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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

GT-76-P, PLANT STUDY BASED ON SICK LIST
WORK REQUEST #34

Period Covered:

DECEMBER, 1944 - FEBRUARY, 1945

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

TITLE: GT-76-P, PLANT STUDY BASED ON SICK LIST
WORK REQUEST #34

Period Covered: December, 1944 - February, 1945.

SUBMITTED BY: E. J. Hess *E.J. Hess*

DATE SUBMITTED: 4/4/45

APPROVED BY: V. Chalupski *V. Chalupski*
18 Apr. 1945

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INTRODUCTION:

In December, 1944, the erratic results being obtained in plant GT-76-P production made it necessary to place the manufacture of this pigment under the supervision of the Development Section. Sick list charge #34 was opened to cover the cost of this work.

In the work request issued by the plant, a number of possibilities were suggested as to the reasons for the tendency toward weakness and the undesirable gelation effect being secured. These included:

1. The possible variation in agitation during the strike due to the use of a make-shift procedure (belt slipping) for securing a necessary slow agitation rate for control of water bleed.
2. The use of copperas crystals on an "as is" basis (possible 5-10% variation in FeSO_4 content).
3. Poor press washing.
4. High dry content of the standard pulp, lot 93214.
5. Unsatisfactory duplicability of the brushout testing method.

Results on a total of twenty-four hatches are available for this report.

A new standard GT-76-P formula, K-2133, has been written based upon the information developed during this sick list study.

SUMMARY & CONCLUSIONS:

A preliminary investigation of conditions under which GT-76-P was being manufactured and tested during the period immediately preceding and just following the issuing of the work request disclosed the following undesirable factors:

1. As indicated by the plant, "belt slipping" as required to meet the agitation rate specified in the "non-bleeding" formula could result in variable stirring during processing which might affect quality.
2. Also as indicated by the plant, the addition times specified in the non-bleeding formula could not be met without the use of an extra vat for dissolving the soda ash added after strike.
3. Hot wash water had been used in a few batches by the plant in an attempt to facilitate press washing. Temperatures as high as 115-120°F (coming through the press) had been used whereas the optimum development temperature for the color is 104°F.

4. Dry contents of the crude press cake had been running below 30% (normal: 31-35%) which may have been due to the failure to maintain the gauge pressure at 125-135 lbs. during the full 35-40 hour washing cycle.

5. Abnormal color bleed during washing which seemed to be the result of using in incorrect grade of filter cloth. This, of course, played a considerable part in the failure to maintain normal washing conditions because of the color bleed from the filter press at the higher gauge pressures.

6. The press was noticeably corroded, parts were plugged, etc. A thorough cleaning was necessary.

7. The standard GT-76-P, lot 93214, in use at this time, was objectionable on two accounts:

- (a) It is of the "bleeding" type so that all batches tested must be better in this characteristic than the standard. The actual bleed of the lot to be tested, therefore, could only be estimated with the usual testing procedure (spot test versus standard).
- (b) At the time lot 93214 was tested against the previous standard (lot 26422) strength was satisfactory on an "as is" basis at 27.4% dry content which was within the desired range (27.0-27.5%). However, at the time the new standard was set up (two or three weeks later) the dry content had increased to 28.5% and the standard, lot 93214, has been used at this figure up to the present. Matching this standard, therefore, has resulted in giving away approximately 4% of strength as it has been necessary to maintain dry contents at 28.5%-29.0% instead of the original specification.

Manufacture under Development Section supervision may be divided into two groups:

I-Production designed to correct manipulation and processing deficiencies in the "non-bleeding" formula for GT-76-P (modified K-1439).

II-Production using new variables in an attempt to correct weakness and gelation difficulties. This was made necessary by the failure of ordinary control variables to accomplish the desired result.

In any discussion regarding control of quality on Pigment Green B, on a batch to batch basis, it should be realized that, even under the best of circumstances, significant test results are not available until ten days after the start of manufacture while in many cases two or even three weeks' time elapses before final conclusions as to quality may be drawn. Attempts were made in this study to secure some advance information as to tinctorial properties by obtaining brushout tests on vat control and crude press cake samples but the results were inconclusive as will be discussed later in this report.

Production under Group I consisted of thirteen batches. Two corrections in the making and processing procedure were made at once, i.e., an extra vat was used in preparing the soda ash solution in advance so as to make it possible to maintain stirring times in the non-bleeding formula and the use of hot water to speed up press washing was stopped. The variables used and the results secured follow:

(a) Eight batches were made using the non-bleeding formula except that 180 pounds of copperas in solution 100% (N-24) were used to replace the same amount of copperas crystals "as is" (N-391). It was during the course of this series of batches that it was discovered that the supposed "low grade" of #8 duck press cloth being used was really XX chain cloth. This type has always given considerable trouble in Pigment Green B filtration especially during the washing cycle and is definitely not recommended.

Although the press was cleaned and scraped and the correct type of filter cloth used in five of these batches, there was little, if any, quality improvement and the dry content of the crude press cake remained low in spite of the normal pressing conditions. Attempts to increase the density of the cake by decreasing pressing volume by 5% and by sandwiching Kraft paper between the press cloths were not successful. A further indication that poor washing was not entirely responsible for quality difficulties was that ~~the~~ attempts to re-work part of two batches by re-filtering and washing in the semi-works gave practically no improvement. Under-washed Pigment Green B pulp normally responds to this treatment.

Of the eight batches in this series only one was O.K. to ship. Two of the remaining batches were shipped straight although both showed a noticeable gelling tendency and one was weak in tint.

(b) Two batches made to check exactly the non-bleeding formula (180# copperas crystals) were unsatisfactory. One was sub-standard in both strength and gelation, the other in strength only.

(c) A check to the original K-1439 procedure (fast agitation, increased strike and stirring times) except for the use of 180# copperas (100%) in solution instead of 190# copperas crystals "as is" finished up O.K. tinctorially but unsatisfactory in bleed and gelation.

(d) Revision of the strike and stirring times to conform to the non-bleeding formula but retaining the fast agitation rate (20 RPM) gave a product that was standard in color and in gelation but sub-standard in bleed. An attempt to improve the bleed by using an intermediate agitation rate (15 RPM) gave the desired non-bleeding product but color and gelation were unsatisfactory.

An examination of the results obtained in the above series of batches indicates that only one of the variables tried, the use of increased agitation rate, gave any definite improvement in tinctorial properties and this only at the expense of bleed resistance. Gelation and density of the crude cake remained abnormal.

After some discussion with the laboratory regarding other variables not in general use, it was decided that, in view of the blueness and dullness of tint of current production, a decreased copperas amount might be tried. An examination of the formula shows that the 180 pounds of 100% copperas figure is equivalent to a 4-1/2% excess disregarding the probable loss of Beta Naphthol during the nitrosation, esterification and clarification procedures. It was finally decided to cut the copperas amount to 170 pounds 100% using the analyzed solution. This weight is theoretically equivalent to a 1-1/2% deficiency of copperas (172-1/2# $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ 100% is the theoretical figure for the standard 273 pounds of Beta Naphthol) but if losses are considered this amount may represent a very small excess (as indicated by the spot test).

Production under Group II, therefore, consists entirely of batches made with decreased copperas amounts. These are divided as follows:

(a) Nine batches were made to check the "non-bleeding" formula except for a decrease in the copperas from 180# 100% to 170# 100%. Of these seven were "O.K. straight" and two were sub-standard (one was too weak and the other was deficient in gelation resistance). This is considered fairly satisfactory performance for this color as the occasional batch sub-standard by brushout or beater test can easily be consumed in lake manufacture or as the rubber color, GT-68-P (when active). Unexpected effects of this new variable was the improved gelation and the return to normal dry content (31-35%) of the crude press cake.

(b) While awaiting results on the first trials of the low copperas amount, a single batch was made adjusting the ester pH close to neutrality prior to the strike. This variable had been used in the production of GT-646-D (dry Pigment Green B) to obtain improved strength. An unsatisfactory weak product was obtained in this case.

(c) A single batch made using a further decrease in the copperas amount to 165 pounds 100% was O.K. to ship but showed no better than average quality (2% weak - vvs blue and dirty). As this copperas figure corresponds to a 4-1/2% theoretical deficiency and because of the very slight yield loss, it was decided to standardize on the basis of the 170 pound amount.

In attempting to set up a shorter time cycle for determining the quality of a batch, a number of brushouts were made on vat control and crude press cake samples. Only tinctorial quality tests can be obtained in this way. The brushout procedure used differs from the standard method, TF-52-9, in that it is necessary to screen the color mixture before brushing. The results received are shown in the following table:

Total batches tested: 15
Batches on which both crude press cake), 4
and vat control tests are available)
Vat control tests only: 11

Performance:

Crude press cake and vat control tests:

Finished: 2 NG in color and gelation;
1 NG gelation only;
1 NG in color only; none O.K.

Crude press cake:

3 NG in color; 1 OK in color

Vat control: 1 NG in color, 3 OK in color

Vat control tests:

Finished: 3 NG in color and gelation;
2 NG in color only;
2 NG in gelation only;
1 NG in gelation and bleed;
1 NG in bleed only; 2 OK

Vat control: 5 NG in color, 6 OK in color.

An examination of the above results shows that although both vat control and crude press cake results are fairly significant tinctorially (crude press tests were 100% in agreement with finished product results; vat control results showed 73% performance), the remaining important characteristics, that is, gelation and bleed, interfere to such a large extent as to seriously affect conclusions. Thus, final performance figures would be - crude press cake results, 25% accurate; vat control results, 22% accurate.

An attempt was made to set up one of the first OK batches made on the new formula, lot 20610, as a new standard on the basis of its satisfactory quality and dry content (27.5%). Unfortunately, however, although it passed the ordinary gelation test (24-48 hours standing without noticeable gelling tendency) two months standing resulted in definite gelation and the batch was not acceptable as standard. Tests are now being run on a second batch, lot 21623, preparatory to restandardization.

Although, with the selection of the proper formula, quality difficulties that have been ascribed as possibly due to variation in agitation resulting from belt slipping have disappeared, a project has been approved to replace the belt drive on the strike vat, vat 202, with a variable speed drive.

Average yields since the density of the crude cake has returned to normal have increased from 94.8% of the goal figure to 99.6%. The single batch made with 165 pounds copperas (a probable deficiency) finished up at 98.7% of goal.

A new formula for GT-76-P based on the modified non-bleeding formula has been written and coded K-2133. New formulas (based on K-2133) for the other Pigment Green B pulp colors, GT-68-P, GT-75-P and GT-79-P have also been written and coded K-2134, K-2135 and K-2136 respectively.

EXPERIMENTAL DETAILS: DSN-45-6.

MPH