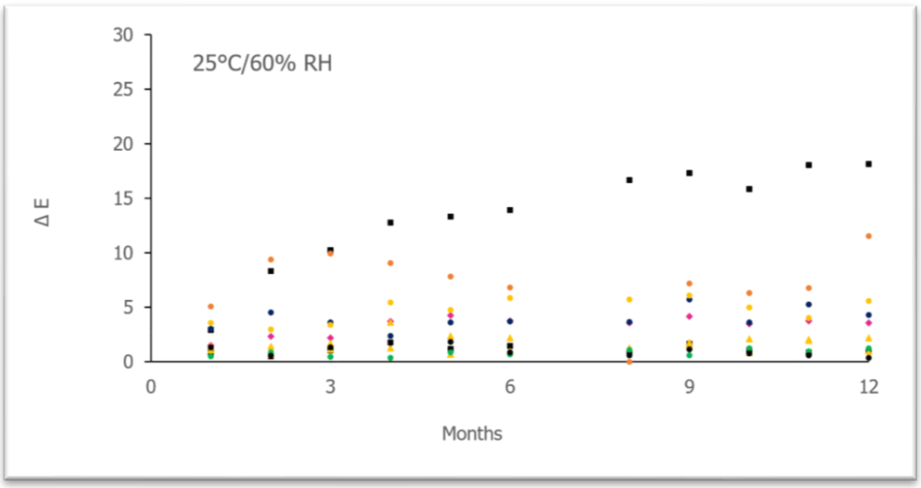
Coated tablets were removed at defined time points and visually assessed for color changes. Samples were also assessed using a Datacolor spectrophotometer to determine any change in color. Delta E (ΔE) was used to determine any color change using time zero tablet color as reference. ΔE is an equally weighted combination of the coordinate (L, a, b) differences between 2 samples in a 3-dimensional plane, ΔL is the lightness difference, Δa is the red/green difference and Δb is the yellow/blue difference.

Results

Figure 1 shows the change in Delta E (ΔE) values for all pigmented coatings at 25°C/60% RH and 30°C/ 65% RH conditions in HDPE bottles. Stable pigments such as copper chlorophyll showed minimal change in ΔE values for the entire duration of the study; whereas, radish anthocyanins showed higher ΔE values after 2 months under all storage conditions. Some pigments, red beet powder and radish anthocyanins extract faded more quickly at 30°C/65% RH. However, other piments such as riboflavin and carmine, were quite stable for 12 months, independent of storage conditions. There was a good correlation in color change trend between ΔE values and visual assessment.

Figure 1. Change in ΔE Values for on Tablet Stability of Pigments in Nutrafinish TiO2 Free Film Coating System at Different Storage Conditions in HDPE Bottles

◆ carmine, ■ radish anthocyanins, ■ caramel, ▲ spirulina, ▲ riboflavin, ● red beet powder, ● chlorophyll, ● safflower, ● curcumin, ● carbon black



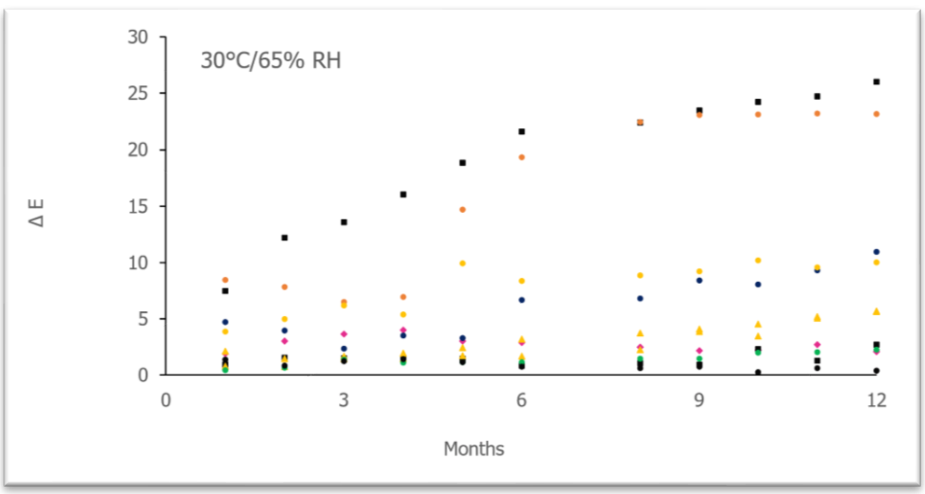
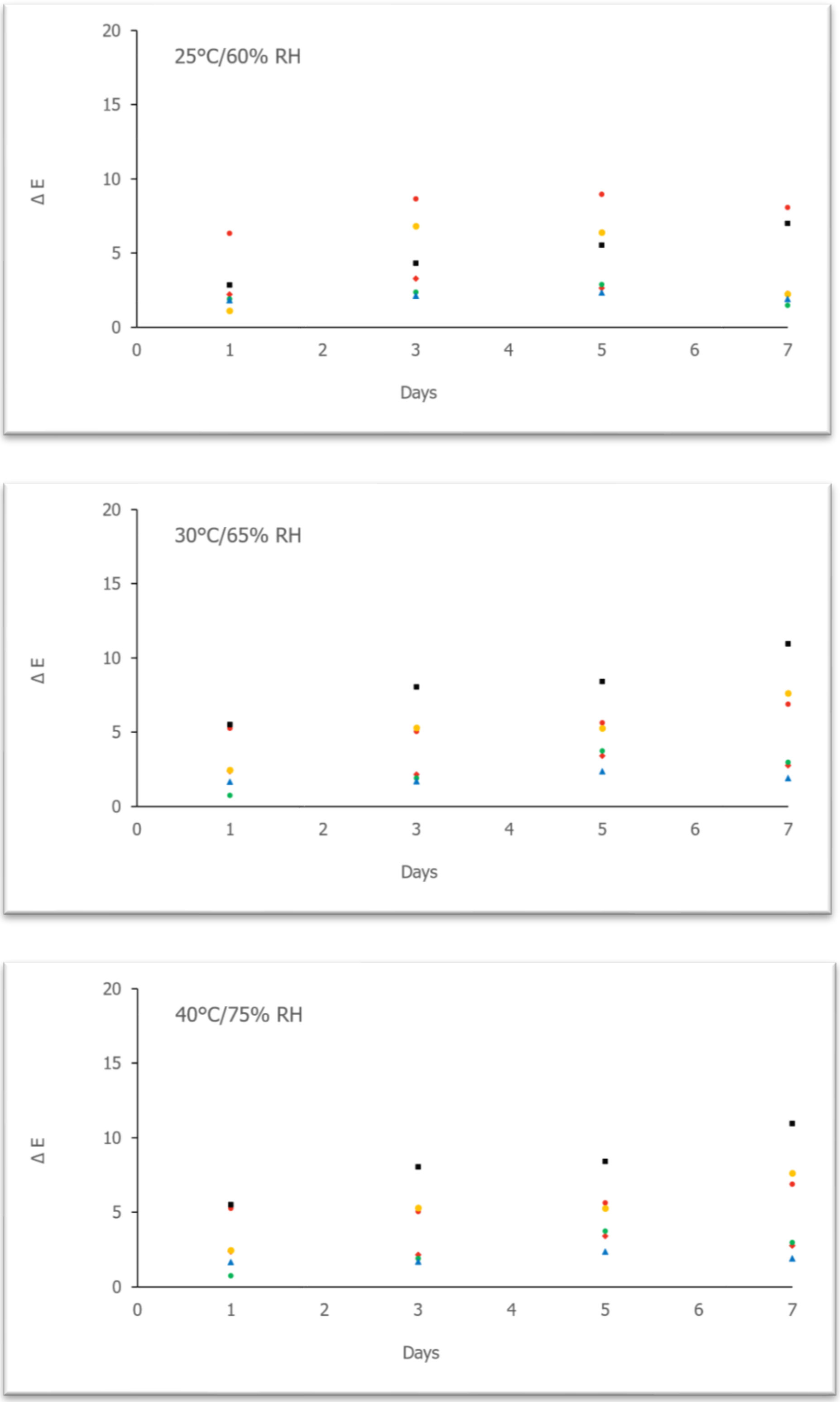


Figure 2 shows the change in ΔE values for selected pigments at 25°C/60% RH, 30°C/ 65% RH and 40°C/ 75% RH conditions in open-dish stability conditions.

Figure 2. Change in ΔE Values for Pigments in Nutrafinish TiO2 Free Formulated Film Coating Coated Tablets Over Time at Different Storage Conditions in Open Dish Study

◆ carmine, ■ radish anthocyanins, ▲ spirulina, ● red beet powder, ● chlorophyll, ● curcumin



Like the results obtained in HDPE bottles, stable pigments such as carmine, showed minimal change in ΔE values (Figure 3). Delta E values significantly increased for less stable pigments such as radish anthocyanins and red beet powder in the order of 25°C/60% RH < 30°C/ 65% RH < 40°C/ 75% RH. This was particularly more pronounced at 40°C/ 75% RH. Red beet powder showed similar results to those shown in Figure 4 for radish anthocyanins. Good correlation was found between ΔE values and visual appearance.

Figure 3. Stability of Carmine used as Coating Pigment at Different Storage Conditions

0 days

1 day 25°C/60% RH

1 day, 30°C/65% RH

1 day 40°C/75% RH

3 days 25°C/60% RH

3 days, 30°C/65% RH

3 days 40°C/75% RH

5 days 25°C/60% RH

5 days, 30°C/65% RH

5 days 40°C/75% RH

Figure 4. Stability of Radish Anthocyanins used as Coating Pigment at Different Storage Conditions

0 days

1 day 25°C/60% RH

1 day, 30°C/65% RH

1 day 40°C/75% RH

3 days 25°C/60% RH

3 days, 30°C/65% RH

3 days 40°C/75% RH

5 days 25°C/60% RH

5 days, 30°C/65% RH

5 days 40°C/75% RH

Conclusions

Some of the naturally derived pigments available for dietary supplement and nutritional applications tend to be less stable on stability, which can impact the shelf-life of the final film coated products. Changes in color during shelf-life of a product may cause consumer concerns. As the demand for the use of naturally derived pigments, to support clean-label products, continues to increase, it's important to evaluate in-use stability to meet shelf-life expectations.

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