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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

RM-417-D, RE-482-D, and RM-484-D
PLANT DEVELOPMENT AND STANDARDIZATION OF
THE DIRECT LAKING PROCESSES FOR EXTENDED
"WATCHUNG" RED COLORS.

Period Covered: February 1945 - September 1947.

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Final Report

Title: RM-417-D, RE-482-D, and RM-484-D
Plant development and Standardization of the Direct
laking processes for Extended "Watchung" Red Colors.

Period Covered: February 1945 - September 1947.

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INTRODUCTION

Concurrent with the development work on the RT-428-D direct laking process in the plant (see Report KN-47-21) a similar study was started on the extended pigments derived from this toner, namely, RM-484-D, RE-482-D, and RM-417-D. There has been no laboratory or Semi-Works study on the direct laking processes for these extended "Watchung" red colors.

SUMMARY & CONCLUSIONS

RM-484-D

The early plant production of RM-484-D was based on the K-2089 toner formula (direct coupling, i.e., diazo into BON solution). In line with experience on the toner the material was very dull in masstone and blue and dull in tint versus the RM-484-D "isolated dye" standard, lot 32639, and was unsatisfactory.

Use of the reverse coupling variable (BON solution into the diazo) increased masstone intensity but depth was also increased. Strength and intensity of tint were improved. A proposed standard, lot 46778, blended from two of these batches adjusted with whiting to standard strength by rubout was rejected because of a slight strength deficiency by linoleum composition grind. A second proposed standard based on this process, lot 48157, (a blend of eight batches) was rejected as being too dark and dull in masstone. Lot 48157 was out-of-line dark versus a board mix of the new direct laking standard for RT-428-D, lot 47185, adjusted to RM-484-D strength with whiting.

Application of the increased coupling volume variable as used in the K-2235 toner formula to lighten masstone gave fairly satisfactory performance and a proposed standard blend, lot 48624, based on material of this type was submitted for evaluation. Preliminary tests indicated that this mix was a trifle too strong and tinting strength was adjusted (lot 49090) to give the following rubout comparison results versus the "isolated dye" standards:

Lot 49090 vs lot 32639: dark, dull - OK - vs strong - blue.

This material was in-line in masstone and tint with the RT-428-D direct laking standard, lot 47185.

Based on the acceptance of lot 49090 as the RM-484-D standard by Sales a standard direct laking formula. K-2264, was written and the Production Division assumed supervision of manufacture in January, 1947.

Meanwhile, acceptance of this standard was with-drawn because of a very slight deficiency in alkali resistance in linoleum. Sales was reluctant to request one of our major customers to accept this deficiency because of their rejection of a proposed standard on another color under similar circumstances.

The Production Division immediately returned manufacture of RM-484-D to Chemical Division supervision but shipments were continued on the basis of the lot 49090 type material.

During the development study and during the period subsequent to the rejection of the proposed standard a number of problems connected with production rate, quality and yield control, and the filtration operation, were encountered. The problems and the corrective measures taken are listed below:

1. The possibility of using vat washing with the direct laking formula was considered. This was made necessary by the chronic bottle-neck resulting from the non-availability of wash presses at the time. Because of the high concentration of the standard 1.5 mol batch in the 10,000 gallon strike vats satisfactory vat washing was practically impossible. Vat washing was only practicable at a batch size of 0.75 mols. As this was economically unsound the vat washing study was dropped.
2. As in the case of RT-428-D, deficiencies in the amount of Red 2B acid used per batch resulting from the use of erroneous supplier's analyses were corrected by analysis and adjustment of the Red 2B acid sodium salt solution in the vat prior to diazotisation. These vat analyses indicated that the supplier's figures were approximately 5% too high. Available data do not show any notable yield improvement with the corrected batches.
3. Masstone control was established by the use of an acidification step after heat development of the toner for light masstone and the use of increased caustic soda in the BON solution for dark masstone.
4. Abnormally long filtration time, resulting from the difficulty of washing out the heavy foam from the development vat, were corrected by the use of Octanol as an anti-foaming agent.
5. Considerable difficulty with plate and frame breakage resulted from attempts to press the standard 1.5 mol batch in a full press volume of 50 cubic feet. A filtration study indicated that a pressing volume of 68 cubic feet (two stump press loads) was needed for the standard 1.5 mol batch size.

On the basis of the above study a new standard formula, K-2425, was written to replace K-2264, incorporating the Red 2B acid sodium salt analysis, the use of the acid after heat development, the addition of Octanol as an anti-foaming agent, and the increased pressing volume. An alternate formula, K-2426, to fit a 50 cubic foot press was also written.

Subsequently, in order to make more efficient use of the 50 cubic foot presses, the batch size was increased to approximately 2.2 mols to fill two full press loads (100 cubic feet volume). This new process, K-2506, differs from K-2425 primarily in that the batch size is increased by approximately 1.5 times, the BON volume before coupling is decreased by approximately 30%, and the large diazo volume necessitates that diazotisation be accomplished in the coupling vat.

Finally, in July, 1947, lot 49090 was re-submitted to Sales as a standard for RM-484-D and after contact with the customer the material was accepted.

The Production Division accepted supervision of RM-484-D manufacture in September, 1947.

RM-417-D

The first plant production of RM-417-D was also based on the K-2089 toner formula (direct coupling, i.e., diazo into BON solution). Difficulties with dull masstones and tints similar to those experienced with RM-484-D were corrected by application of the reverse coupling variable.

A proposed RM-417-D standard, lot 48150, was prepared by mixing three batches made on the reverse coupling formula. At first this lot was rejected for standard because of its dark, dull masstone compared to the "isolated dye" RM-417-D standard, lot 29113. However, when it was shown that lot 48150 was a close masstone match to an extension of the RT-428-D direct laking standard, lot 47185, the material was submitted to selected customers and was finally accepted as the RM-417-D standard in August, 1946.

A formula based upon that used for the component batches of the new standard was set up as the RM-417-D standard formula, K-2266, during February, 1946.

Meanwhile, while awaiting acceptance of the new standard, a variable study similar to that outlined for RM-484-D was run. A resumé of the results obtained follows:

1. As was the case with RM-484-D manufacture, analysis of the Red 2B acid sodium salt solution showed that the suppliers analyses on this intermediate averaged about 5% higher than the actual figure. Correction of this deficiency apparently gave an approximate 4% yield improvement.
2. Slightly lighter masstones were obtained by increasing the diazo volume before coupling by about 30%. Use of the acid treatment after heat development was a much more effective means of obtaining light masstone material. Masstone depth is increased by a 2-3% increase in the caustic soda used for dissolving the BON.
3. Difficulties with foaming during heat development which resulted in extended vat wash-out times were corrected by the addition of Octanol as an anti-foaming agent.
4. Vat washing was not practicable because of the high concentration of the toner slurry and the consequent poor settling characteristics.
5. The pressing volume for the 1.5 mol batch was increased from 50 to 68 cubic feet in line with experience with RM-484-D.

Much of the above information was used in revising the K-2266 formula to put it on a more practical basis. Analysis of the Red 2B acid solution, addition of acid after heat development, use of Octanol as an anti-foaming agent, and increase in the filtration volume were all incorporated in the new standard K-2423 formula set up in December, 1946. K-2424, an alternate formula, designed to fit a 50 cubic foot pressing volume, was set up at the same time.

The Production Division accepted supervision of RM-417-D manufacture in January, 1947.

RE-482-D

The low sales volume and potentially large batch size of this 8% extension of RT-428-D for paper made it difficult to line up a development study on this color because of the comparatively few batches made.

Based on the results obtained on two batches manufactured during this period the following conclusions may be drawn:

1. At a batch size of approximately 1/3 mol (2045#) either reverse or direct coupling may be used. The diase volume is low enough in the case of the direct coupling to permit addition of the diase in 15 minutes or less. Vat washing may also be used at this batch size.
2. At the regular 1.5 molar batch size (8819#) the regular reverse coupling procedure must be used. The settling rate is poor so that press washing is necessary.

Both of the batches were close checks to one another and were graded dull - 30% strong - blue by brushout versus the "isolated dye" RE-482-D standard, lot 27822. After strength adjustment, these batches were close in masstone and strength but blue in tint versus the standard.

A new direct laking standard RE-482-D formula, K-2267, was set up in February, 1946, to replace the "isolated dye" type procedure, K-9349. This process was of the 1.5 mol size using a toner formulation similar to RT-428-D, K-2235. An alternate formula, one-half the K-2267 batch size, to fit in a 5000 gallon vat was set up at the same time and designated K-2277.

Revised RE-482-D formulations based on the new toner formula, K-2422, were set up in December, 1946. The variables used were the Red 2B acid analysis, use of Octanol, etc., changes previously discussed. K-2427 was set up as the normal 1.5 mol (8643#) size. The K-2428 alternate procedure was set up on a 0.75 mol (4321#) size to permit vat washing if desired.

Because of the low production volume the "isolated dye" RE-482-D standard, lot 27822, was retained.

The Production Division accepted supervision of RE-482-D manufacture in January, 1947.

PATENT SITUATION

There is no patent restriction in the manufacture of Red 2B colors. It is not believed that this study has resulted in any patentable developments.

GLOSSARY

RM-484-D A 45% extension of RT-428-D on Whiting.

RM-417-D A 42% extension of RT-428-D on an equal parts mixture of Alumina hydrate and Blanc fixe.

RE-482-D An 8-1/2% extension of RT-428-D on a 90-10 mixture of Barytes and Alumina hydrate.

RT-428-D The calcium-strontium salt of the coupling of Red 2B acid diazo and Beta hydroxy naphthoic acid.

"Watchung" Red Colors All Calcium Red 2B dyestuff pigments.

Red 2B Acid Orthochlor meta toluidine para sulfonic acid.

BON Beta hydroxy naphthoic acid.

Direct laking Preparation of the finished pigment from the basic intermediates without isolation or removal of the mother-liquor from the dyestuff.

EXPERIMENTAL DETAILS

RM-484-D DSN-48-1 Parts I & II
RM-417-D and RE-482-D: DSN-48-2.