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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

PIGMENT GREEN B TONER PULP, K-1885
PIGMENT GREEN B TONER (DRY) K-1913

Period Covered: 8-10-42 to 5-28-43

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SUBMITTED BY: A. D. Reidinger — DATE SUBMITTED: 8/25/44
A. D. Reidinger

APPROVED BY: A. A. Brizzolara — DATE ISSUED: 9/8/44
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INTRODUCTION:

This series of experiments had as its original objective a study of the effect of various surface active agents in the Pigment Green B formula, GT-68-P, for the particular purpose of obtaining a dry toner possessing good ink texture. A previous example of this type of product, K-1313, developed to compete with Sherwin-Williams Permansa Green B, CP-594, was high in materials cost and not as good in texture as desired. During the course of this study, the current scarcity of castor oil derivatives made it necessary to find a substitute for para soap, at present used in GT-68-P. Consequently a number of agents were tested with this object in mind.

SUMMARY & CONCLUSIONS:

Of seventeen soaps and dispersing agents which were substituted for Para soap in GT-68-P formula, sodium oleate was found to be most promising for use in manufacture of the pulp and also for production of a dry toner possessing good grinding properties and tinctorial strength.

A formula for the pulp color, incorporating use of this soap, was written for semi-works trial (see K-1885). After a few changes a passable duplication of GT-76-P, Pigment Green for wallpaper, was obtained in lot 6486. The latter also gave a satisfactory dry toner which was designated as K-1913. A separate report on the semi-works development of the formula and its application to the preparation of Pigment Green lakes will be prepared.

In substituting sodium oleate for para soap it was found necessary to modify the GT-68-P formula in three respects.

1. Adjustment of the filtered ester to higher pH than in standard formula. The filtered ester normally has a pH = 6.0-6.2. When sodium oleate is added at this point, it is precipitated in a flocculent condition and with few exceptions the products obtained were dull and weak. At pH 7.1-7.2 and higher, the sodium oleate remains in solution and is apparently more effective in its action, resulting in greater strength and intensity. In the semi-works, better results were obtained by adjusting the ester to 7.3-7.4.

2. The amount of soap used. Although replacement of para soap with sodium oleate on a pound per pound basis gave satisfactory toner pulps, the lower amount of oleate used in both K-1885 and K-1913 (22 lbs. per mol beta-naphthol) was found to result in stronger dry toners. This should be advantageous in the preparation of Pigment Green B lakes. In the laboratory the strongest dry toner was obtained with an equivalent of 11 lbs. per mol charge.

Some variation in water content of different samples of sodium oleate was encountered. The material used in this series of experiments contained 60% solids (after 24 hrs. at 90°C).

3. Amount of copperas used. In accordance with current plant practice the amount of iron salt used in K-1885 is 5% less than in the standard GT-68-P formula or 90# $FeSO_4.7H_2O$ per mol beta-naphthol. This is very slightly lower than the theoretical amount (92.7#) derived by assuming the formulas $(C_{10}H_6O.No)_3Fe'''$ or $(C_{10}H_6O.No)_3Fe''$ Na for Pigment Green B.

Control of the excess iron is important since higher amounts resulted in weaker, duller pulp colors. Use of analyzed copperas solution is preferable.

As a result of a separate study of water bleed of the toner pulp which is reported separately, variations such as lower rate of agitation and reduction in stirring periods were included in the process for K-1885.

As stated above, the product obtained on K-1885 is a practical match for GT-76-P, the wallpaper code. It is believed that satisfactory GT-75-P for paper dyeing can also be made since the laboratory product, although weaker than standard, was equal to the Para Soap control. An evaluation in rubber will be made by the Rubber Laboratory when samples of plant batches are available.

The dry toner, K-1913, is superior to Pigment Green Toner K-1313, in ink texture and body. Like K-1313, the new toner is more intense in undertone and tint than Sherwin-Williams Permansa Green with which it was intended to compete. The latter has slightly better light-fastness especially in zinc oxide extensions than some of our products but semi-works results indicate that this property can be controlled and improved.

The goal yield for the formula K-1885 was found to be 370 lbs. compared to 375 lbs. for GT-68-P (1.9 mol charge). The estimated formula yield for the latter is 355 lbs.

Of the other soaps and surface active agents which were tested in GT-68-P, several showed possibilities as substitutes for para soap. Turkey Red Oil, Prestabit Oil of General Dyestuffs, and Nopco 1700X of National Oil Products Company produced good pulp colors but weak dry toners. As in the case of sodium oleate some modification of the formula or change in amount may be required to give the best results.

Three samples of Speno Sol from Spencer Kellogg were of interest both for the pulp and the dry color obtained. A slightly granular condition of the pulps obtained would be objectionable.

Ammonium and possibly sodium rosinate also gave satisfactory tinctorial properties but caused an objectionable degree of bleed in water.

Since none of these soaps had been found satisfactory as replacements for Para Soap in other codes such as RL-211-D, no further work was done in improving their behavior in GT-68-P.

EXPERIMENTAL DETAILS - Notebook 965

Replacement of Para Soap in GT-68-P by other agents.

965-39 Para Soap, Turkey Red Oil, Sodium Oleate, Aerosol OT
965-40 Para Soap, Dupinol WA, Compound #8, Alkanol B
965-41 Para Soap, Ammonium Naphthenate and Rosinate
965-43 Para Soap, Ammonium rosinate
965-49 Para Soap, NH_4, Na and Ba rosinates.
965-60 Para Soap, Speno Sol, Prestabit Oil, Turkey Red Oil
965-61 Para Soap, Nopco 1338, CN, 990
965-62 Para Soap, Nopco 1700X, Speno Sol 3A & 3B
965-65 Sodium Oleate, Prestabit Oil, Nopco 1700X, Nopco R

Development of Sodium Oleate formula.

965-44 Variation in amount of Sodium Oleate
965-46 Comparison of copperas samples, rate of agitation & stirring
965-48 Acidification of Pigment Green slurry after strike. (time
965-50 Lakes of Sodium Oleate Toner.
965-51 Variation in pH of filtered ester
965-52 Rate of agitation during addition of sodium oleate
965-53 Reduction in amount and form of copperas
965-54 Variation in pH of filtered ester
965-55 Comparison samples of sodium bisulfite
965-56 Reduction in amount of copperas
965-57 Comparison para soap & sodium oleate at higher pH of ester
965-58 Variation in pH of filtered ester
965-59 Reduction in copperas at higher pH of ester
965-63 Variation in amount of sodium oleate at high pH
965-64 " " " " "
965-66 " " " " "
965-70 Point of addition of sodium oleate
965-71 Goal yield of sodium oleate formula, K-1885

Samples were tested as pulps by brushout in glue-clay dispersions and as dry colors by rubout in varnish drier.