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E. I. DuPont de Nemours & Company

# LABORATORY STUDY OF "AFFLAIR" FILTRATION

## PROGRESS REPORT

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PREVIOUS RELATED REPORTS: None

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## ABSTRACT

Laboratory and pilot plant evaluation of the "Dart" automatic pressure filter showed unacceptable levels of damage to the $TiO_2$ coating of "Afflair" presscake. The rotary drum vacuum filter laboratory evaluation was promising for use in a future plant expansion. Installation of a new rotary kiln at Newport would be less expensive and more productive than installing an acid resistant rotary dryer and using the current Edge Moor rotary kiln.

## Introduction

• Last July, there was interest in expanding "Afflair" production by increasing the throughput capacity of the Edge Moor kiln. It was estimated that half the heat load on the kiln went to dry the roughly 50% water from the presscake. Complete drying of the presscake prior to feeding it to the kiln might double the kiln's current capacity of 1.5 million pounds (calcined basis) per year.

• Variations of presscake solids content between 45% and 65% have led to fluctuations in the completeness of calcination. Severe feed problems occur at the lower solids content. One benefit of dry kiln feed would be easier handling and more consistent high quality product from the kiln.

• Manpower needs for dumping the plate and frame presses now used for "Afflair" are not evenly distributed. The presses take about 12 hours to load and to wash the presscake. This takes little manpower. Dumping the presses takes four men half a shift. An automated filter would make the process more continuous and would eliminate fluctuations in scheduling manpower.

## Objectives

• To determine the suitability of the "Dart" automatic filter press for producing "Afflair" presscake of high solids content and good quality for feed to the Edge Moor kiln.

• To investigate the feasibility of using a rotary drum vacuum filter press to attain high solids content presscake, to operate on a continuous basis, and to reduce manpower requirements.

• To consider other alternatives for reducing kiln feed and calcination problems associated with low solids presscake.

• To provide throughput, scale-up, and economic information for design and economic evaluation of the above processes.

## Summary and Conclusions

- The "Dart" filter press damages the $TiO_2$ coating on "Afflair" flakes and is not suitable for plant use. The plant could use an automatic filter press which would not put mechanical stress on the filter cake.

- Rotary drum vacuum filtration is a promising method for continuous processing of "Afflair". Replacement or expansion of present filtration facilities with two polypropylene rotary drum vacuum filters in series with a repulp tank should be considered if present facilities become inadequate. The bare equipment costs (mid-'75) of two 60 $ft.^2$ rotary filters with a repulp tank is $100,000. This would be adequate for 4 million pounds (dry basis) per year throughput (currently 2.2 million).

- Installation of a new rotary kiln at Newport would require less investment than installing a new, acid resistant rotary dryer (bare equipment cost of $150,000 *vs.* $200,000 in mid-'75). The new kiln could be designed to both dry and calcine the material with better control of product quality than is possible with the presently used Edge Moor kiln. This would expand throughput capacity from the present 1.5 million pounds per year to 3.5 million.

## Patent Situation

No patent proposals have been submitted or planned.

## Program

No further work is planned at this time. If the rotary drum vacuum filter is to be considered further, more extensive testing with plant slurries would be required to optimize filtration conditions and to get more precise estimates of plant scale equipment required.

## Publication Status

Material is not suitable for publication.

## Special Safety Precautions

None beyond the usual precautions for handling "Afflair" hydrolysis slurry (60°C., 12% $H_2SO_4$ solution) and working with pilot plant scale mechanical equipment.

## Environmental Considerations

Effluent to sewer contains sulfuric acid, dissolved solids, and suspended solids. Iron oxides, titanium dioxide, and mica are the principal solids involved.

## Acknowledgement

The assistance of W. H. Severns, D. S. Watson, R. J. Wilber (of Dart Industries), and J. E. Flood in arranging for test facilities and in interpreting test results was greatly appreciated.

Table of Contents

"Dart" Press Operation

The Dart-Hoesch automatic pressure filter is based on a vertical stack of filter plates, each of which contains

a filter cake chamber

a compression membrane (separating two chambers)

a hydraulic pressure chamber

a filtrate discharge chamber

Figure I shows the arrangement and a sample filtration cycle suitable for the "Afflair" operation. Under manual or timer-relay control, the filter press

pumps slurry into the filtercake chamber

compresses the cake lightly

pumps wash water into the filtercake chamber

compresses the cake at higher pressure

admits compressed air into the filtercake chamber

opens the plate stack and advances the filtercloth to discharge the filtercake

Hydraulic pressures up to 250 psig can be applied to the filtercake. The filtercloth is a single long sheet with the ends zippered together to form a continuous loop of cloth. During filtercloth advance at the end of the cyle, the filtercake falls off the cloth into bins beside the press and the cloth is washed (if desired) in a separate tank.

The bench scale press, model MP-300, has a single square of filtercloth (0.04 $M^2$ or 0.43 $ft.^2$ in area) and is filled by "blowcasing" (air pressurizing a sealed feed tank). The pilot plant press, model P-2.5, has five plates with a total filter area of 2.5$M^2$ or 27 $ft.^2$. Plant scale presses include the model P-25, with 25$M^2$ or 270 $ft.^2$ of area and a mid-'75 bare equipment cost of $325,000.

"Dart" Press Test Work (NB-E-6309, pp. 3, 21, 29, 31, 33, 36, 48, 63, 64, 68)

Tests 1M and 2M were run on the MP-300 press. D. S. Watson brought some NF-140, batch 40-32 to Paramus, N.J. on July 10, 1974. The NF-140 is a deep gold and does not have any acid to be washed out, just salts. The press achieved 77% solids and gave a projected production rate of 1000 pounds per hour for the model P-25. We need only 270 pounds per hour to get enough material for sales of 1.5 million pounds per year. The 20% excess capacity is required to make up for product yield loss and to wash material prior to deep gold treatment. See Table I for data.

Tests 1 through 4 were run at Newport on the P-25 press under my supervision on December 18 and 19, 1974. Slurry from batch 8-119 was taken right after dilution of the hydrolysis mixture. During the test, the slurry was kept stirred by re-circulation using the 20 hp centrifugal feed pump for the Dart unit. Overnight, the circulation was off and an air powered stirrer was used to keep the slurry from settling out. The results of tests 1-4 became progressively lower in Total Specular Reflectance (TSR). A control sample, 3' taken from the press feed tank before test 3 showed that the pressed material was lower in luster than the unpressed material and that both were lower in TSR than the control taken before any material had gone through the pump or press. Damage by multiple passes through the pump as well as by the press was indicated. Solids contents for the NF-108 presscakes were 66-78%, the best values resulting when a hot water wash and a high cake compression pressure were used. A production rate of 680 lbs. per hour was projected for the P-25 press.

Tests 3M, 4M, 5M were run January 17, 1975 in Paramus, N.J. on the MP-300 press. I brought 20 gallons of NF-152 (a large flake pearl) slurry with me from batch 52-18. Good quality material with solids content of 83% was achieved. Replicate tests by Dart the following week (6M, 7M, 8M) gave similar results. The ratio of wash water to initial slurry water was only 1.7 here compared to plant practice of about 7.

Tests 9M, 10M, 11M were run by Dart in Paramus on February 21, 1975 using the MP-300 press. The slurry was a deep gold NF-140, batch 40-43, which had been treated in January and held by Dart for about a month. Newport's past experience indicates that this storage does not affect the material adversely. The wash ratio used was low and a re-wash in our laboratory improved the TSR of the product pressed by Dart to nearly the control value. The Dart filter cake was 72% solids.

Tests 12M, 13M, 14M were run by Dart at Paramus on February 25, 1975. Slurry from NF-157 (wet classified mica coated with $TiO_2$ and later deep gold treated), batch 57-8, which had been stored by Dart for about a month, was used. The wash ratio used was only 0.7 in one case, and the TSR improved markedly on rewash, but not close to the control. Opacity was also greater than for the control (laboratory filtered and washed material from the same slurry after a month of storage). A 74% solids content was achieved.

The Dart press is designed to increase percent solids in presscake by squeezing out presscake water. While it did improve percent solids for "Afflair", the decrease in luster and increase in opacity are unacceptable at the pressures used in these tests. While the press could be run at low pressures to avoid flake damage, this would not appreciably increase percent solids. It would also use an expensive press capable of high pressure operation where a less expensive low pressure automated filter press could be used.

Laboratory Work on Flake Damage (NB E6309 pp. 45-47, 76-77)

To more fully evaluate the damage caused by pressure applied to the presscake, we formed 1/2 inch thick presscakes inside a 1.2 inch diameter "Delrin" die, placed it in a laboratory press and applied loads of 250 to 10,000 pounds on the 1.13 square inch area. The drop in TSR is clearly evident as applied pressure increases. Electron micrographs of damaged flakes show displacement or loss of large areas of the $TiO_2$ coating. Damaged flakes show up in increasing numbers and coating loss is greater for the higher pressure treatments.

Table II gives the data for the laboratory pressure treated samples.

Rotary Filter Feasibility Study (NB E-6309 pp. 50, 66, 67, 68; NB E-8370 pp. 4, 5, 12)

Filter pads may be used to determine the best conditions for rotary vacuum filtration and to estimate the quality and scaled-up throughput of rotary drum vacuum filters. For details, see the letter to R. H. Wetzel from O. E. Ringwald, November 29, 1972, based on J. E. Flood consultation. A one-hundredth square foot stainless steel Bird-Young filter pad was used for this study. The filter cloth presently in use by the plant worked well.

Slurry was picked up on the filter, washed and air dried (by pulling air through the filtercake) for varying lengths of time to determine optimum conditions of operation and to estimate the size of plant scale equipment. Lot 38-28 (a high luster pearl) slurry contained 0.3 pounds of solids per gallon and was filtered at 60°C. Wash water was 20°C. tap water. Line vacuum was used only when a vacuum gage showed between 20 and 25 inches of mercury vacuum to be present in the line. No bleed through the cloth was observed. Lot 40-43 (a deep gold) slurry also filtered well at 2.7 pounds solids per gallon.

The filter pad assembly readily produced a thick (1/2") cake of reasonable porosity (1.35 GPM/Ft.$^2$) in a short time (30 sec.). "Afflair" presscake should have a soluble salts content of under 1% (dry basis) after washing. Higher values adversely affect product quality by creating sintering during calcination and by contributing their coloring to the product. A single filtration and wash did not reduce the salt content below 1% in a reasonable length of wash time, so a repulp, a second filtration, and a second wash were added. This is equivalent to using a repulp trough and a second rotary drum vacuum filter in series with the first.

Graphs of the data for Lot 38-28 are given in Figure 2. For a two minute cycle with 25% pickup time, a filtration rate of 17 pounds solids per hour per square foot of filter area was achieved. Lot 40-43 gave 4.8 times this throughput rate at 9 times the solids concentration in the slurry. Typical hydrolysis slurry after dilution contains 1.2 pounds per gallon, so a rate of 25 pounds per hour per square foot can be projected as a conservative estimate. A 50% scale-up factor reduces the throughput for plant sizing purposes.

For 1984 projected "Afflair" sales, the required filter capacity would be 2.6 million pounds per year, requiring a 32 ft.$^2$ filter at 75% utilization. J. E. Flood has priced a 6 foot diameter by 4 foot length (60 Ft.$^2$ filter area) polypropylene rotary drum vacuum filter at $40,000 each (bare equipment value). Two of these filters with an intermediate repulp tank would be required.

The rotary drum vacuum filter would reduce manpower requirements somewhat, but would not improve solids content of the presscake beyond the 45-65% now obtained with a plate and frame press. There is little economic incentive for replacing the present filter presses with a rotary drum vacuum filter at this time.

Predryer Feasibility Notes

An alternate solution would be to install a pre-dryer at Newport. This should reduce kiln feed handling problems, improve kiln temperature control, and increase kiln capacity for calcination. Justification of the cost would be based on improved quality and kiln capacity (fewer shifts of operation could handle the same throughput). The improved quality would result in a higher effective yield, since less material would have to be blended off in low quality grades or discarded.

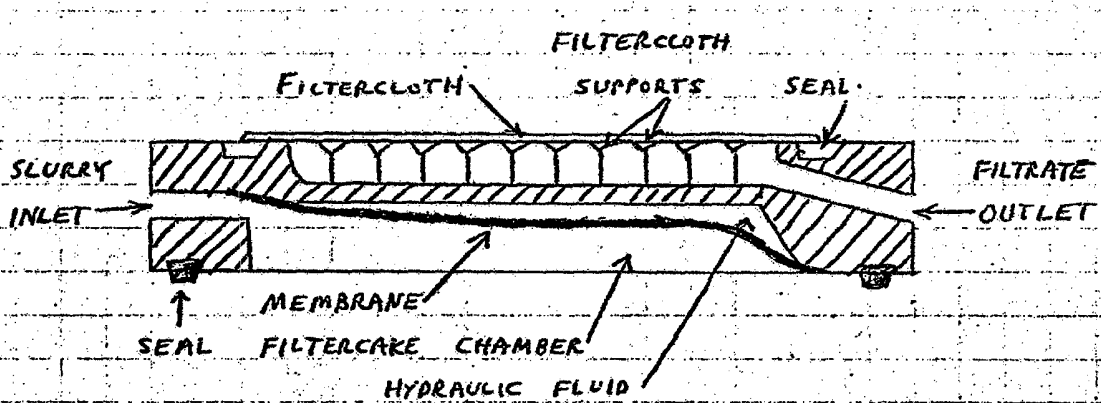
P. Y. McCormick (our kiln consultant from Louviers) has priced a Bartlett-Snow pre-dryer 4 feet in diameter by 25 feet long (enclosed heated length) with a design rate of 500 pounds dry basis per hour at $200,000.

Since a pre-dryer would cost more than a new, higher capacity rotary kiln ($150,000), it might be preferable to accept the feed and quality problems in return for a lower investment outlay.

References

None.

# DART - HOESCH AUTOMATIC PRESSURE FILTER PLATE




## SAMPLE CYCLE

I. 8 MIN FILL TO 40 PSIG, 110°F, SLURRY 1.5#/GAL, 1" CAKE

II. 5 MIN CAKE COMPRESSION TO 67 PSIG

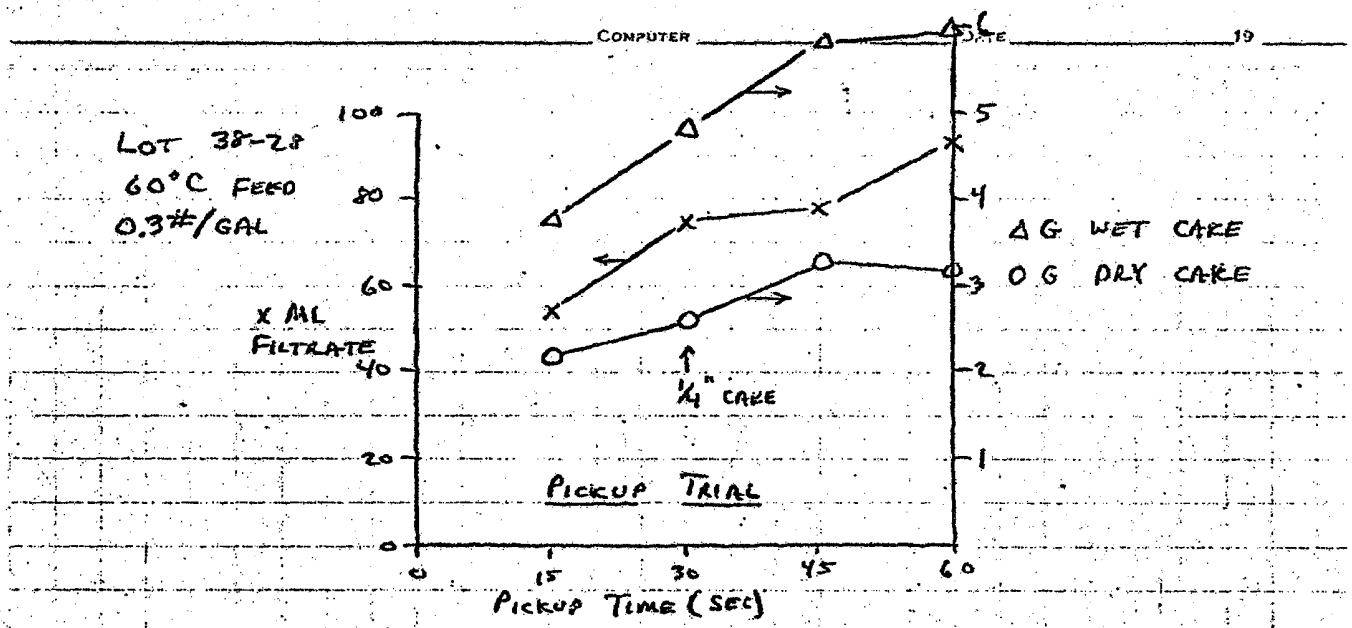
III. 30 MIN WASH AT 70 PSIG, 110°F, 7 TIMES FILTRATE VOLUME

IV. 3 MIN FINAL EXPRESSION TO 170 PSIG

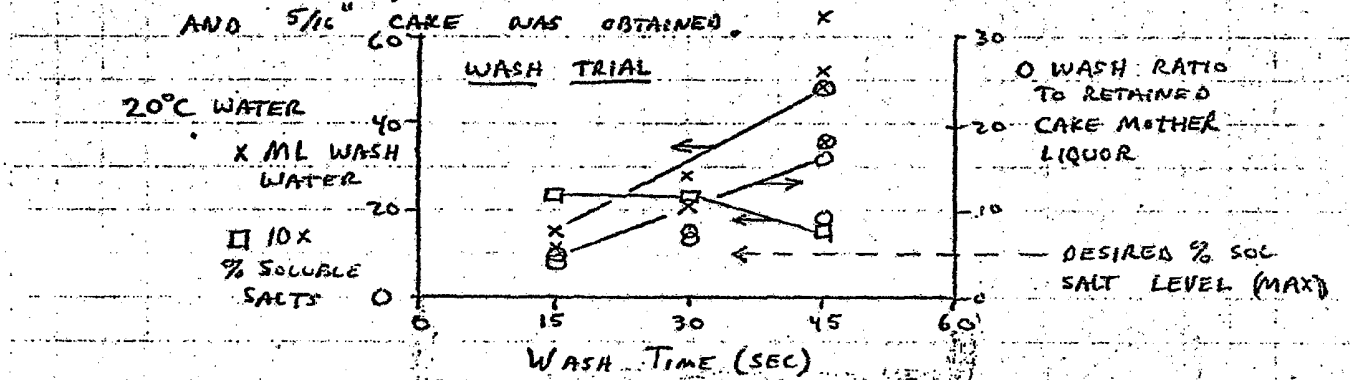
V. 3 MIN AIR BLOW AT 50 PSIG

VI. 3 MIN. CAKE DISCHARGE + FILTERCLOTH ADVANCE, 75% SOLIDS

FIG. 1 — FILTER PLATE DETAIL, CYCLE PARAMETERS FOR "DART" PRESS




FOR WASH TRIALS, 30 SEC PICKUP TIME WAS USED, 3½ G DRY BASIS AND 5/16" CAKE WAS OBTAINED.




FOR DRYING TRIALS, 30 SEC PICKUP, 30 SEC WASH.

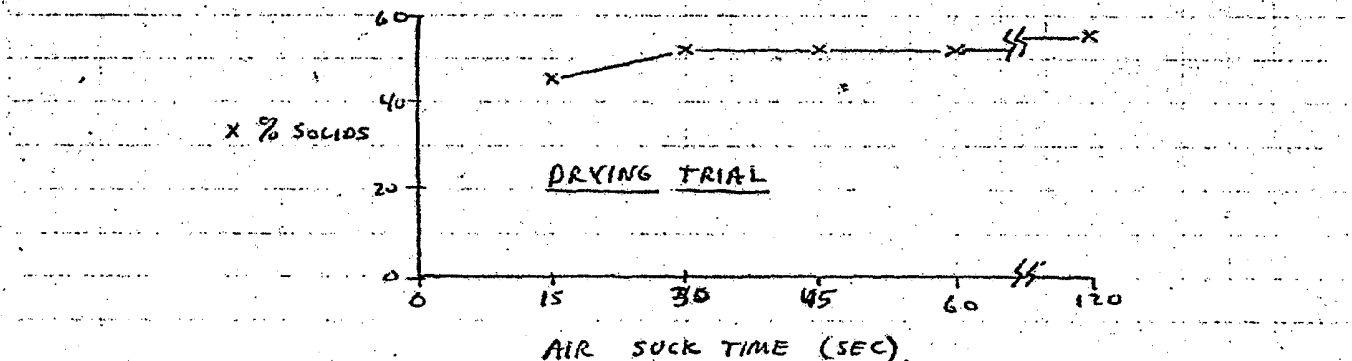



FIG. 2. ROTARY FILTER - FILTER PAD TEST RESULTS.

TABLE I

DATA FOR TEST SAMPLES RUN ON THE DART PRESS,
LABORATORY PRESSED CONTROLS, AND PLANT STANDARDS

| Grade /Batch | Test No. | % Soluble Salts | Total Specular Reflectance | Control | Standar |
|---|---|---|---|---|---|
| NF-140/32 | | | | | 408 |
| | 1M | nm | 170 | None | |
| | 2M | nm | 146 | " | |
| NF-108/119 | | | | | 632 |
| | 1 | nm | 481 | 512 | |
| | 2 | nm | 463 | " | |
| | 3 | nm | 443 | " | |
| | 3' | nm | 477 | 528 | |
| | 4 | nm | 432 | 512 | |
| NF-152/18 | | (0.01 Control) | | | 974 |
| | 3M | 0.11 | 902 | 919 | |
| | 4M | 0.06 | 989 | " | |
| | 5M | 0.06 | 962 | " | |
| | 6M | 0.10 | 944 | 954 | |
| | 7M | 0.11 | 947 | " | |
| | 8M | 0.07 | 967 | " | |
| NF-140/43 | | | | | 408 |
| | 9M | nm | 303 | 318 | |
| | 10M | nm | 292 | " | |
| | 11M | 0.44 | 288 | 326 | |
| | 11M Rewash | nm | 311 | " | |
| NF-157/8 | | | | | 506 |
| | 12M | 0.12 | 366 | 445 | |
| | 13M | nm | 373 | " | |
| | 14M | 0.25 | 390 | 476 | |
| | 14M Rewash | nm | 413 | " | |

(nm means not measured)

TABLE II

DATA FOR LABORATORY PRESSURE TREATED SAMPLES

| Grade /Batch | Die Pressure (PSIG) | Total Specular Reflectance | % of 0 PSIG TSR |
|---|---|---|---|
| NF-152/18 | 0 | 918 | 100 |
| | 2200 | 707 | 77 |
| | 4400 | 494 | 54 |
| | 8800 | 350 | 38 |
| NF-138/28 | 0 | 845 | 100 |
| | 220 | 684 | 81 |
| | 440 | 619 | 73 |
| | 880 | 568 | 67 |
| | 2200 | 440 | 52 |
| | 4400 | 373 | 44 |
| | 8800 | 301 | 36 |

Indexing Terms

"Afflair"

Automatic filter press

Rotary drum vacuum filter

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