Serial No. KN-67-13
Copy No. 1

E. I. DU PONT DE NEMOURS & COMPANY
256 VANDERPOOL STREET
NEWARK, NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

ELIMINATION OF PIGMENT AGGREGATION DURING DRYING

Period Covered MARCH 1966 to JANUARY 1967

FILE: 114
DATE: 8/23/67

NJ 9346

KN-67-13

N33704

KN-67-13
Copy No. 1

1. Numerical File
2. Research Office File-Newark (114)
3. Newark Library File (114)
4. M.Hunt/E.Gonick, Pigments, Wilm.
5. W.S.Struve/A.A.Brizzolara
6. H.R.Linton/F.F.Ehrich/B.H.Perkins/Newark Library
7. P.J.Monahan, Newark (Vital Records)
8. N.G.Fisher, Central Research Dept., Wilm.
9. R.A.Hageman, Newark
10. Extra
11. Extra
12. Extra
13. Extra
14. Extra
15. Extra

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

SUBJECT: ELIMINATION OF PIGMENT AGGREGATION DURING DRYING

PERIOD COVERED: MARCH 1966 to JANUARY 1967

SUBMITTED BY: G. P. LEWIS DATE SUBMITTED: 7/14/67

APPROVED BY: H. R. LINTON DATE RELEASED: 8/23/67

ABSTRACT

The role of surfactants and pigment extenders, in addition to process and equipment modifications, outlined in KN-66-6 is explored. This report summarizes initial results of investigating non-aggregating drying techniques.

TABLE OF CONTENTS

INTRODUCTION

The tinctorial and physical properties of pigments are greatly influenced by the dispersibility of the pigment in the selected vehicle. With few exceptions, increased dispersibility yields superior products giving better gloss, strength, intensity and in some cases better lightfastness. Improving pigment dispersibility has been a continuing goal of the Colors Research group. This work has covered the role of surfactants and pigment extenders in addition to process and equipment modifications. Recent work in this area is best summarized by C. Manger (KN-66-6) in a report on pigment aggregation. A major conclusion of the report is that aggregation of pigment particles takes place during the drying process. Pigment particles drawn together in the wet state by surface forces cement or fuse to one another, as the moisture is removed during drying. These aggregates in turn require a considerable expenditure of energy for adequate dispersion in the vehicle, and in some cases cannot be reduced in size. The extent of aggregation may be illustrated by noting that with individual crystallites .1 micron or smaller, the average particle size after drying and size reduction may be 1-5 microns.

This work indicated that elimination of aggregation during drying could significantly increase pigment dispersibility. This report summarizes the initial results of a program to investigate non-aggregating drying techniques.

The program evolved after discussions with ESD and ERD personnel on February 28, 1966. The discussions centered around a research program to investigate the nature of pigment aggregation on an individual particle basis, or development program to evaluate several types of drying processes with the objective of minimizing aggregation during drying. It was felt by the Pigments Department that the latter program could lead to a successful process with a minimum expenditure of funds. If the development did not yield any leads, a basic research program would then be started. It was agreed that the work would be carried out by the Pigments Research group with ESD acting as consultant.

Mr. Paul McCormick of ESD provided advice on the program and we are indebted to him for his help in this work.

SUMMARY AND CONCLUSIONS

A program to investigate pigment aggregation during drying of "Monastral" Maroon RT-792-D has been completed. Drying techniques evaluated were rotary vacuum drying, fluid bed drying, spray drying, dielectric drying, freeze drying, vacuum belt drying, drying with agitation, and fluid energy combined drying and grinding. The fluid energy and fluid bed processes gave pigment particles with excellent dispersibility. However, the yield for the fluid bed was only 30%, and fluid energy drying and grinding was selected for the second phase of the drying study.

It was also shown during this work that dispersibility can be improved by simultaneous laking (CaSx) and extraction rather than the present method of extraction, isolation and subsequent laking.

A complete evaluation of the fluid energy mill to establish optimum performance conditions and to study the process economics is recommended for the next part of the pigment aggregation project.

PATENT SITUATION

No work of a patentable nature is expected to result from this program.

EXPERIMENTAL DETAILS

A. Preparation of Sample Material

Aggregation of pigments is a severe problem with many organic pigments because of their inherently small crystallite size. (.01 - .1 micron). The large surface area, the Van der Waals and surface tension forces, leads to many pigment agglomerates in the wet state. These agglomerates consist of many individual crystallites which cement or fuse together as dehydration takes place. It has previously been shown that the majority of aggregates found in the dry state are formed as the final amounts of water are removed. This occurs at moisture contents below 15% by weight.

The material selected for the experimental program was an organic quinacridone solid solution pigment, "Monastral" Maroon RT-792-D, with crystallites of .1 micron and below. This pigment is a 90% toner with calcium "Staybelite" as the extender. The metallic rosinate was formed by the reaction between sodium "Staybelite" and calcium chloride. This treatment has been shown to be effective in decreasing aggregation during tray drying. A portion of the tests was also carried out on RT-801-P. This is identical to RT-792-D but contains no metallic rosinate. RT-801-P is not sold commercially, but was included to study aggregation without the aid of the CaSx extender.

Prior to drying, the pigments are generally in the form of presscakes of 30-35% solids. The cakes, which have been compacted at pressures up to 100 psig, may contain agglomerates which are not readily dispersed. It was desirable to exclude this type of aggregation during the tests, for a more accurate evaluation of the drying methods. To accomplish this, the material was obtained from production as final vat controls. These are 4-5% by weight pigment slurries. The RT-801-P final vat control is the slurry after extraction of dispersion mill powder with dilute sulfuric acid. In production, this slurry is pressed and washed, reslurried and laked with CaSx. This final laking slurry is the RT-792-D final vat control. (FVC).

The FVCs were washed to 4000 ohms in the Semi-Works Shriver and concentrated to about 9% pigment solids. The Shriver dewaters by continuously flowing the slurry past a filter cloth and only a small quantity of water is removed per pass. In this manner continuous washing and thickening is obtained without the formation of a heavy presscake. The washed products were reslurried and dispersed in an Abbe-Lenart mixer. The batches were next homogenized at 2000-4000 psig. It was felt that these procedures would insure a good initial dispersion prior to the drying test. These operations are not an economical means of processing the material in production.

Preparation of Sample Material (Cont'd)

As controls for the experiment, presscake of RT-801-P and RT-792-D from the same lot was obtained. This material was reslurried in the Abbe and one half of the slurry homogenized.

Samples were taken, tray dried, pulverized in the five inch mill (SW) at high speed, and used for comparisons (control) against the experimental drying methods.

B. Spray Drying (NB-1831-9)

Spray drying was evaluated because of the rapid drying and low particle residence time in the system. Figure 1 is a schematic of a typical production spray dryer. Air (1), heated to 500-650°F by a direct gas fired furnace is used for dehydration. The feed (2) which must be a pumpable slurry is pumped to a centrifugal atomizing disc or a standard two fluid atomizing nozzle. Heat transfer and mass transfer are very rapid with the air and dried pigment emerging at the bottom (3) of the dryer and exhausting into a cyclone or dust collector. The temperature of the particles cannot exceed the boiling point of water (212°F or less) during the dehydration process and the final pigment temperature is regulated by the outlet air temperature. This is generally 150-200°F depending on the final moisture content desired. The test work was carried out using the Bowen Laboratory Dryer, with both a #5 atomizing nozzle and an ultrasonic nozzle. The feed was a 9% slurry delivered to the nozzle with a sigma-motor pump. Feed rates varied from 150 to 350 cc/min. Inlet air temperatures were 600°F and 800°F and outlet air temperatures ranged from 250°F to 400°F.

No problems were encountered during the test except for occasional plugging of the nozzle. The material did not cake on the wall and the drying chamber was lightly coated with fine dust. The product was very soft to the touch, and a moisture content below 1% was readily obtained.

C. Freeze Drying (NB-1831-11)

One of the most promising techniques uncovered in previous work on pigment aggregation was freeze drying. It was recognized during the previous program that physically separating the pigment particles during drying should decrease aggregation.

In the freeze drying process, the wet presscake is first frozen. The frozen cake is then placed in a suitable container which is evacuated to a pressure of 3mm Hg or lower. This is below the triple point for water. The frozen cake remains below 32°F as long as the pressure is less than 4mm Hg. Below this

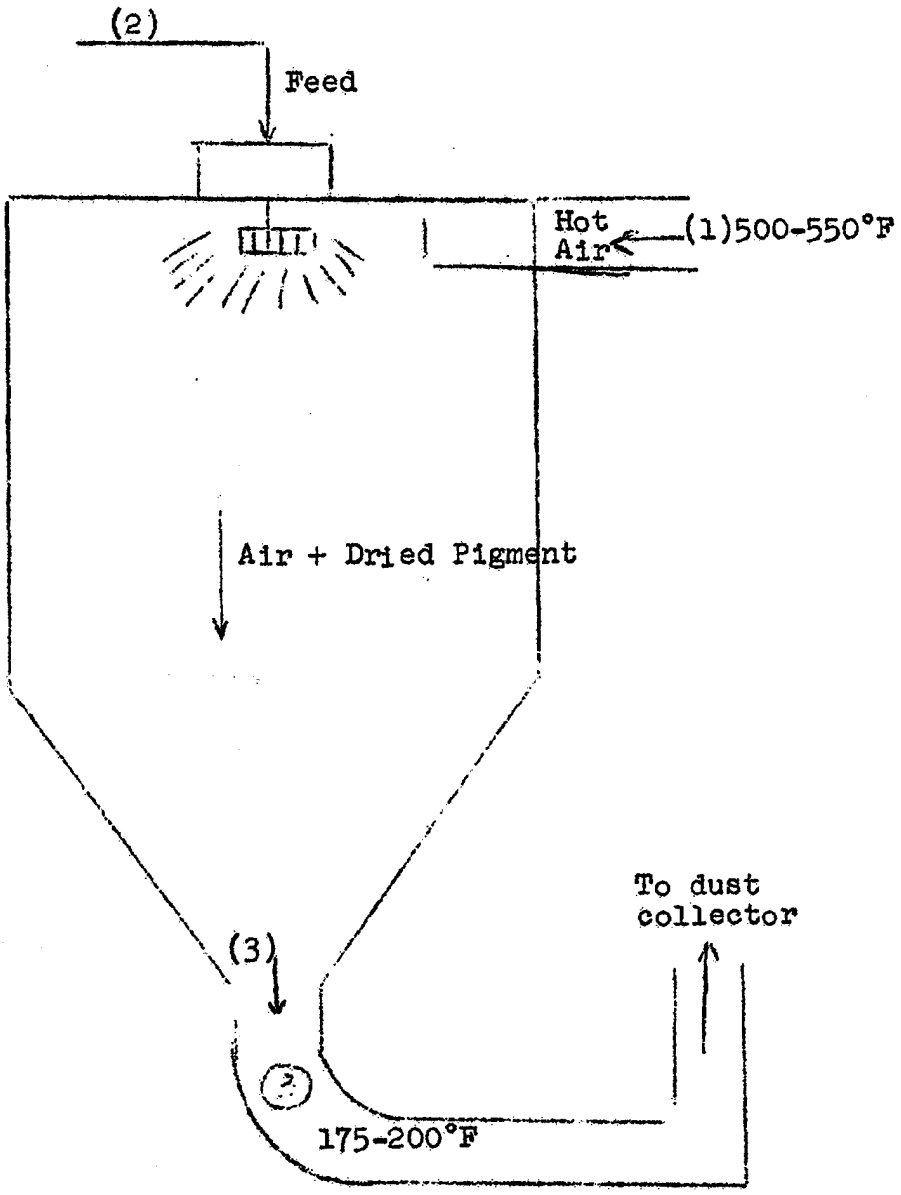

FIGURE 1 - SPRAY DRYER

Freeze Drying (Cont'd)

triple point water can exist in only two phases, solid ice or water vapor. As heat is applied to the vessel while under vacuum, sublimation of the ice takes place. The pigment particles are immobilized in the ice, while the ice is subliming, and do not contact each other. With minimum mobility and initial contact, little or no aggregation should take place. The freeze drying process is costly in terms of the low drying rate and the long residence time. In addition the process equipment is costly and requires many accessories for automatic processing. However, based on the excellent results obtained in the laboratory, the technique was included in this test program.

The feed samples were made up from the original 9% pigment solids material, by drying the slurries in a tray dryer at 200°F to 25% and 50% solids. Frequent agitation prevented local over-drying. A minimum solids content of 25% would be needed for an economical freeze dry process. Prior work showed only a slight decrease in dispersibility for freeze dried cakes with initial moisture of 50%. Samples at this level were also tested. The economic advantages for a process with a 50% feed compared to a 25% solids feed are very significant.

The tests were conducted at the Stokes Division of Pennsalt Chemicals using a small production size freeze dryer. The material was spread out on 8" x 8" steel pans, frozen overnight and then dried. The vacuum ranged from .1 mm Hg to 1.0 mm Hg. Five thermo-couples were inserted within each sample. Measured temperatures started at -20°C and continued to rise as drying proceeded. The dryer shelf temperatures were 25°C to 35°C. Vacuum was maintained throughout the night, and the dryer shelf temperature was auto-matically programmed to decrease in temperature with time. This insured that local melting would not occur, particularly at the bottom of the dryer pan. Little shrinkage of the cake after drying was observed and only slight dusting occurred. When dry the pig-ment was in the form of soft friable lumps.

D. Rotary Vacuum Drying (NB 1831-12)

The rotary vacuum tests were also conducted at Stokes using a one cubic foot capacity, steam jacketed and agitated dryer. The test was carried out since this drying technique is considerably more economical than freeze drying. It was felt that the rapid drying brought about by the high vacuum and the relatively high shell temperature coupled with agitation should decrease pigment aggregation. The sample for the test was the original 9% slurry of RT-792-D. Steam at 260°F was used in the jacket and a vacuum of 2.5 mm Hg was drawn on the vessel. The agitator was set at 6 RPM. As the slurry hit the heated wall, evaporation proceeded very rapidly with considerable foaming action. This foam helped keep the particles apart.

Rotary Vacuum Drying (cont'd)

As the solids increased, the foam broke and large lumps began to form. These lumps were carried up by the agitator screw and were subsequently broken up. Drying was continued for a total of 24 hours and the final product was powdery with very little lumps.

E. Thin Film Vacuum Belt Drying (NB-1831-26)

Following up on the evidence that rapid drying (regardless of the actual technique) is beneficial towards minimizing aggregation, the thin film vacuum belt dryer was evaluated. The dryer is manufactured by the Votator Division of Chemetron Corp., and the tests were conducted at their Louisville plant using a production size dryer.

A schematic of the dryer is shown in Figure 2. The dryer consists of a vacuum chamber, heating and cooling drums, and an endless flexible belt. Feed is introduced under vacuum suction into the feed pan. The feed must be in the form of a pumpable slurry. A roller deposits the slurry or paste in a thin film on the belt. The thickness of the film can be varied from several mils thick to one-sixteenth of an inch depending on the drying rate required and the adhesion of the material. Electric heaters and a steam heated drum are used for dehydration, and the moisture content along the belt may be controlled with these heaters. The product is cooled at the smaller drum and a knife edge scrapes the material off the belt and onto a screw conveyor. The product discharges into one of two air locks and is then packed out.

The belt speed can be varied from 20 to 50 FPM, giving residence times ranging from 8-10 seconds up to several minutes. The product generally is in the form of very light, fluffy flakes.

Fifteen runs were made using the original 9% pigment slurry as feed. The chamber vacuum was maintained at 13-15mm Hg with steam ejectors. No mechanical problems were encountered and moisture contents of .8 to 1.3% were obtained. The product discharged nicely from the belt and the tests indicated that high drying rates would be possible.

The combination of vacuum and high temperature generally results in a froth or foam which would aid in avoiding aggregation. The RT-792-D product did not however give a stable foam during the tests. Several runs were made using 3% carboxy methyl cellulose (CMC) additive to promote foaming, but the foaming characteristics of the material were still poor.

F. Dielectric Drying (NB 1831-13,17)

Dielectric drying was included in the test program because

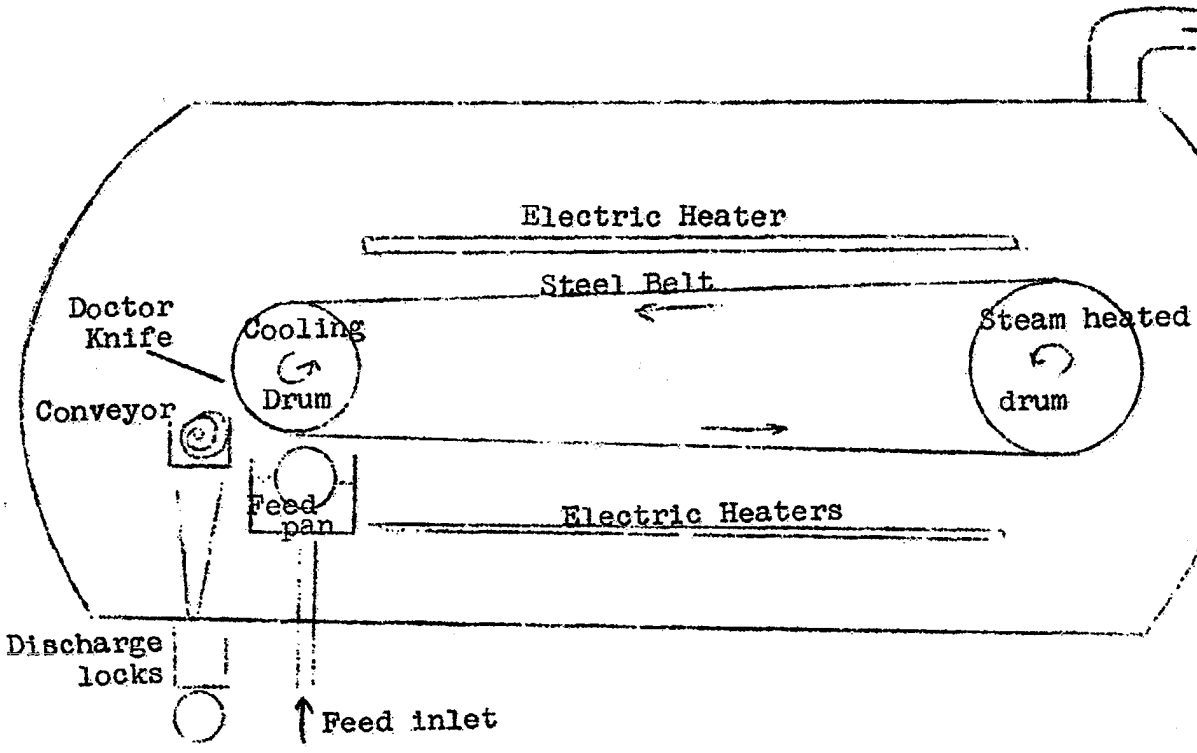

FIGURE 2

THIN FILM VACUUM BELT DRYER
(VOTATOR DIVISION)

Dielectric Drying(Cont'd)

it had the potential of alleviating batch production problems as well as minimizing aggregation. Dielectric radiation is electromagnetic energy with a frequency of 5-10 megacycles. Water is a good absorber of dielectric energy and rapid heating takes place. The quinacridone pigments do not absorb dielectric energy readily and therefore remain cool. When all the water has been evaporated, no heating of the pigment will take place even in the presence of the dielectric field. The rapid evaporation rate (compared to tray drying) should give a more dispersible pigment. The second advantage of dielectric drying is in the uniform internal heat generation. The water in the pigment is heated internally and the heat generation takes place regardless of the depth of the cake. This technique allows dehydration of a pressbox loaded with pigment several feet high. This in turn could eliminate the need for shoveling from the press box onto tray dryer pans. No crusting or hardening of the outer surface takes place in dielectric drying.

The tests took place in the 2KW batch dryer located at Orchem-Chambers Works, and in the 5KW & 30KW dryers of the Fitchburg Paper Company. The feed material was both 8% slurry and 25% presscake.

In the Orchem batch dryer, the sample was loaded in a 6" petrie dish to a depth of 1/2 inch. The separation between plates was 2" and the voltage difference was 1000 volts. A typical drying rate was 3 grams water per minute under these conditions. There was considerable frothing during the initial stages of drying. The samples underwent considerable shrinkage and cake cracking, and the final product was hard and lumpy and the product resembled tray dryer lump.

Using the 30KW dryer, rates of 20 gm H_2O/minute were obtained for the slurry samples while the rate for the presscake samples were 10 gm H_2O/minute. Apparently the more water present, the higher the average drying rate. The product from the Fitchburg unit was also very hard and gritty.

G. Drying with Agitation (NB 1831-19,24)

Most of the drying techniques previously tested required expensive equipment to produce vacuum or to generate specific types of energy. For the purpose of evaluating low cost techniques, drying with simple agitation was tried. The tests were carried out using the Semi-Works Dopp Kettle and the Baker-Perkins Mixer.

The Dopp Kettle is a vessel about four feet high and four feet in diameter, and is steam jacketed. The center shaft had 12 counter rotating paddles which revolved 180 degrees clockwise and then 180 degrees counterclockwise. Every other paddle rotated in opposite directions, and the paddle had spring loaded scrapers to wipe the walls. The feed was 8% pigment slurry. Severe foaming

Drying with Agitation (Cont'd)

took place during the initial drying stages. The test was terminated before complete drying because drying took place only at the walls which were heated by 15 psig steam, but the paddle retained wet cake which could not be dried.

The Baker-Perkins Mixer had a one gallon capacity. Steam in the outer wall jacket was used for heating. The mixer has counter rotating paddles much like an engine crankshift, which gives good agitation and scraping action on the wall. Foaming occurred initially and the pigment then formed large lumps which subsequently broke up during the final drying stage. The drying and final product characteristics were very much like the rotary vacuum dried material.

H. Fluid Bed Drying (NB 1831-30)

The fluid bed process was evaluated because it offered a combination of rapid thin film drying coupled with an elutriation of fine particles. The fluid bed concept tested is shown in Figure 3.

The dryer was a simple cylinder 18 inches in diameter, in which a bed of glass beads was heated and fluidized by hot air. The pigment slurry (8% solids) was sprayed onto the bed through an atomizing nozzle. As the pigment dried on the beads, the air stream carried the particles out of the dryer. A cyclone collector separated the heavy particles from the fines, which were subsequently collected in a dust collector.

The test was conducted at the Fuller Company Division of General American Transportation Corporation in Catasaqua, Pa.

The superficial fluidizing velocity was 6.1 ft/sec, and inlet air temperatures were 450°F to 560°F. This gave rise to actual bed temperatures of 250-300°F. The bed consisted of -12 to +20 mesh glass beads. The test proceeded well and the fluid bed principle was shown to be feasible from an operating standpoint. It was found that about 70% of the product came out in the cyclone with the remainder trapped in the bag collector.

The product was extremely powdery and soft with little or no grit obtained. This was a result of the rapid drying of the thin film which formed on each bead, coupled with the attrition occurring in the fluidized bed. The process looked quite attractive for a large quantity single product line.

I. Fluid Energy Drying and Grinding (NB 1831-20)

The final technique tested was a combined drying and grinding process using heated high pressure air. The testing was

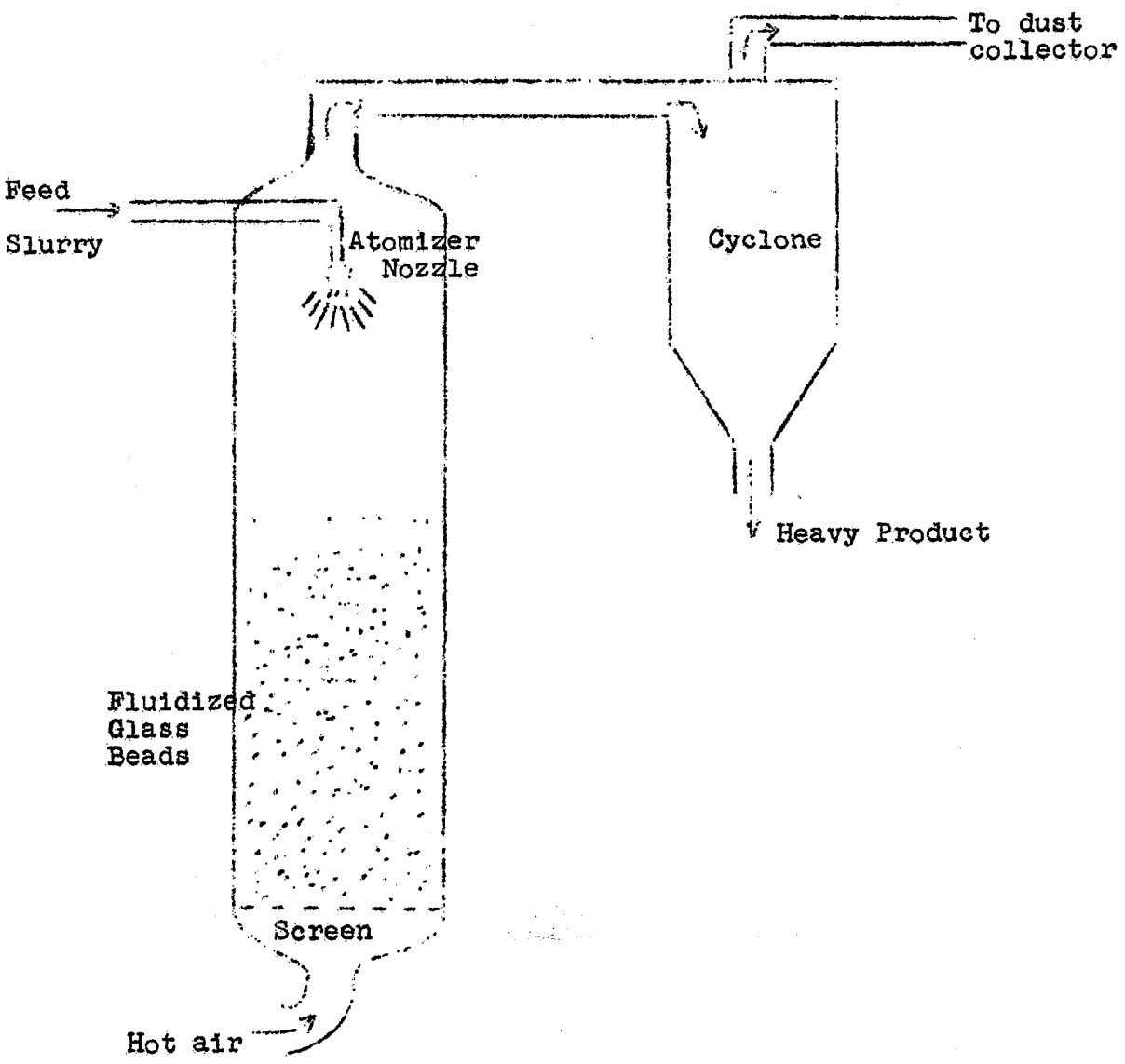

FIGURE 3

FLUID BED DRYER

Fluid Energy Drying and Grinding (Cont'd)

carried out at Fluid Energy Processing Company in Hatfield, Pa. A schematic of the dryer tested is shown in Figure 4. The drying action is very rapid due to the hot air and the large surface area of the particles. The pigment is fed as shown as a crumbly presscake or it may be pumped in as a slurry. Heated air or superheated steam enters through several nozzles with drying and grinding taking place in the first half of the unit. The classifier outlet is at the top and variable size separation may be achieved. Oversize particles recycle within the dryer and are reground down to the desired particle size.

The tests were conducted with a separate lot of RT-792-D presscake at 35% solids. The cake was dry enough to be fed to the dryer using a vibratory or screw feeder. Inlet air temperatures varied from 450 to 600°F while the outlet air temperature was maintained at 225°F. Inlet air pressures ranged from 20 psig to 100 psig. It is more economical to dry using low pressure air due to the large volumes required. Therefore, several tests were conducted where the drying was carried out at low pressure, and the product was reground in a smaller machine using high pressure air.

The product dried very well and the moisture contents were all below 1%. The dryer did not plug up with cake feed, and the particles were mostly less than one micron. Approximately 10 to 12 lbs. of air per pound of pigment were used for the grinding tests.

J. Dry Pigment Grinding (NB 1831-28)

An additional series of tests were conducted using the Engineering Test Center 2" micronizer. The work was carried out by R. Stuber for the purpose of investigating the quality of products ground with high pressure gas after previous drying. Helium gas was used for the tests in order to achieve high grinding fluid velocities. The dry samples submitted were lump RT-792-D and material previously dried by freeze drying, rotary vacuum drying, fluid bed drying and thin film vacuum belt drying.

K. Variations in Process Conditions (NB 1831-29,34)

It has been known for some time that coating of the "Monastral" maroon with calcium Staybelite decreases the aggregation on drying. If the pigment is not coated or laked in this manner, the product dries to very hard and gritty lumps, which cannot be broken down even after pulverization.

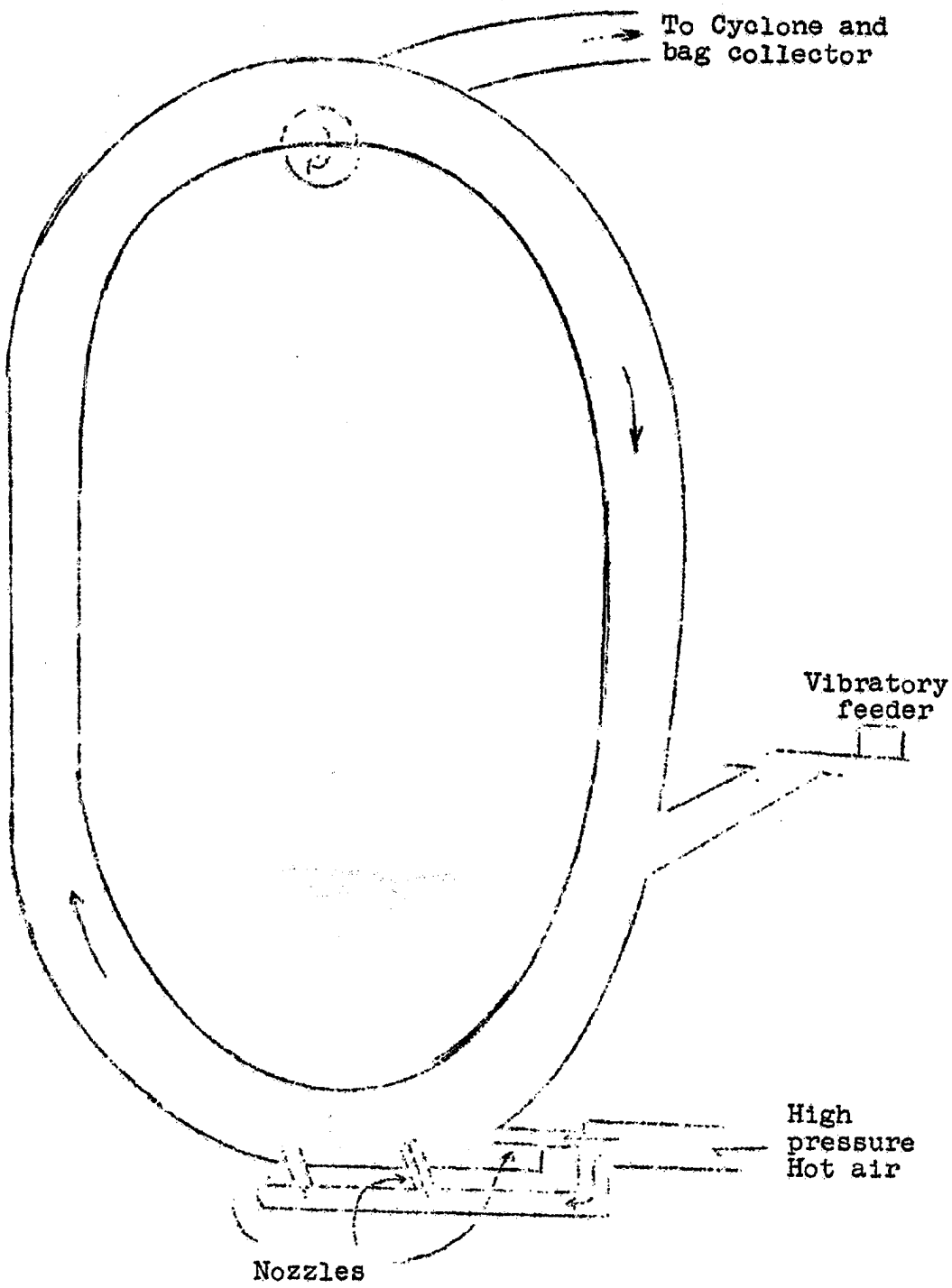

FIGURE 4

FLUID ENERGY DRYING AND GRINDING MILL

Variations in Process Conditions (Cont'd)

The present finishing technique for the "Monastral" maroon consists of:

(1) Dispersion milling of the crude pigment with alum;

(2) Extraction of the alum by dilute(5%) sulfuric acid;

(3) Pressing and washing in a plate and frame press;

(4) Reslurrying the cake in an Abbe-Lenart Mixer;

(5) Laking by addition of a sodium Staybelite-caustic solution followed by addition of calcium chloride to precipitate the metallic calcium Staybelite.

If the Staybelite is to be effective, it cannot be precipitated on pigment agglomerates or flocculants. The pigment must be extremely well dispersed prior to the Staybelite laking. It seems obvious that with all of the steps outlined previously, the particles have a good chance to agglomerate, decreasing the effectiveness of the laking process.

The pigment is in a state of excellent dispersion after the dispersion milling operation. It seemed advantageous to attempt laking with Staybelite at this point. The sodium Staybelite is a stable solution only in basic solution, while the alum-pigment mixture is acidic. A milling salt giving a basic solution is necessary to carry out the operation. Borax was chosen as the milling salt and both maroon QA crude and gamma QA crude were evaluated.

The process was carried out in the Semi-Works ball mill. The charge consisted of eight pounds of pulverized crude, 50 pounds of Borax, 580 gms of "Perclene" and 180 gms of Emcol P10-59, and 1000 pounds of cylpebs. Milling was carried out for 24 hours with samples being taken every four hours. The milling mixture was processed as follows:

> The Staybelite solution (44.5 gms) was added to 2.5 liters of water at 150°F. This solution was made up of 60 gms sodium Staybelite, 18 gms 50% caustic solution and 500 gms distilled water. To the 2.5 liter mixture 300 gms of the mill powder was added. "Teflon" coated bar magnets were placed in the jar to remove the iron chips formed during milling. The slurry was stirred at 150°F for 1/2 hour. A solution of one gram calcium chloride in 30 cc of water was added over a period of 15 minutes. The one gram of calcium chloride was the stoichiometric amount required to precipitate calcium Staybelite as 10% of the pigment weight. This laked slurry was stirred for 30 minutes at 150°F, followed by washing and filtration. The cake was tray dried and pulverized and freeze dried and pulverized.

Variations in Process Conditions (Cont'd)

A control experiment simulating plant operation was performed as follows:

> The mill powder was extracted with water only, then filtered and washed, reslurried in a sodium Staybelite solution, laked by the metallic rosinate precipitation and refiltered, washed, dried and pulverized.

RESULTS

A. Sample Evaluation

The samples were evaluated by the standard techniques. These included minimum and maximum work rubouts, 5/15 cut soft vinyl, and milling into 30J and 32J paint vehicles. The paints were evaluated as 5/95 tints, 10/90 metallics and in transparent drawdown. The rubouts indicate dispersibility of the pigment but they did not correlate well with the paint samples. The vinyls were a measure of the dispersibility of the product as received and do not necessarily indicate the quality of the product in other applications.

The most important application for the "Monastral" maroon is in automotive finishes. The systems are the 32J enamels and the high gloss "Lucite" acrylic lacquers. The primary effect desired is the intensity of the pigment, and the transparency of the metallic paint panel. This transparency is exhibited as the "flip-flop" effect, where the panel looks light when viewed head on, and goes dark when viewed at a low angle. A transparent film requires that the average particle size of the pigment particles are less than .4-.5 micron. In normal use the pigment must be ball milled or two roll chipped in order to achieve adequate dispersion.

It was felt that the test program would be highly successful if transparent paint panels were obtained by sand milling, since this size reduction technique is very economical for the paint manufacturer. Furthermore, the sand milling would allow a better comparison of the various drying processes. The level of work in the sand mill is much less than that in a ball mill and this greater expenditure of work could level out all the results so that all the processes would appear equally favorable or unfavorable. A laboratory sand mill requires up to 70 gms of pigment. We did not have this quantity available for all the tests. Therefore, sand milling techniques using only a few grams of sample were developed. The method finally used, was a Hochmeyer disperser with a glass disc to simulate the sand mill blades with a one pint stainless steel container and 20 mesh grinding sand. This operation resulted in product quality closely resembling the quality of the products obtained by normal sand milling.

The technique selected (NB 1831-15) was Hochmeyer milling with sand at 4000 RPM for 30 minutes. The grinding formula was:

5.0 gm pigment
16.7 gm Aeroset 777-60
22.2 gm Xylol
100 gm sand
Grind 30 minutes at 4000 RPM
Add: 11.1 gm Aeroset 777-60
11.1 gm Xylol
Stir well with spatula
Let the mill base down with composite in the ratio of two parts millbase/ one part composite.

Sample Evaluation (Cont'd)

The composite is:

Aeroset 777-60	139 gms
Melmac 243	179 gms
Xylol	13 gms

Evaluation was made by examining the millbase drawdown for transparency and by judging the "flip-flop" of the metallic panels. Strength comparisons were also made but this parameter was not considered critical.

Additional evaluations were also made by high speed mixing with the Hochmeyer. The same formula as above was used, but no sand was added as a grinding aid.

B. Comparison of Toner and Extended Pigments

Evaluation of all the samples of RT-801-P and RT-792-D were made from freeze drying and spray drying. The toner samples (RT-801-P, no CaSx) were inferior to the extended pigment (RT-792-D 10% CaSx) when dried by both techniques. The toner was hard and gritty and was not readily dispersed. This indicated that the extender was required regardless of the type of drying utilized. All other tests were carried out with RT-792-D only.

No meaningful differences were observed between the samples which were merely reslurried. A slight difference between the freeze dried samples at 25% solids and 50% solids was observed. The 50% solids material was inferior to the 25% solids sample. The difference however was slight and the economic benefits gained by drying from 50% solids outweigh the slight quality disadvantage.

C. Comparison of Drying Techniques

When evaluated in the 32J (sand milled) none of the samples were superior to a ball milled paint or to a CAB chipping for high gloss "Lucite" lacquer. The pigments will still have to be processed in the same manner. However, within the sand milled series, there were wide differences between the drying processes. Based on transparency and strength increase the techniques can be classified as given below:

Good:
- Equal to control
- Dielectric drying
- Thin film vacuum belt drying
- Drying with agitation
- Rotary vacuum drying

Comparison of Drying Techniques(Cont'd)

Better: Slight improvement over control
Freeze drying
Spray drying

Best: Significant improvement over control
Fluid bed drying
Fluid energy drying and grinding

The fluid bed and fluid energy techniques were outstanding in dispersibility. The "flip-flop" of the metallic panels was outstanding, and the strength and intensity were significantly improved. These processes definitely decreased aggregation during the drying process.

The drawback to the fluid bed process is low yield of high quality material. The best product was the fines obtained from the dust collector. The larger particles which came out in the cyclone were defensive in quality compared to the control. The yield of fines in the dust collector was only about 30%, and even with a better collection system the yield would be too low for economic justification.

The fluid energy process resulted in 100% yield of high quality pigment, because of the internal recycling of oversize particles in the mill.

In view of the results obtained, the fluid energy process gives a more dispersible product, yielding higher gloss, with better transparency and strength.

D. Preliminary Fluid Energy Cost Estimates

Following the evaluations and selection of the fluid energy mill, preliminary costs were obtained for several combinations of drier-grinders. These are given in Table 1. Operating costs for heat and power range from $.51 to $2.4 per cwt, while capital investment varies between $100,000 and $200,000. The combinations in the table are:

a) Single stage - a combined drier-grinder where simultaneous drying and grinding takes place using high pressure air.

b) Double stage - a low pressure drier followed by a high pressure grinder. Two inlet feed solids contents are presented for each combination, to show the effect of initial dewatering. The first two stage combination with 10 psig air in the drier and 100 psig air in the grinder reflect conditions tested. The last two stage combination with 3 psig air in the

TABLE 1

APPROXIMATE FLUID ENERGY OPERATING AND CAPITAL COSTS

PIGMENT PRODUCTION FOR 500 LB./HR. OF FINISHED DRY PRODUCT

NOTE: ALL VALUES ARE APPROXIMATE

	ONE (1) STAGE		TWO (2) STAGE (A)		TWO (2) STAGE (B)		TWO (2) STAGE	
	Drier (A)	Grinder (B)	1st Stage Drying	2nd Stage Grinding	1st Stage Drying	2nd Stage Grinding	1st Stage Drying	2
Feed Solids %	32%	64%	32%	100%	64%	100%	32%	
Feed Moisture %	68%	36%	68%	Trace	36%	Trace	68%	
Total Feed Rate	1565#/hr.	780#/hr.	1565#/hr.	500#/hr.	780#/hr.	500#/hr.	1565#/hr.	5
T_1 at Mill Intake	500°F	500°F	500°F	225°F	500°F	225°F	500°F	
T_2 at Collector Exhaust	225°F	225°F	225°F	175°F	225°F	175°F	225°F	
P_1 Pressure Mill Manifold	50 psig	50 psig	10 psig	100 psig	10 psig	100 psig	3 psig	1
Mill Type	Grinder-Drier	Grinder-Drier	Jet-O-Drier	Jet-O-Mizer	Jet-O-Drier	Jet-O-Mizer	Jet-O-Drier	Jet-
SCFM (Approximate)	4600	1350	4600	700	1350	700	4600	
HP, Compressor Blower (Approximate)	625	183	276	123	81	123	101	
Gals./hr. for heating air (#2 oil - 143,000 BTU/lb.) Indirect Fired Heater	20.4	6	20.4	1.25	6	1.25	20.4	1.
*Power & Heat ¢/lb.product	1.85	6.54	1.37		0.62		0.98	
Power & Heat Total Cost/yr. based on 3,000,000#/hr.	$55,700.00	$16,150.00	$41,100.00		$18,600.00		$29,400.00	
TOTAL COST OF EQUIPMENT	$123,900.00	$79,500.00	$135,900.00		$109,000.00		$133,900.00	

*Oil at 11¢/gal., Electricity at 1.5¢/KW hr.

drier represents a recommended operating condition yielding low operating cost and a reduced capital investment. Subsequent to this report, this combination was tested, and found to be satisfactory.

E. Dry Grinding Evaluations

The work done using the two inch micronizer on helium gas showed that grinding can yield a highly dispersible product. However thermal degradation of the pigment occurred due to the high total temperature associated with the high velocity gas. The pigment was quite blue and in fact some of the material fused on the micronizer walls.

F. Comparison of Laking Techniques

The samples of the QA maroon and the QA gamma crudes dispersion milled with Borax and simultaneously extracted and laked with CaSx were evaluated by rubout, 5/15 vinyl and in 32J sand milled and ball milled paints. A significant improvement in intensity, strength and transparency was obtained compared to the control which was first extracted, filtered and then laked. These experiments show that laking of the pigment in its most dispersible form (mill powder) improves the effectiveness of the Staybelite in decreasing aggregation.

The best products were obtained at mill times of 12-16 hours. With less milling, the particles remain sufficiently large so that weak, dull products are obtained. Longer mill times result in a very small average particle size with a correspondingly large surface area. This material aggregates severely on drying because of the large surface area, again resulting in a weak, dull, and non-transparent product. The process change gives a superior pigment, but the technique has several disadvantages. First, the Borax grinding aid will cake in the mill at temperatures of 140-150°F. In the Semi-Works it was possible to keep the mill temperature at 90°F with cooling water, while in the plant the mills run at 120°F-140°F even with refrigeration cooling. Secondly, the requirement of an alkaline environment for the laking means that magnets must be used to eliminate iron particles. It is not certain that this can be done in a large scale operation. Finally, some of the QA products will be HT drowned rather than dispersion milled and the quantity of pigment remaining to be dispersion milled may not warrant further work.

RECOMMENDATIONS

A. Fluid Energy Drying and Grinding

The fluid energy process has been shown to offer significant improvements in pigment dispersibility. Work in this area should be continued, with evaluation of all of our organic pigments. This program has been started by D. Eastham at Newport using a small mill. The purchase of a larger mill (2-1/2") is recommended for the Newark Semi-Works. This will allow rapid evaluation of many codes as well as the establishment of optimum drying and grinding procedures. The total cost for this project should not exceed $20,000.

B. New Laking Techniques

The process of simultaneous extraction and laking also yields a product superior to the control sample. The continued use of dispersion milled products should be re-examined and if the quantity of pigment is sufficient, the new laking technique should be revaluated. Furthermore, all process operations where a reaction takes place which requires a dispersed material, should be examined to determine the place at which the best pigment dispersion can be obtained. The reaction should take place at this point in the process, if possible.

GPL:mm