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NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Flushed Colors (Original Copy)

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Color Research Report.

Flushed Colors

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Introduction:

Work was continued during ~~the past~~ ^{this} month on the study of ~~the~~ vat flushing of colored pigments, i.e. a transfer of the pigment from the water phase to an oil phase directly in the strike vat without going through the pressing operation.

The early part of the month was devoted orientation experiments on several Duchs and Duols and some P.T.A. colors. The latter part of the month was devoted to a study of methods of treating

RT-364-D. ^{on water} Considerable success has been obtained. The contents of this report deal only with the coalescing effect of the pigment and oil from the water slurry as an ink mill was not readily available to efficiently carry out milling experiments of the products obtained. Therefore no Summary & Conclusions: conclusions have been made regarding this effect on functional and printing ink properties.

In the orientation work, moderate success was obtained with Duol Reds, RT-283-D & RT-242-D. Fairly good coalescing of ~~the~~ ^{from the water slurry} pigment and oil was obtained of the pigment & oil from the water slurry was obtained, the product being in the form of small ink pellets. Coalescing was obtained with RT-300-D but results were only fairly satisfactory. Partial coalescing only into pellet form was obtained with RT-379-D and results were considered unsatisfactory. Trials on ~~the~~ P.T.A. Colors, RT-330-D and GT-354-D were entirely unsatisfactory. While ^{the pigments} did eventually transfer, upon long stirring and some heating, from the water phase to the oil phase, it was not in the form of pellets but "gobs" of ink which would be very difficult to handle in a strike vat.

As orientation work last month showed that a boiling development gave distinctly better settling and consequently more

rapid coalescing than temperatures of 190°F or lower, the boiling development was used in ~~in~~ all experiments but the last (469-27).

A comparison of the results of eleven series run on RT-364-D shows that there has been some unexplained variations ~~in check~~ ~~and~~ in the settling time and stirring time as well as condition of the pellets in check runs from series to series. It should be noted here that time of stirring depends upon the number of times that the color is settled and water drawn off, for, the coalescing proceeds much more rapidly from a concentrated slurry. Therefore, any conclusions drawn at this time are only tentative and ~~will~~ must of necessity be verified (or not) by further experiments.

Following is a summary of the effect of the variables studied:-

Varnish Variable.

1. In the strike - Varnish alone - no heating after washing - no data.
2. In the strike - with 5% Mineral Spirits - no heating after washing. (17 C, D, & E)

Increase in Varnish gives smaller & more uniform pellets but requires somewhat longer stirring time in proportion to amount used.

3. In the strike - with 25% Mineral Spirits - no heating after washing. (18 E & F)

Increase from 25% to 100% of cuts the amt. of foam or color floating on the surface upon settling and gives finer pellets but otherwise has little effect.

4. In the strike with 5% Zinc Naphthenate suspension - no heating after washing. (23 C, D & E)

Increase from 25% to 62.5% has no effect; to 100%

gives slightly less dense settling and doubles the time required to coalesce into pellets.

5. In the strike - Varnish alone but with heating ^{to 60°C} after washing. (26 Series)

There is a distinct advantage in having some varnish in the strike. Density of settling appears to be directly proportional to the amount of varnish present in the strike. Otherwise, the amount of varnish in the strike appears to have very little effect on the final coalescing.

6. In total amount used when 25% along with 15% of Mineral Spirits is used in the strike - no heating after washing. (19 B, C & D)

Total of 50% of Varnish apparently is not enough to coalesce, 75% will coalesce into pellets with nearly twice the stirring time required for 100%.

Mineral Spirits Variable

1. With 25% of Varnish in the strike - no heating after washing (18 Series)

Mineral Spirits aids settling but 25% no better than 5%. Increase in amt. has little effect on stirring time but gives finer pellets in proportion to the amount used.

2. With 100% of Varnish in the strike - no heating after washing (Series 20)

Mineral Spirits ~~can~~ aids settling in proportion to the amount used, even up to 50% but lengthens the time of stirring in proportion to the amount used. It has little effect on the size of the pellets. No object in using any M.S. when 100% of Var. is used in the strike.

Zinc Naphthenate (5%)

(with 25% of Varnish in the strike)
Zinc Naphthenate, in all forms tried, aided settling and the final coalescing into pellet form. However, ^{the} previously precipitated form (to faintly acid on litmus) gave the best all around results.

In one experiment with no heating after washing 5% of Zn. Naph.

and 15% of Mineral Spirits (with 25% of Var in the strike) gave identical results. In a later experiment, which was heated to 60°C after washing, the same comparison was distinctly in favor of the Mineral Spirits although both gave excellent results. In this same series (25) 2% of Zn. Naph. was just as good as the 5%.

Processing Variables ~~at~~ after washing at time of adding 2nd amt. of Varnish.

Heating to 50°C gives slightly better incorporation of the varnish and shortens the stirring time slightly but otherwise has little effect. Heating to 60°C (Zn. Naph. was used in the strike) cuts the coalescing time 33% & gives very uniform 1/8" pellets.

Colloid milling after a very gentle premix was impractical as the color & varnish separated or flushed in the mill as an ink, clogging the mill.

Adding the varnish into the vortex of agitation caused by a "lightning mixer," ~~gave~~ and stirring for 15 minutes gave a very rapid and uniform incorporation of the varnish which coalesced very rapidly when stirring was reduced to normal. However, it is not known whether ^{you not} this went thru the pellet stage as it was not observed until after a large ball had formed.

Stirring overnight after washing with only 25% of Varnish present aids settling ~~settling~~ somewhat but ~~does not~~ has little effect on the incorporation of the balance of the varnish nor on the stirring time required to form pellets.

Miscellaneous

If settling troubles are encountered because of "up" settling or foam or color floating on the surface after flooding, it has been

found that this can usually be corrected by more or less stirring at flood volume with a slow rate of agitation.

Heating with a closed steam coil was found to be detrimental if anything. This should probably be checked again before definitely abandoning it.

Temperature of Development

One experiment was run in which ^{included development} temperatures of 150°F, 180°F and 212°F ~~with dry controls at each temperature~~ all with 100% of Varnish in the strike. The 150°F batch settled so sharply up that water was drawn from under the color. The 180°F ~~strike~~ ^{batch} settled both "up" & "down" but was made to settle down by stirring for 2 hours at flood volume. The 212°F batch of course settled down. All coalesced readily into pellets. As the experiment was designed primarily to study tinctorial effect a final report will be made next month.

Experimental Details

Volume No. 469

Series 9. Color RT-242-D. Orientation. Study of vat flushing. Adding varnish to soda salt before striking gave fairly good coalescing effect. The amount used apparently not critical as long as full amount is eventually added during the stirring process.

Series 10. Color RT-242-D. Extension of Series 9. Adding varnish ~~to~~ after the strike was completed gave unsatisfactory coalescing results; not as favorable as adding varnish to ^{the} soda salt.

Series 11. Color RT-330-D. Orientation study of vat flushing. Results negative.

Series 12. Color RT-300-D. Orientation study of vat flushing. Coalescing was obtained but not in a satisfactory form because of the large size of the pellets. Boiling development gave better settling and more rapid coalescing than 175°F development. Continued stirring at flood volume corrects up settling.

Series 13. Color RT-350-D } Orientation study of vat flushing.

Series 14. Color GT-354-D } Results negative. Does not coalesce with pellets upon stirring. These PTH pigments apparently take up varnish only after heating to 175°F or higher.

Series 15. Color RT-379-D. Orientation study of vat flushing. Results negative as did not coalesce upon stirring. Varnish on the strike improves settling in proportion to the amount present. Boiling development gives distinctly better settling than 160°F development.

Series 16. Color RT-283-D. Orientation study of vat flushing including effect of ^{fine} Naphthenate. RT-283 appears to be capable of ~~being~~ being flushed by the method.

Series 17. Color RT-364-D (boil). Comparison of effect of Ethyl Naphthenate and Mineral Spirits - Varnish variable with

mineral spirits - Effect of closed steam coil vs open steam.

Mineral Spirits ^(5%) (25% Var. in strike) gave a better settling than Ethyl Naphthenate (5%) but the latter showed better coalescing on the stirring process, altho neither was very satisfactory.

Increase of Varnish in the strike. Keeping the Mineral Spirits constant at 5% inhibited settling slightly ~~but gave smaller and more uniform pellets~~ and increased ^{the} stirring time, but gave smaller pellets and much more uniform coalescing.

Series 18. Color RT-364-D (boil). Check on 19C with Mineral Spirit and Varnish Variable.

The use of Mineral Spirits with 25% of Varnish in the strike aids settling materially. However 25% of M.S. is no better than 5%. Increasing the Varnish from 25% to 100% with 25% of M.S. improved settling slightly and also cut down the color and scum which tends to float on the surface.

The control (no M.S.) in the stirring operation, while completely coalescing ~~satisfactory~~, gave pellets too large to be practical; 5% M.S. was worse giving one large ball and incomplete ~~the~~ coalescing; 15% M.S. was about equal to control; 25% M.S. gave satisfactory coalescing with about 1/8 to 1/4" pellets; Holding M.S. at 25% and increasing the varnish to 100% gave the best results - a fine uniform pellet.

Series 19. Color RT-364-D (boil). Series based on 18 D varying the varnish content at time of stirring. Also effect of stirring after 1 water off before adding extra varnish. Also check on 18F.

100% Varnish in strike gave no improved settling this time over 25%.

Stirring overnight after 1st. water off (with ^{only} 25% of Varnish present) improved settling distinctly. Because of the more concentrated slurry, this sample (19E) also formed small uniform pellets first upon adding Var. up to 100% and stirring.

25% Var. + 15% Mineral Spirits in the strike, 2 waters off, 75% Var. added and stirring gave fairly good pellet formation. 19D (a check run on 18D) was slightly better in this respect than 18D.

A decrease in the total varnish in the final stir increases the time required to give smaller and more uniform pellet formation than 100% total varnish from a 25% Var. strike.

The 100% Var. strike gives the finest and most uniform pellet formation. ~~Increase in the amount of M.S. in this strike lengthens the time of stirring required to form pellets.~~

Series 20. Color RT-364-D (boil). Mineral Spirit variable with 100% of Varnish in the strike.

Mineral Spirits, used in conjunction with the Var., improves settling, particularly in the first water but slows up distinctly the coalescing into pellet formation. The size of the pellets eventually formed are little effected. ^{Stirring one hour after the first water flooding, before settling was found to be beneficial in reducing foam and color which tends to float and will be used in subsequent experiments.}

Series 21. Color RT-364-D (boil). Study of operating variables after striking with 25% of Varnish.

Simply adding the extra 75% of Var. (after washing 2 waters) and stirring is a workable process. However, this extra ~~later~~ 75% varnish addition is slow to incorporate and tends to remain in small globular form.

Colloid milling before the stirring period seems to be impractical as the ink flushes out in the mill, clogging the mill.

Heating to 50°C gives a very slightly better incorporation of the extra varnish and shortens the time of stir a little but otherwise has little effect.

Stirring overnight after 1 water off and before adding

the extra varnish, while aiding the initial settling had very little effect on the final stirring process.

Series 22. Color RT-364-D (boil). Study of effect of adding Sodium & Zinc Naphthenate in different

Cell forms of Na & Zn Naphthenates added to soda salt before the varnish (25%) aided settling and the final coalescing into pellet form. Previously precipitated Zinc Naphthenate (to faintly acid on litmus) gave the best all around results. (22 E)

Color RT-364-D (boil).

Series 23. Comparison of Mineral Spirits with Zinc Naphthenate (25% Var. in strike); a Varnish variable in the strike with Zn Naph.; comparison Zn Naph. with Var. only at 100% Var. in strike.

Mineral Spirits and Zn Naphthenate gave substantially check results. Increase in Var. ^{in strike} from 25% to 62.5% had practically no effect. Increase in Var. in strike to 100% ~~and~~ gave slightly less dense settling and doubled time required to coalesce into pellets. Zn Naphthenate in the 100% Var. strike gave more uniform pellet formation than the control without Zn. Naph. However, the control in this case was not as good as previous similar strike, 20 B.

Series 24. Color RT-364-D (boil). Manipulation variables after washing on 22 E type of strike.

Control with normal stirring was not satisfactory and did not check previous ~~report~~ results.

Heating to 60°C after taking 2 waters off & before adding the second portion of varnish (75%) gave very satisfactory and rapid coalescing and probably should be adopted as standard practice if ~~this~~ the 60°C temperature is not objectionable from a

functional standpoint.

Premixing the 75% of Var. with 10% of the washed slurry was unsatisfactory because the violent agitation of the soda mixer caused a flushing of that 10% of color into the varnish - i.e. did not disperse the varnish into fine particles. This resulted in large balls in the final stir.

Introducing the 75% of Var. into the entire batch with very violent agitation (electric mixer) appears very promising from an experimental viewpoint, as coalescing took place very rapidly when agitation was reduced to normal.

Colloid Milling in the 75% of Var. is apparently unsatisfactory as the color flushes in the mill.

Series 25. Color RT-364-D (boil). Comparative effect of methods of flushing, with & without assisting agents.

25% of Varnish alone in this series (25°C) was a good check on 24°C and the series as a whole again indicates that heating the concentrated, partially treated, washed slurry to 60°C before adding the balance of the varnish accelerates the coalescing into pellets.

15% of Mineral Spirits used as an "assisting" agent with 25% of Varnish in the strike gives ^{apparently} distinctly beneficial, compared to the straight 25% of Varnish and slightly better results than Zinc Naphthenate.

There is practically no difference between 2% & 5% of Zinc Naphthenate used as an "assisting" agent.

Series 26. Color RT-364-D (boil). Varnish Variable in the strike. No "assisting" agents. 24°C Manipulation.

Density of settling appears to be directly proportional to the

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amount of Varnish present in the strike. Otherwise, the amount of varnish in the strike appears to have very little effect on the final coalescing when the heating to 60°C procedure is used after washing. There does, however, appear to be a distinct advantage in having some varnish present in the strike.

Series 27. Color RT-364-D. Temperature of development variable with 100% of Varnish in the strike.

It appears from this experiment that any temperature of development on RT-364-D can be made to form pellets by the proper stirring procedure.