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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Final Report

NT-425-D Cost Reduction Study - Semi-Works and Plant

Period Covered: February 1944 - July 1945

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

Title: RT-425-0 COST REDUCTION STUDY - SEMI-[illegible] AND [illegible]

Period Covered: February 1944 - July 1945.

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LABORATORY: [illegible]

REPORT DATED: Y. Chalupski

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INTRODUCTION

A laboratory process, K-2042, was developed in order to [illegible] the cost of the standard calcium lithol red RT-425-D. This process requires dry shading of the struck product with "Duol" red dark RT-432-D in order to obtain the blue tint of RT-425-D. The base [illegible] produces a strong lithol, which is diluted and cheapened by addition of resinate.

This process was studied in the semi-works and then taken to the plant; after an experimental program was completed there, a plant standard was set up. The new formula is K-2059, which is [illegible] of K-2058 (the actual struck pigment) shaded with RT-432-D.

SUMMARY AND CONCLUSIONS

Semi-Works

All of the semi-works study was done on the same basic laboratory formula, K-2042. In this process, the diazo is made by dissolving [illegible] tobias acid in hydrated lime, to which is added sodium nitrite and hydrochloric acid; lime and beta-naphthol are slurried together and then acidified with acetic and hydrochloric acid. The diazo and beta-naphthol suspension are mixed and the material coupled by adding caustic soda. Sodium resinate solution is then added and the material developed by heating. A series of 26 semi-works batches was made over a period of about 5 months in which numerous chemical and operating details of the process were investigated. Early results indicated that material similar to the laboratory product could be made; such pigment is [illegible] and yellow compared with the RT-525-D standard and requires shading with dark calcium "Duol" (RT-432-D) to produce a replacement for [illegible].

Apparently the most important point in obtaining satisfactory quality using the new process is the proper control of pH throughout the process; much of the semi-works investigation was concerned with [illegible] particularly the amount and quality of lime used with the tobias acid and beta-naphthol, the amount of caustic soda used in the coupling, [illegible] the pH after coupling and before development. Both agricultural and chemical grades of lime were tried; satisfactory results were obtained with both when the correct quantities were used; but there appears to be some non-uniformity in both grades, so that unexpected variations [illegible] occur from time to time. The mechanical conditions in reacting [illegible] lime with tobias acid and beta-naphthol also appear to be important. In the case of the beta-naphthol some difficulty was encountered [illegible] the beta-naphthol collecting as a solid unreacted lump under the agitator. Optimum pH after coupling was determined in the sem- [illegible]

A modification of the formula described above, in which [illegible] sodium resinate solution is added to the beta-naphthol before [illegible] tion of the diazo, was given two semi-works trials. [illegible] it was thought that this procedure might [illegible] the semi-works results were undesirable because of dull [illegible]. [illegible] miscellaneous variables tried in the semi-works [illegible] acid [illegible] increased strength gave [illegible] strength; stirring the tobias acid with a deficiency of lime, [illegible] later addition of the rest of the lime gave [illegible] results; [illegible] of [illegible] pH to give lower ink showed no effect on [illegible].

Representative semi-works batches of satisfactory quality were evaluated in ink; compared with the old standard they had somewhat longer mixing time, slightly heavier body but with more ink length, satisfactory livering and litho resistance properties, and excellent gasburne. These batches were also tested for Dulux bleed and found satisfactory.

Plant

In the first plant batch, a development temperature higher than that used in the semi-works was employed in order to get a lighter masstone. This batch was light and weak, due not only to the high development temperature but also to the fact that the beta-naphthol was stirred with the lime for an excessively long time; in the second plant batch this long time of stirring was reduced and the development temperature was lower, resulting in a product similar to the semi-works material in quality except for slight dullness of masstone. A temporary working standard, lot 7249, was prepared from a check batch (92 parts lot 15969, 8 parts dark "Duol" RT-432-D). During the next few months attempts were made to correct dullness of masstone by various means, including increasing the amount of rosin used, increasing the amount of lime in the tobias acid, the use of chemical grade lime (with adjustment in the amount to correct for higher available alkali) and maintaining the alkalinity during development by adding caustic soda as needed. Very slight improvements were obtained at optimum alkalinity, as discussed in the semi-works section above. Use of a deficiency of lime with the tobias acid, followed by a later addition to make up the deficiency, as discussed in the semi-works section, resulted in weak material in the plant. Reduction in coupling volume also gave weakness. A mix of 11 normally prepared plant batches shaded with 8% of RT-432-D was, therefore, made up and recommended as a new standard. Compared with the old type of RT-425-D, this mix (lot 43807) was dark, 8% strong and blue in tint. This mix was made up in September 1944 and was used as a working standard until it was finally set up officially in July 1945. A new standard formula for the main component of RT-425-D was written; this is K-2058, which is standardized to RT-425-D by mixing with RT-432-D. This mix is designated K-2059.

The new process for RT-425-D represents a twelve percent reduction of ingredients cost; the differential is less in out-of-pocket and total cost. The following data, from the second quarter replacement costs are not completely comparable because of revisions made in the methods of costing:-

	Ingredients	Out-of-Pocket	Total
New process K-2059	32.75	50.83	73.44
Old Process K-1550	37.01	52.53	75.39

A more detailed discussion of the variables which effect quality may be found in the operating notes for K-2058, a copy of which is included under Experimental Details of this report.

EXPERIMENTAL DETAILS

See Development Section Data Books DSW-45-21 for semi-works and DP-45-22 for plant work.

Notes on RT-425-D Component K-2058

K-2058 is representative of the new low ingredient cost Calcium Lithol toner designed to replace K-1559. As the new product is lighter and yellower than the original RT-425-D it is necessary to use RT-432-D in small amounts this material to approximate the RT-425-D standard tinctorially.

The principal reasons for the approximate $6.19 ingredient cost savings per 100# color obtained with the new formula are the following changes from K-1559:

1. The substitution of hydrated lime for the calcium chloride and most of the caustic soda.

2. Omission of the "Lithosol" Mono Acid F.

3. The use of 80# of resin per mol in the strike to give increased yield.

4. The final dry mixing in of about 8% of the comparatively low ingredient cost pigment, RT-432-D. S

Some experience has been had in the plant with the following control variables:

Intermediate Quality: - As is the case with most Lithols the quality of the "Lithosol" Acid TK (Tobias Acid) has a definite effect on tinctorial properties. Masstone is most affected by slight variations in ingredient purity.

Amount of Hydrated Lime: - In "Lithosol" Acid TK Slurry: As far as is known best plant results are obtained by controlling the pH of the Tobias Acid partial solution within the specified pH range of 10.5-11.5. The present standard amount of chemical grade hydrated lime (44 pounds per mol) should give the desired result. If agricultural grade lime is used the initial amount should be increased to 46 pounds per mol.

In cases where too much lime has been added (resulting in pH tests in the order of 12.0 for the Tobias Acid solution) a definite tendency toward the production of dark, weak material has been noted. When pH tests are abnormally high it may be possible to adjust pH before diazotization with acid in order to secure a satisfactory product. There has been no experience in the plant with this, however.

A low initial pH on the Tobias Acid solution (in the range of 9.8 gives more complete solution but a lighter, weaker pigment is usually obtained. One or two pounds of lime should be added to adjust pH in a case of this type.

In Beta Naphthol Slurry: There should be no difficulty in controlling pH of the Beta Naphthol slurry within the desired range (11.0-11.5) using 44 pounds per mol of chemical grade hydrated lime or 55 pounds of the agricultural grade.

Alkali for Coupling: -

Alkali Amount: The standard amount of 60 pounds per mol is believed to be close to the optimum. Lowering the amount in the Fred-Works to 5[illegible] pounds per mol gave low coupling pH tests and slight diazo excesses. Carrying through the development under these conditions gave light, dull [illegible] weak tints.

Increasing the coupling alkali amount gives high coupling pH tests which requires additional acetic acid to carry through the pH adjustment after coupling. If the adjustment is not made high development pH conditions tend to give dark, dull, weak products.

Rate of Addition of Alkali: The present standard addition rate for the coupling alkali of 4-6 minutes is believed to be close to the optimum. There is no plant experience with variation in time of addition.

Coupling:-

pH after coupling: pH tests on the coupling after the 15 minute stirring period usually range between 10.0 and 11.0 and are adjusted, when necessary, to a pH of 9.8-10.2 before the addition of the sodium resinate. When pH tests are below 9.0 a diazo excess may show up which should disappear when the proper pH adjustment is made.

Diazo and Beta Naphthol Excess After Coupling: The normal control tests for this coupling are a strong excess of Beta Naphthol and a negative test for free diazo. Occasionally, however, a slight test for excess diazo together with a faint test for Beta Naphthol are obtained even though the [illegible] test is normal. No definite explanation can be made for this phenomena although it is believed that the conditions under which the Beta Naphthol is precipitated are responsible. In any case, as a very dull masstone is secured if the pigment is heat developed in the presence of free diazo, it is necessary to remove the excess diazo by coupling with Beta Naphthol. This is best accomplished by adding 2 or 3 pounds of Beta Naphthol dissolved in warm water, with about 1/4 pound of caustic soda. Normally, this first addition of Beta Naphthol should remove the diazo excess but in some cases as much as 10 pounds of Beta Naphthol are needed to give the desired effect. Batches of this last type are usually of poor quality but when only small amounts of the intermediate are needed the batch usually is close to the normal quality.

Development:-

Temperature of Development: The standard development temperature ([illegible]°F) gives optimum depth and strength. Increasing the development temperature to 175°F. gives lighter, somewhat weaker products.

pH of Development: The normal pH drop during the development is in the order of 0.4-0.8 units. Some of the batches, however, drop as much as 2.0 units (reason unknown) with the result that a lighter, flatter masstone and weaker product is secured. This can be controlled, to some extent, by small

pH at intervals during the heating cycle and adjusting the pH if necessary with 2 or 3 pounds of caustic soda. This should be done before the temperature reaches 130°F. in order to obtain the desired effect. A very small amount of experience with this treatment indicates that the tendency toward loss of strength is eliminated but no improvement in masstone is obtained.

Possibility of "OK Strength" Manufacture: Manufacture on the K-2058 formula has been controlled on both the light and dark side of the original RT-425-D standard, lot 25956. However, all manufacture with the new formula has been definitely yellower than the desired shade which makes it necessary to dry mix light masstone material made on the K-2058 formula with approximately 2% RT-432-D to give the required masstone and tint.

Attempts to use an accessory agent with the new formula in the manner of the original RT-425-D (Mono Acid F used) were unsuccessful. In most cases dark, yellow products were secured; where blue tints were obtained the products were very dull in masstone and weak in tint.

At the present time the only possibility for setting up an "OK Straight" formula is to slurry mix with the proper amount of RT-432-D either in slurry or reslurried press cake form. This procedure should be tested when more experience with the K-2058 formula has been obtained and better control established.

Yield:

Yields have varied over a fairly wide range (17%) but the average (479 pounds per mol) is very close to the original estimate of 480 pounds per mol. A goal yield has been calculated and amounts to 498 pounds per mol.

E. J. HESS
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