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NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

FINAL REPORT

DEVELOPMENT OF A PRIMROSE YELLOW TO MATCH THE #90 YELLOW OF W. P. FULLER COMPANY

Period Covered: FEBRUARY, 1949 - MAY, 1949.

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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

FINAL REPORT

TITLE: DEVELOPMENT OF A PRIMROSE YELLOW TO MATCH THE #90 YELLOW OF W. P. FULLER COMPANY

PERIOD COVERED: FEBRUARY 1949 - MAY - 1949

SUBMITTED BY: Julius Jackson J. Jackson DATE SUBMITTED: 8/30/49

APPROVED BY: B. H. Perkins 9/29/49 DATE ISSUED: 9/29/49

INTRODUCTION:

For some time the plant had been experiencing considerable difficulty in manufacturing light, intense, green masstone Y-488-D suitable for sale to the W.P.Fuller Company. The Chemical Division was asked to develop a match for this customer's #90 Primrose Yellow standard.

SUMMARY AND CONCLUSIONS:

1. A batch process was developed which, in the laboratory, gave products superior to the Fuller standard. Semi-Works attempts to manufacture this material yielded unsatisfactory results, the products being extremely dark, green and dull in masstone.

2. Plant development of this product resulted in products which were considerably lighter and greener in masstone than those made by the standard process for Y-488-D. Such products were difficult to handle in the press room due to thixotropy. Several variations of this process were run in attempts to correct this situation.

3. A mix of several plant batches coded Y-488-D - 90860, was made up and submitted to the customer. This mix compared by rubout as follows with the Fuller standard (C#51214 - T.F.247-6).

Lot vs C#	M.T.	Str.	Tint
90860	vs dk.,red,intense	OK	red
Y-488-D,SD-48377	dk., v.red	=	=
In Varnish Drier			
90860 vs Y-488-D	lt.,intense	105=100	s.red

The mix was ultimately rejected by the customer because of excessive redness of masstone, despite the intensity advantage.

RECOMMENDATIONS FOR FUTURE WORK:

1. Plant evaluation of the leads suggested in the letter J.J. — VHC - 7/21/49 (see copy attached) may correct this situation.

2. A single source of vehicle (Archer-Daniel-Midland Pale Grinders acid refined linseed oil) be kept for all laboratories.

PATENT REVIEW:

Nothing of a patentable nature was discovered in this work.

EXPERIMENTAL DETAILS:

Simultaneously with a sick list study to improve the quality of Y-488-D (see KN-49-24), the Chemical Division was asked to produce "Super-standard" material (lighter and greener in masstone) for possible sale to the W. P. Fuller Company.

A review of recent production was carried out, and several attempts were made to produce lighter - greener material, by decreasing the quantity of chromate used in Y-488-D manufacture and applying certain of the techniques used in the above mentioned study. These techniques had given Y-488-D lighter in masstone than standard. Two batches were manufactured simultaneously; one batch in the plant green unit, and a second batch in the yellow unit. A definite superiority was shown by the material made in the green unit (probably due to better agitation). All products, although greener than Y-488-D, were excessively dark and dull compared to the Fuller standard (C#51214). Other variables, such as changes in the amount of soda ash utilized in white base, lead excess changes, variations in sodium chromate, strike temperature changes and variations in agitation, were studied in the laboratory without success. It was apparent, therefore, that a new process would have to be developed to produce pigment to match this competitive sample.

Several years ago, the writer had studied the possibility of manufacturing Y-488-D continuously. During the course of these studies, a continuous process had been developed which gave pigments which were lighter and greener than Y-488-D. (See N.B. 1121). Laboratory studies were made on this process and compared with those prepared by the Y-488-D method. A marked advantage of the new process, as compared to the standard method, was quite evident. This process utilized sodium fluoride for masstone intensity, and was essentially a phosphate-stabilized, heat developed rhombic phase yellow. The heat development step was quite unusual for a primrose yellow as light as Y-488-D. The process was modified to make it suitable for batch manufacture without difficulty. After several laboratory runs had been made, two attempts were made to manufacture similar material in the semi-works. These attempts were unsatisfactory, yielding dark, green, dull pigment without any apparent reason for failure.

Because of the very limited experience with Y-488-D in the semi-works, it was decided to attempt the manufacture of this material in the plant (Y-488-D - 65554).

The process behaved very satisfactorily in the plant. However, the press cakes were extremely thixotropic.

Laboratory investigation revealed that the thixotropy was a function of the amount of tetra sodium pyrophosphate used in the process and a second batch was made, decreasing the quantity of this material (Y-488-D - 65994) by elimination of the final portion. Unfortunately, as the amount of pyrophosphate was decreased, a tendency toward greater masstone redness was observed in the plant, and a compromise in quantity of pyrophosphate had to be made.

Several attempts were made to correct this situation by decreasing the amount of initial sodium carbonate used in white base preparation for greener masstones and by giving the press cake an aluminum sulfate wash after the regular press washing. By these means, products were obtained which were not overly thixotropic despite the large amount of pyrophosphate used (see Y-488-D, 66540).

A mixture, coded 90860, of three plant lots (66185, 66470, 66540) was submitted to the customer for evaluation and was rejected because of objectionable redness (see attached letters).

A special testing method T.F.247-6 has been set up for testing pigments for the W.P.Fuller Company. Considerable variation in tinctorial effects were noted, when different laboratories rubbed out the same product using variously aged samples of the specified vehicle (Archer-Daniels-Midland Pale Grinders Acid Refined Linseed Oil). Investigation showed this variation was exclusively the result of differences in behavior of our pigments in different samples of this vehicle. Unfortunately, the competitive pigment was not so sensitive to such change in the test vehicle. It is recommended that all such vehicle be kept in one can, and used by the various laboratories concerned when necessary, rather than being divided into individual supplies for each laboratory.

Letters pertinent to the problem are attached, summarizing the current status of the problem.

COST PENALTY

A total mill cost penalty of $1.15/100# versus Y-488-D, was indicated primarily to increased ingredient cost, and cost of heat development.

FORMULAS

A copy of a typical plant and laboratory formula is attached.

REFERENCES:

DSN-49-32
N.B. 1121
N.B. 1142

NEWARK, N.J.

JUNE 15, 1949.

MR. J. W. CRONIN F WILMINGTON

Y-488-D TO MATCH W.P.FULLER'S #90 PRIMROSE YELLOW C#51570

This letter will serve to summarize the status of this problem, which has been dropped from the Inorganic Group program.

Products have been prepared at a cost penalty of $1.00/100 lbs., which are very slightly red and intense versus the Fuller standard in masstone, as evaluated by standard Fuller testing procedure TF-247-6. They represent a definite improvement over Y-488-D in approaching the Fuller standard. All tints are very close to the C# in strength and hue. After 3 days outdoor exposure in GE-2527 alkyd, our products are at least equal to the C# (See exhibits 1-3-4 attached to Mr. Cronin's copy). After 7 days exposure (See exhibit 2) or after 24 hours in Weather-O-Meter, our products are considerably redder than C#. Y-488-D standard compares quite favorably with the C# by Weather-O-Meter evaluation (but is too dark and red by Fuller TF_247-6).

These products, which have been prepared by slightly varying types of formulas are unstandardized at present but the Production Division has agreed that their manufacture should not present any serious difficulty.

The following tables summarize our evaluations of these products:

Readings vs C# in Acid Refined Linseed Oil (TF 247-6)

Lot No.	MT	STR.	TINT	L/Fastness
65554	v.sl.red	s.strg.	vs red	v.sl.better
66185	s.lt.,s.red, int.	=	vs red	v.sl.worse
66470	vvs.dk., s.red, int.	=	s.red	*
66340	v.sl.lt., int.	=	vs red	*
Y-488-D, SD-48377	dk., v.red	± =	± =	vvs.worse

*not available as yet due to slow drying characteristics of Fuller rubout vehicle.

Readings vs C# in H-108 - Alkyd Rubout Tests *

	Original Reading MT	After 3 Days Outdoor Exposure	After 7 Days
65554	= depth, vs red	s.lt.,s.red, s.int.	dk.,red,int?
66185	vvs lt., vs red	red, s.int.	dk.,red
66470	vs lt.,vs red, vs int.	s.lt.,s.red, s.int.	v.dk.,v.red
66540	vvs lt.,vs red,vs int.	s.lt.,s.red,s. int.	v.dk.,v.red

*See exhibits 1 - 4.

Lots 66185, 66470, and 66540 totaling ±3200 lbs. were mixed under lot #90660.

Rubouts of this product by Control Laboratory read as follows:

	MT	STR.	TINT
Fuller TF 247-6 vs C#	vs dk.,red,int.	OK	red
<u>In Varnish Drier</u>			
Lot vs Std. Y-488-D	lt., int.	105=100	s.red

We have also included (Cronin's copy only) two exhibits of some additional studies conducted during this investigation. It appears at this time, that rebaking the <u>ground</u> pigment is highly beneficial to stability and color retention as evaluated by outdoor exposure in alkyd. Two exhibits of this type material are shown. Exhibit #5 represents a rebaked portion of the grind of lot 65994 (different from previous types). It is definitely superior to the competitive product in alkyd by outdoor exposure but is quite similar to our Y-488-D standard in tinctorial properties, as evaluated by the Fuller method (TF 247-6). Exhibit #6 shows the effect of rebaking the grind of lot 66185 (similar to components of mix) as evaluated in alkyd by Weather-O-Meter and outdoor exposure.

The effect of this rebaking procedure is new to us, and we have not attempted any studies of this in the plant. We would appreciate your evaluation of the value of this lightfastness improvement. Your comments as to the significance of tests in alkyd, in light of the use of acid-refined linseed oil in the customer's standard test, are also solicited.

JULIUS JACKSON
CHEMICAL DIVISION

NEWARK, N.J.

JULY 21, 1949

MR. R. P. ENSLIN
ROOM 522
111 SUTTER STREET
SAN FRANCISCO 4, CALIF.

W. P. FULLER & COMPANY
PRIMROSE YELLOW Y-488-D

Refer to letter RPE to JWS - July 12, 1949.

We regret that the customer has rejected our new offering of Y-488-D - Lot 90860. In my opinion, and also that of Mr. A.J. Hughes of the Newark Control Group, this lot represented a definite improvement in approaching the Fuller standard. It is much closer to their standard than Y-488-D SD-48377 or any recently submitted samples. We would not have submitted this sample had we not believed this to be so.

We were of the opinion that despite the redness of masstone, the greater masstone intensity and cleanliness would be of interest to the customer.

Since the Chemical Division is no longer working on this problem, certain suggestions that may correct the masstone redness will be attached to Mr. Chalupski's copy of this letter for possible plant trial.

JULIUS JACKSON
CHEMICAL DIVISION

NEWARK, N.J.

JULY 21, 1949.

V. H. CHALUPSKI

Y-488-D - FOR FULLER

Since Mr. Enslin reports that the lot 90860 submitted was too red in masstone, I would like to make the following suggestions for possible plant trial by your group.

Use Basic Process Y-488-D - lot 66540. Apply following variables -

(1) To reduce redness of masstone -
(a) decrease initial N-3 added to N-41 and replace by equivalent amount at adjustment of pH just before heat development. ±25 lbs.

(b) decrease N-8. Raise N-108 ±10 - 15 lbs. (will probably sacrifice some masstone intensity).

(2) To reduce thixotropy of presscake use 25 lbs. N-15/1000 gals. water for press washing. If some thixotropy remains increase N-15 in 10 lb. steps.

(3) Dry 12 hrs. at 140°F. Bake 12 Hrs. at 220°F.

JULIUS JACKSON
CHEMICAL DIVISION

LABORATORY PROCESS - Y-488-D

Lead nitrate		165.5 gr.
	in	3600 ml
	at	70°F
	pH	5.0

Add in 3 minutes

Sodium carbonate		14.0 gr.
	in	100 ml

Stir 2 minutes
Add in 10 minutes

Sodium bichromate	47.6 gr.
Sodium sulfate	2.8 gr.
Sulfuric acid	8.2 gr.
Aluminum sulfate	9.3 gr.
Sodium fluoride	2.0 gr.

in	900 ml
at	70°F
pH	1.15

pH after strike 1.4

Add

Tetrasodium pyrophosphate	4 gr./20 cc
Sodium carbonate	18.4 gr./200 ml

to pH 6.0

Heat to 190° in 1 hour.

Add with 2 minute intervals

Aluminum sulfate	9.3 gr./100 cc
Tetra sodium pyrophosphate	4 gr./200 cc
Sodium carbonate if necessary	

to a pH 6.0

Filter wash $SO_4^=$ free

Dry 16 hrs./140°

Bake 5 hrs. at 220°F.

Y-488-D for Fuller
Based on 66540

Est. Yield

Code: Y-483-D K#
Cook: 1593 Size:
Diazo Vat: 260,261
Coupling Vat:
Strike Vat: 260,261
Non-Bleeding

Approved By:

VAT	NAME: INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS
Prep 1.	1. Weigh and hold at Hogshead for step 13	140	3	140
	2. Weigh and hold at vat 365 for step 33	170	3	170
	3. Weigh, dissolve in 40 gallons water at 80-120°F in barrel and hold at strike vat for step 43	93	15	93
	4. Weigh and hold at Hogshead for step 36	40	479	40
	5. Weigh and dissolve in 30 gals. water at 120°F for step 45	40	479	40
S.V. 2.	6. Run 30" (+ 3600 G.) water into strike vat.			
	7. Run in from scale tank.			
	Fe M T %		41	1655
	Fe M T %		41	
	8. Adjust to 46" at 73-77°F			
	9. Adjust to pH 4.9-5.1 with N-7 or N-33. Record: pH °			
	10. Set agitator at 21 RPM			
Hogs Head 3.	11. Run 29" (89 G.) water into Hogshead.			
	12. Turn on steam.			
	13. Add N-3 weighed in step 1 in 2-3 minutes.			
	14. Heat to 80-120°F.			
	15. Stir to dissolve.			
Misc. 4.	16. Weigh and hold at scale tank 361 for step 21	93	15	93
	17. Weigh and hold at scale tank 361 for step 22	28	108	28
S.V. 5.	DO NOT START THIS STEP UNTIL REMAINDER INGREDIENTS PREPARED			
	18. Run N-3 solution prepared in steps 11-15 into strike vat on the surface in 3-5 minutes.			
	19. Stir 5 minutes.			
361 6.	20. Run 17" (283 G.) water into scale tank 361.			
	21. Dump in N-15 weighed in step 16.			
	22. Dump in N-108 weighed in step 17.			
	23. Stir to dissolve.			
	24. Run in (stir tank 355 before) M T 322 %		27	62
	25. Run in " M T %		8	476
	26. Adjust to 59" (979 G.) at 73-77°F			
	27. Test and record: pH °			
	28. Adjust to pH 1.1-1.2 with N-7 or N-33. Record pH			
	29. Stop agitator			
S.V. 7.	30. When step 19 stirring completed, strike scale tank 361 at 73-77°F without agitation into strike vat at 73-77°F., thru the under surface strike line as rapidly as possible. Record: Time started °. (usual time ±15 min.) Record: Total Mins. °.			
357 8.	31. Run 20" (85 G.) water into vat 365			
	32. Turn on agitator and steam.			
	33. Add N-3 weighed in step 2 in 2-3 minutes.			
	34. Heat to 80-120°F.			
	35. Stir to dissolve.			

Continued on page 2.

Vat	Page 2 CODE: Y-488-D 1563 Est.Yield. INSTRUCTIONS	USE LBS.	N-CODE	ACCT. LBS.
	36. Dump in N-479 from step 4 - stir 2 min.			
	37. Dump in N-3 from step 35			
	38. Record pH _____.			
	39. Adjust to 5.9-6.1 with N-3			
	40. Record N-3 used _____.			
	41. Heat to 180°F in 60 minutes.			
	42. Hold at 180°F for 15 minutes.			
	43. Dump in N-15 from step 3			
	44. Stir 2 minutes.			
	45. Dump in N-479 from step 5.			
	46. Stir 5 minutes.			
	47. Record final pH _____.			
	48. Flood to maximum volume.			
	49. Press without agitation			
	50. After pressing and washing press room will notify making to prepare alum solution for press washing. +90°F.			
	Make up to 1000 gals.	50	15	50

CODE: Y-488-D Goal: 1593
PROCESS: Date:

MAKING AREA NOTES

Est. Loss Lbs.
Remarks

PRESSING AREA INSTRUCTIONS

Vat
Date
Time
Released by
Press No. #2 North #2 South
No. of Cakes 18 18
Press with-OUT agitation
Wash to 8 hours. Record: ohms
Make first test after Hrs.
Load on 2 cars.
Special Instructions: Non-Bleeding
After washing notify making to prepare alum solution - pump thru press cake and dump.
Samples
Deliver to

PRESSING AREA NOTES

DRYING AREA INSTRUCTIONS

Type L.V. H.V. Hurricane
Hours -- 12 + 12) P&S
Temp. -- 140 220) only

Lump container See chart.
Special instructions Non-Bleeding
Do not pull any color within 25 ft. of Dryers.
Use Liners.
Samples
Deliver to

DRYING AREA NOTES

GRINDING AREA INSTRUCTIONS

Grinder No. 10 L.S.P.
Special instructions: Non-Bleeding.

Samples: 3 envelopes.

Deliver to: Control Laboratory
Do not mix before sampling.

Packaging: See chart.

GRINDING AREA NOTES

Y-488-D NOTES

1. A decrease in the amount of sodium carbonate added in the white base, accompanied by a simultaneous and equal increase in the second sodium carbonate addition, makes for lighter, greener masstones.

2. The thixotropy of the press cake increases with an increase in the amount of final tetra sodium pyrophosphate. Removal of this step eliminates most of the thixotropic character of the press cake, but results in the production of darker and redder masstone products.

3. Increases in total alum quantity makes for the production of lighter masstone material.

4. Washing the press cake with alum solution after pressing and washing materially decreased the thixotropy of the press cake. Some further increase in the volume of solution should be attempted to obtain better distribution of the alum. If this proves to be only partially satisfactory, an increase in quantity of aluminum sulfate used should be attempted.

5. Further studies on variations in the amount of sodium fluoride and of the quantity of sodium bichromate should be investigated as possible means of decreasing the masstone redness of the product.

6. The effect of higher strike temperature (90°F) should be investigated.