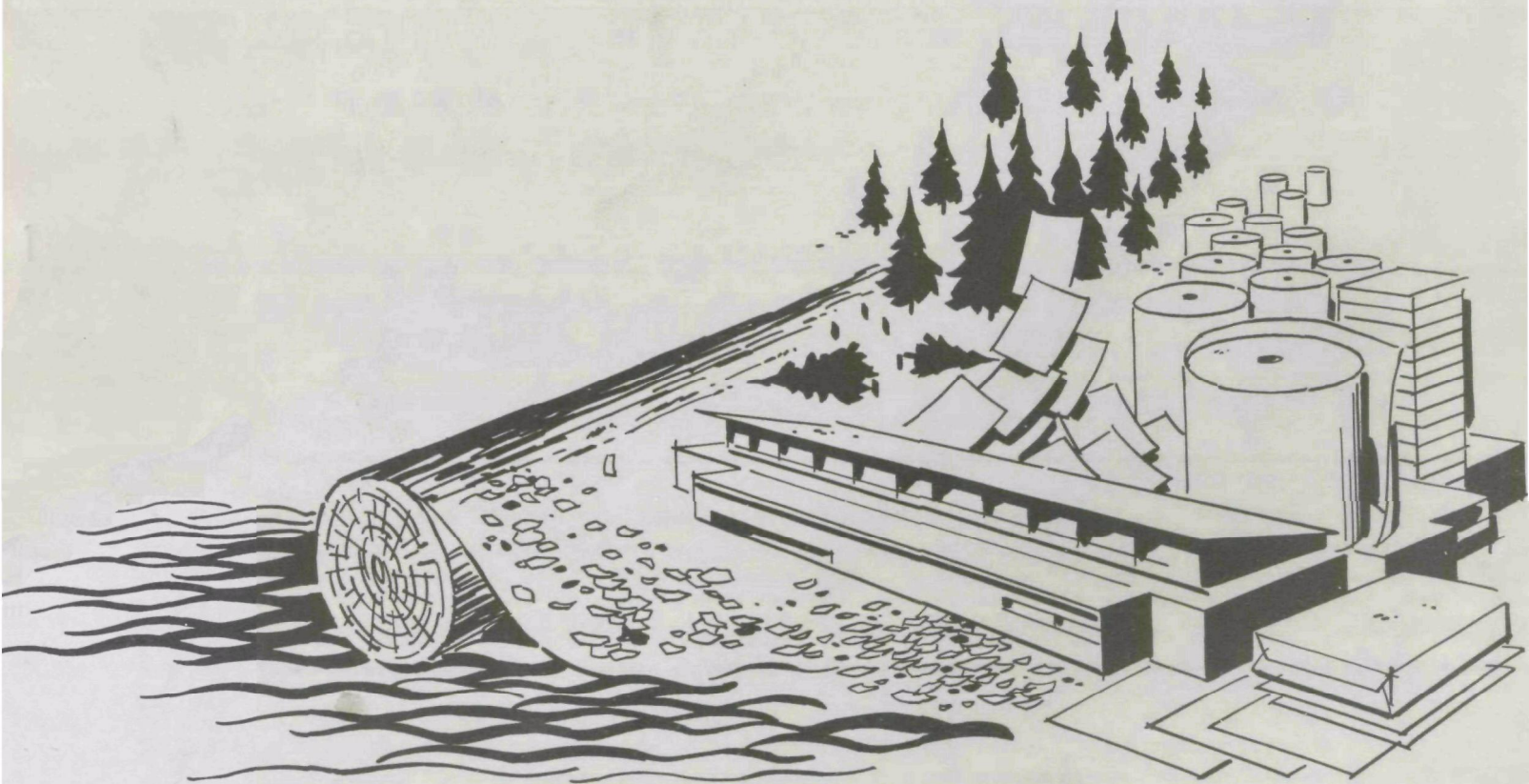




SLUDGE MATERIAL RECOVERY SYSTEM FOR MANUFACTURERS OF PIGMENTED PAPERS



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SLUDGE MATERIAL RECOVERY SYSTEM FOR
MANUFACTURERS OF PIGMENTED PAPERS

by

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for the

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EPA Review Notice

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ABSTRACT

Disposal of the sludge that results from primary treatment of waste waters generated during the manufacture of coated and/or filled papers has caused many problems.

A process has been developed and tested in a full scale mill trial wherein the pigment present in the waste sludge was reclaimed through incineration of the volatile components of the sludge. In essence, the process consisted of operations in which the sludge resulting from the primary treatment process was diluted to less than 0.75% solids, centrifuged, dewatered to 30% solids, shredded, dried, and burned in a rotary kiln. The ash that resulted - the pigment - was then pulverized and used as filler pigment in the papermaking process.

During the trial it was found that a pigment of acceptable abrasiveness and a GE brightness of 84-85% could be produced provided that sludge centrifuging was practiced and the temperature in the kiln was kept below 1600°F.

A full scale system capable of processing 40 tons per day (dry basis) primary treatment sludge would produce reusable filler grade pigment at a net cost of \$50 per ton. When compared to the delivered cost of virgin filler clay (\$38 per ton) it can readily be seen that full scale pigment recovery utilizing this system is not economically justifiable at this time.

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SECTION I

CONCLUSIONS

1. Gritty materials, such as sand, can effectively be separated from primary treatment sludge through the use of Bauer type centricleaners provided the sludge is first diluted to less than 0.75% solids.
2. Sludge at less than 0.75% solids can effectively be dewatered to 30% solids through the use of a rotary drum vacuum filter.
3. By carefully controlling both temperature and retention time, a mixed pigment of 84-85% GE brightness and acceptably low abrasiveness can be obtained through the thermal incineration of centricleaned dewatered primary sludge in a rotary kiln.
4. If the temperature in the rotary kiln is allowed to exceed 1600°F. an excessively abrasive pigment will be obtained which is not suitable for use in the paper-making process.
5. Grinding and classifying in a Bauer Pulverizer-Classififier produces a pigment of acceptable particle size for use in the papermaking process.
6. Reuse of the reclaimed pigment as a wet end filler at the paper machines is technically quite feasible.
7. Reuse of the reclaimed pigment as a coating pigment is not feasible because of the problems associated with dispersion of the material.
8. Because of the high capital cost involved in the recovery of this pigment, it is not economically feasible to use this recovery process at this plant at this time.

SECTION II

RECOMMENDATIONS

Future work in the area of filler pigment recovery through the use of incineration techniques should be discontinued unless more than 40 tons per day of sludge can be processed.

More effort should be expended to develop an economical method of either removing the calcium oxide present in the reclaimed pigment or converting it to calcium carbonate. If this can be accomplished, the pigment may then possibly be of more value as a replacement for the more expensive coating pigments.

Efforts devoted to developing methods of recycling the sludge without removal of the volatile components, should be explored more extensively. The possibility exists that centricleaning sludge may render it suitable for use in some grades of paper.

SECTION III

INTRODUCTION

Because of the lack of a better method, numerous industrial plants are disposing of their dewatered waste treatment sludges through various landfill operations. This, unfortunately, is not considered a satisfactory long term disposal procedure because of the lack of available land area, the potential air pollution problems associated with the decomposition of the organic materials present in the sludges and the potential water pollution problems which may result from the leaching out of the sludge piles by surface waters. Consequently, with the tremendous increase in construction of new treatment plants throughout the paper industry, it is imperative that a more suitable sludge disposal method be developed.

This project was geared to accomplish that goal for that part of the industry which, as a result of its production, produces waste sludges which have high inorganic pigment content and relatively low fibrous content. (In general, a high pigment content sludge is one with more than 25 to 30% pigment.) It can generally be said that most paper mills producing significant amounts of coated paper or board will have waste sludges of "high pigment content." With the assistance of the American Paper Institute it has been determined that, based on 1968 production figures, 14.5% or 3.2 million tons of the printing papers produced were coated. In the board industry 4.3% (1.0 million tons) of the total production was coated. In addition to these there are also some mills which manufacture high filler content papers which in some cases would have high ash wastes. No statistics were obtained on these because of the difficulty of obtaining grade mix data with which to estimate the pigment levels in the wastes.

In any event, it can be seen that, with more than nine percent of the total paper industry producing coated paper products, there is a widespread need for the development of a sludge disposal process which will afford an economic method of recovering the pigment present in that type of waste.

Background Information

In the early sixties the S. D. Warren Company realized that it would some day have a problem disposing of the sludge produced in its primary waste treatment plant. At that time a thorough literature review was completed which revealed that dewatered sludge could be disposed as follows: barging it out to sea since the company is near the coast, stockpiling it on land, reusing it to make products such as wallboard, bricks, ceramic building materials and many other similar products. However, the general concensus of the literature was that the best solution to this problem revolved around the removal of the organic materials present in the dewatered sludge with the recovery of the ash in a form that is reusable in the same papermaking process.

Three general methods are potentially available to accomplish this — biodegradation, chemical oxidation or reduction and pyrolysis. The biodegradation process is not yet well enough researched to be of practical interest. Oxidation-reduction processes are basically complex and tend to be prohibitively expensive. Pyrolysis appeared to be the method which would afford the simplest and least expensive alternative to ridding the sludge of the troublesome organic impurities.

In view of the above, the following course of research was pursued.

Developmental Research

Developmental research work in the field of sludge incineration began by S. D. Warren in early 1962. At that time the company did not have a primary treatment system in actual operation and it was necessary to manually filter selected waste streams in the mill to obtain the necessary sludge samples for the incineration studies. The repulping wash water, which had a flow of 0.76 mgd at a total suspended solids loading of 66,300 lbs./day, was filtered with Sweco screens.

Samples of the recovered sludge were ashed in a laboratory muffle furnace at temperatures ranging from 1290°F. to 1620°F. The results of these pyrolysis studies were relatively inconclusive as was indicated by the variability in brightness of the pigments which were obtained.

Because of the lack of personnel and equipment needed to obtain large samples of sludge from our wastewaters, the project was allowed to become dormant until the latter part of 1965. At that time the company had a 40 foot diameter Graver clarifier in operation. In general, only the broke plant waste waters were being included in that system. Analysis of the clarifier underflow revealed that the sludge had the following solids composition: 81.7% clay, 8.0% CaCO_3 , 5.0% TiO_2 , 5.5% organic matter, 0.9% water soluble salts, and 0.1% Fe_2O_3 .

It was decided to initiate pilot scale field incineration trials to determine whether or not pigment of acceptable brightness and abrasiveness could be produced.

First Rotary Kiln Trial

Traylor Engineering, Division of the Fuller Company/General American Transportation Corporation of Pennsylvania was equipped with a 12" diameter by 15' long laboratory rotary kiln which had variable temperature control, variable kiln slope and speed control. The kiln was also equipped with a pre-dryer section which had scraper knives to control feed stock size to the kiln.

Four 55-gallon barrels of 30% solids sludge were shipped to Traylor Engineering for incineration in their laboratory kiln in September 1965. The sludge was hand fed into the pre-dryer section at 43lb./hour and thus dried to 70-75% solids. The kiln temperature was varied from 1500°F. to 1800°F. during the trial. If the temperature was held too far below 1500°F. the product was greyish in appearance - presumably because of incomplete combustion of the organic materials. On the other hand, if the temperature was too high (1600-1800°F.), although the brightness was excellent, the product was exceedingly hard and abrasive. The optimum operating condition was found to be 1500-1550°F. with a 90 minute retention time in the kiln.

A 500 pound sample of the above calcined material was sent to the Bauer Brothers Company of Springfield, Ohio for pulverizing and classifying in their Hurricane Pulverizer-Classified. This unit was an air attrition mill with an integral classifier so that the unit pulverizes, classifies, and extracts unwanted impurities.

The results of the pulverizing trials revealed that pigment with an average particle diameter of 0.54 microns could be obtained relatively easily and economically. With an inlet pigment GE brightness of 84.0%, a final classified pigment GE brightness of 87.0-88.0% was produced. This, of course, was the result of removing the coarse low brightness sandy materials present in the calcined product.

Samples of the calcined, pulverized and classified product were then evaluated at the S. D. Warren Company Research Laboratory. Both abrasion tests and hand sheet filler use tests were run on the recovered product. The pigment abrasion was tested using the copper disc method and found to be within acceptable limits although it was slightly higher than filler clay abrasion values and about the same as Freeport Kaolin's N-90 calcined clay.

The handsheet tests indicated that the retention of the recovered pigment in the sheet was the same as regular pigments. The opacifying properties of the pigment were equal to N-90 clay and superior to regular filler clay. Sheet brightnesses were slightly lower than N-90 clay filled sheets and better than filler clay filled sheets.

Early in 1966 a 180 foot diameter primary clarifier with 15 foot sidewall was constructed. Paper mill wastes were collected and treated with this equipment. Normal feed flow is 10 MGD and the .5 MGD of sludge is removed at 2.5-3.0% solids. An existing Impco pulp washer was rebuilt for use as a vacuum filter to dewater the clarifier underflow sludge. Thus, sludge from the primary treatment system became readily available for succeeding trials.

Copeland Process Trials

Early in 1968 a sludge incineration trial was run at the Battelle Memorial Institute facilities in West Jefferson, Ohio. During this trial the Copeland Process fluo-solids kiln was utilized. Presumably the combination of vigorous agitation and mixing of the solids in the fluidized bed and the enormously large surface area of the bed particles would contribute to very efficient combustion within the unit.

The predominant advantage of the fluo-solids incinerator over the rotary kiln unit is that the calcined pigment can be recovered as a non-agglomerated fly ash which should be reusable without further processing.

Seventeen 55-gallon drums of 30% solids dewatered clarifier underflow were shipped to Battelle for incineration. A number of mechanical and operational problems were encountered with feeding the sludge into the incinerator. However, most of these problems were corrected and the sludge was calcined at 1300-1750°F. and samples of both the fly-ash and bed products were obtained after quasi-steady state was achieved. For the most part the samples obtained at the lower temperatures were quite greyish colored and somewhat abrasive. The samples obtained at the higher temperatures were more deeply beige colored and appreciably more abrasive.

The fly ash product obtained during this trial was generally less than 110 mesh particle size and of relatively high brightness (78.6% GE). However, the abrasiveness of this product was 60% higher than the bed product obtained at the same conditions. Presumably this was the result of carry over of sand from the bed into the fly ash product. (The trial was started with a sand bed with the thought that as the trial progressed the sandy material would be displaced by agglomerated calcined pigment. Unfortunately, this phase of the trial was never completed because of the long times required to replace the bed.)

Samples of the pigment obtained from the bed product were used in handsheet studies at the S. D. Warren Company

laboratories. After grinding and screening it was found that a significant amount of grit and silica was collected on 80, 120, 200, and 325 mesh screens. This may have been due to the original sand bed present in the system. Evidently the calcined pigment agglomerated around the sand particles. The sand was then separated from the clay during pulverizing.

Handsheets prepared with this filler material were generally of lower optical qualities than either conventional filler clay filled sheets or those filled with the pigment which was recovered during the earlier rotary kiln trial.

In view of the high abrasiveness of the pigment obtained during the above trial it was then decided to examine the effects of abrasive materials present in the treatment system before incineration on the final abrasiveness of the recovered product. Samples of sludge were obtained both with and without inclusion of the debarking operation waste (high sand content). These samples were then incinerated in a laboratory muffle furnace at 1000°F. After calcining and grinding the pigment abrasiveness of each was determined. It was found that the calcined pigment with the debarker waste included was 70% more abrasive than that without the debarker waste.

After having determined that the calcined pigment abrasiveness might well be partly due to the sandy materials present in the waste, a repeat incineration trial using the Copeland Process system was initiated. In this case, however, the sludge was obtained during a period during which no debarker waste was being added to the treatment system.

As an integral part of this trial, differential thermal analyses (DTA) were run on dried sludge and normal clay filler. The results of these analyses indicate that alterations in the physical properties of clay (e.g., abrasiveness) may occur at 1110°F. and 1960°F. It was then decided to calcine the sludge between 1280°F. and 1580°F. and vary the fly ash recycle rate and thereby effectively

control the retention time in the incineration zone of the unit.

Unfortunately, the results of this trial were virtually the same as those obtained during the first fluo-solids incineration trial reported above. The brightness was relatively low despite the increased recycling and the pigment abrasiveness was again quite high, presumably because of sand carry over into the fly ash product.

Second Rotary Kiln Trial

In order to determine whether or not the results obtained in the Traylor Engineering rotary kiln trial of 1965 were applicable to the incineration trials on the sludge obtained from the installed primary treatment system, a repeat trial was run in early 1969. In this case, sludge from the entire primary treatment system was used instead of from just the broke plant. The incineration conditions were the same as those of the previous trial. The temperature was again kept below 1600°F. with a retention time in the unit of 65 minutes. The product which was obtained was in pellet form and of very high brightness.

Samples of the pellets were again sent to Bauer Brothers for pulverizing and classifying in their pilot unit. After pulverizing, a 100 pound sample was sent to the S. D. Warren Company for further evaluations.

Pigment brightness measurements on the recovered pigment indicated that a very good product had been obtained. (GE brightness = 86.5%). Handsheet furnish studies revealed that the pigment had superior optical properties to filler clay and retention properties which were as good as normal filler clay. Unfortunately, the abrasive properties were again considerably higher than desired.

In order to resolve the abrasiveness problem, samples of isolated broke plant waste and clarifier sludge were dewatered and calcined at 1500°F. in a muffle furnace for 2 1/2 hours. The abrasiveness of each recovered sample was then measured as before. The clarifier sample was

again found to be about twice as abrasive as the broke plant sample. The most probable reason for this increase is that the clarifier sample has other sandy type wastes included in the sludge. These gritty materials of course contribute very significantly to the abrasive problem.

In order to complete the research program it was proposed that more extensive incineration trials be run on the dewatered sludge produced in the primary treatment system. Two important additional considerations would be made.

(1) Primary clarifier sludge would be degrittied prior to incineration in an effort to decrease the abrasiveness problem and (2) the quantity of recovered pigment produced should be great enough for use in a full scale papermaking trial.

Thus it was proposed that a mill scale pilot run be organized to solve remaining technical problems and determine the technical and economic feasibility of the process.

SECTION IV

EXPERIMENTAL PLAN AND EQUIPMENT

Based upon the knowledge and experience gained through research in the preceeding years, an experimental plan was developed which would lead to the production of usable pigment from primary clarifier sludge. The necessity of degritting sludge prior to incineration had been satisfactorily shown in the laboratory, although never tried on the pilot scale. Only limited amounts of recovered pigment, enough for handsheet analysis, had been obtained. It was desired to produce a quantity of pigment great enough to allow its use in a full scale paper machine trial.

Accomplishing the objective of producing usable pigment, in quantity, from primary clarifier sludge required that modifications be made to existing S. D. Warren equipment, and pilot scale equipment be contracted from outside for certain special work.

Basically the experimental plan consisted of the following steps. (1) Obtain sludge free from the dirt and grit that caused part of the previous abrasiveness problems. (2) Incinerate the degrittied sludge to remove unwanted organics. (3) Pulverize the incinerated sludge to produce a uniform, fine particle size. (4) Run a full scale paper machine trial using the recovered pigment in place of virgin filler. (5) Print the paper produced as a final test of the papers quality. Naturally evaluations were to be undertaken after each step to determine the product quality before continuing the work.

Each of these phases was indeed completed. Detailed information explaining the equipment and methodology involved in executing each step is presented forthwith.

Modifications to Existing Equipment

Before discussing the modifications incorporated for this project a brief explanation of the existing sludge collection and dewatering system is in order.

Waste waters are collected throughout the paper mill and pumped to a 180 foot diameter primary clarifier. At 10 MGD flow the detention time is about 7 hours and the overflow rate 400 gal/day/sq.ft. Influent pH varies from 6.0 to 10.0 and effluent pH from 6.5 to 7.5. Temperature varies with seasons of the year from influent values of 60° to 80°F. and effluents from 35° to 90°F. Influent BOD ranges from 200 to 300 mg/l and is reduced to 100 to 150 mg/l in the effluent for a 35 to 50% reduction. Suspended solids are reduced 75% from an average influent of 3000 mg/l to 750 mg/l. Complete removal (100%) of fiber and settleable solids is achieved.

Underflow sludge at 2.5-3.0% solids is pumped to a 9000 gallon surge chest located in the mill. Sludge is then pumped from the surge chest with a centrifugal pump, to an 8' diameter x 10' face rotary drum vacuum filter. A flocculation agent, Calgon WT 2660 (a cationic polymer) is injected just prior to the vacuum filter to aid coagulation and the dewatered sludge comes off at 25-30% solids. Sludge is conveyed to trucks for disposal as land fill. Figure one is a schematic of the existing system.

The first objective to be met for the mill scale trial was the production of dewatered sludge free from abrasive materials such as sand and gravel. Two alternatives were available to accomplish this end. (1) A grit removal system could be installed on mill wastes ahead of the clarifier to remove abrasive materials or (2) a portion of the under flow sludge at 2.5-3.0% could be diluted to 0.5-1.0% and centricleaned. After considerable analysis it was decided that a sludge dilution and centricleaner system would prove most feasible to provide the amount of degritted sludge required for this pilot project. Economics and ease of operation were the determining factors.

Modifications necessary to incorporate the degritting equipment were minimal. The existing system was changed as follows: 1. The 2.5-3.0% solids sludge from the primary clarifier was diluted with fresh water to obtain

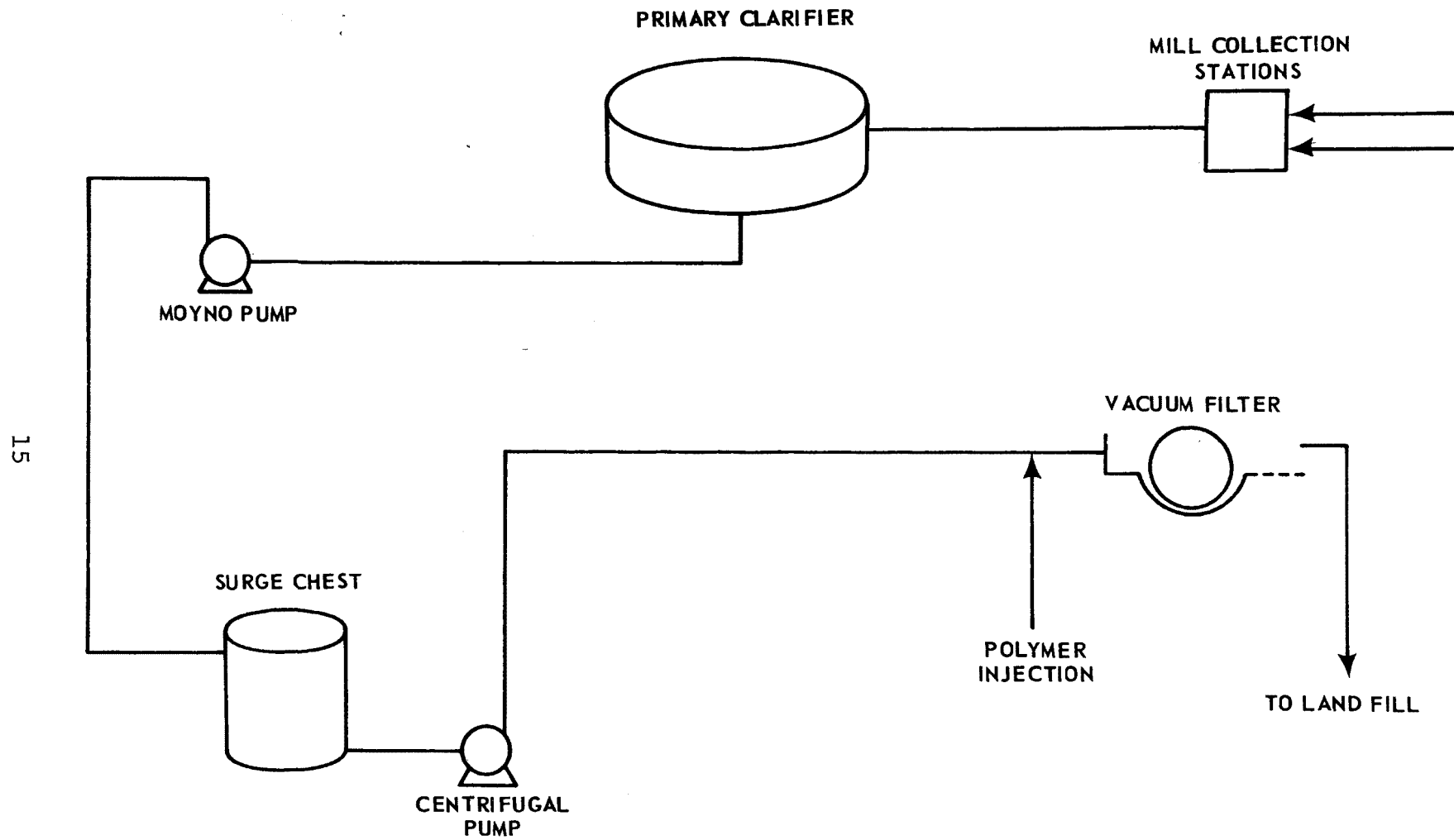


FIGURE 1
EXISTING COLLECTION SYSTEM

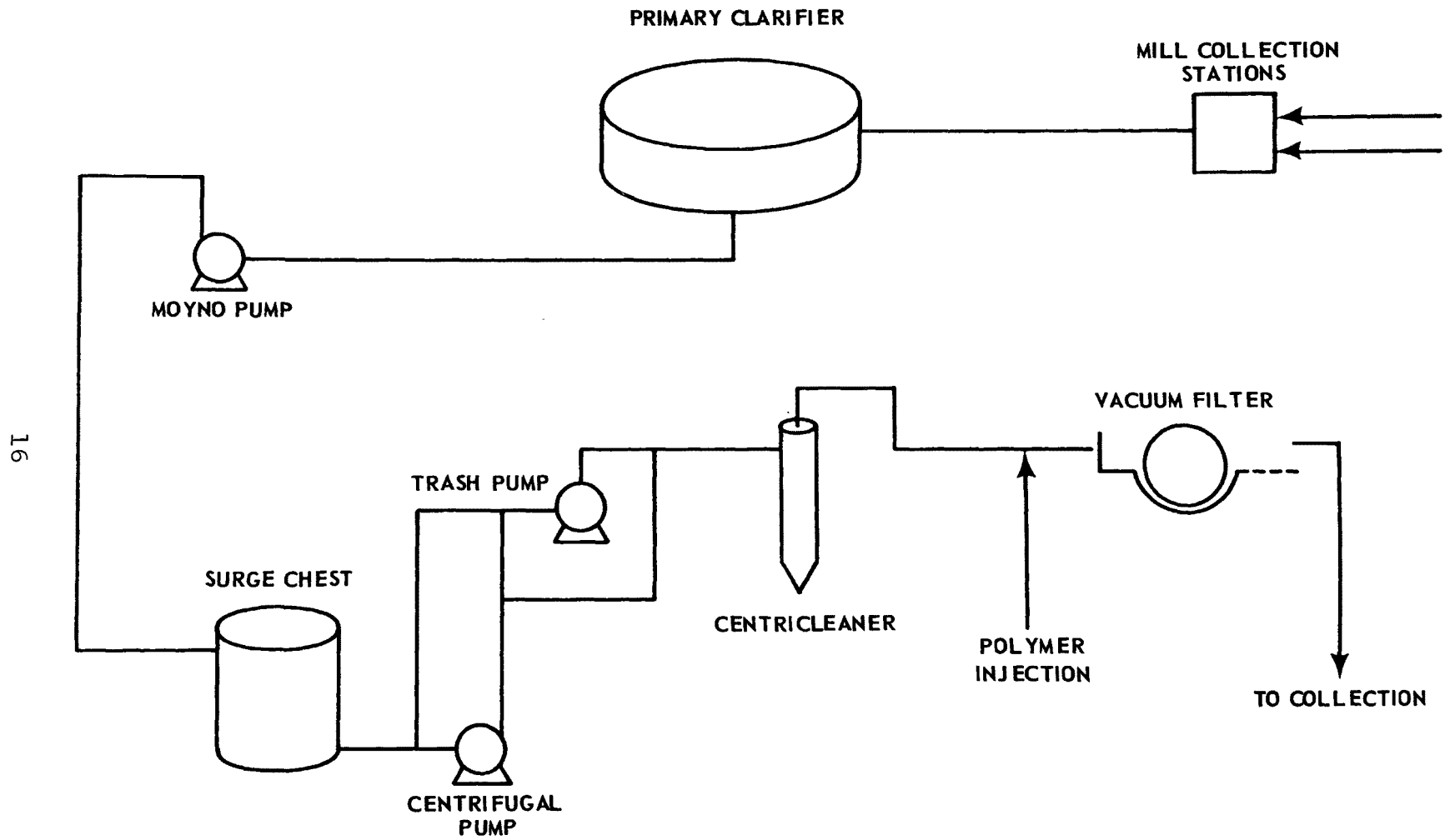


FIGURE 2
MODIFIED COLLECTION SYSTEM

0.5-1.0% solids sludge in the surge chest, the surge chest being used as a dilution tank. Consistencies in this range are necessary to enable the centricleaner to operate at maximum efficiency. It was necessary to run a fresh water line to the surge chest. 2. A 6" x 6" Gorman-Rupp sludge pump was procured and installed for series operation with the existing centrifugal pump located between the surge chest and vacuum filter. The additional pump was required to overcome the 40 psig pressure drop imposed by the Centricleaner. 3. A Bauer Brothers Model 606-110P Centricleaner was installed after the new pump and prior to the vacuum filter to remove the abrasive sand and gravel. Figure two is a schematic showing the location of the pieces of equipment that were incorporated. The interrelationship and operation of these pieces of equipment is described in the next section.

Dirt and Grit Removal

Primary clarifier sludge was diluted with fresh water from 2.5-3.0% solids to 0.4-0.7% solids in the surge chest. Because of our particular situation and the economics involved a batchwise operation was required.

Diluted sludge was pumped, using two centrifugal pumps in series, to the Bauer Centricleaner. Accepts were diverted to waste until a flow of 100 gpm at 37-40 psig was attained. Centricleaner discharge was submerged. Accepts were then valved to the vacuum filter headbox. Flocculating polymer was injected between the centricleaner and vacuum filter to aid in dewatering.

Vacuum filtration was accomplished with a vacuum of 9-10 inches of mercury at a drum speed of about 5.2 revolutions per hour. The degrittied sludge was dewatered to 25-30% solids.

The sludge blanket, or cake, was discharged via a screw conveyor into polyethylene lined 55 gallon drums. A small amount of formaldehyde was added to each drum to prevent bacterial decomposition and the liners tied off. Drums were fitted with covers for shipment.

Data was collected throughout the grit removal and collection period. Log sheets were maintained to monitor routine operating conditions such as sludge flow rate, pressure drop across centricleaner, reject flow rate, drum vacuum and surge chest level.

Samples were obtained of feed and centricleaner accepts and rejects for analysis. No operational problems were encountered during this work.

Incineration

Incineration of dewatered sludge was contracted to the Fuller Company, a Division of General American Transportation Corporation (GATX) in Catasauqua, Pennsylvania. As previously noted, the Fuller Company had some experience with S. D. Warren sludge.

The equipment used in this program was a 3' x 30' rotary kiln lined with 70% alumina brick, 4 1/2" thick. It has a variable speed range between 0.8 and 4.2 RPM and a slope control between horizontal and 3/4" per foot.

The exhaust end gases of the kiln pass through a brick fall out chamber and then to a bag collector or to atmosphere when using natural draft. Temperatures in the kiln hot zone were measured optically and by direct thermocouple probes within the load. View ports were located in the firing hood. The kiln was fired with #2 fuel oil.

Sludge was shredded in a pug mill prior to incineration. The pug mill used in this program was an 8' long unit with variable speed drive. It consisted of a single shaft chopping blade arrangement with additional blades added to the last 3' of the unit to insure satisfactory shredding. The pug mill was hand fed from the drums for this test.

The Fuller Company kept complete records and reported results to the S. D. Warren Company. No functional or mechanical, operational problems were encountered with this equipment.

Pulverization

Pulverization of degrittied, incinerated sludge was contracted to the Bauer Brothers Company a subsidiary of Combustion Engineering, Inc., located in Springfield, Ohio.

A Hurricane Pulverizer-Classified was used to grind the sludge fine enough so that 99.9% of the particles would pass through a 325 mesh screen. It was set with 24 fingers (rotary arms which flail the incoming pigment agglomerates and thereby reduce their size) in the classifier. A reject rate of 3-5% was set. Two angle iron deflectors were used on the classifier shelf.

Bauer Brothers Company kept complete records and reported results to the S. D. Warren Company. No functional or mechanical operational problems were encountered with the Pulverizer-Classified.

Paper Machine Trial

Degrittied, incinerated, pulverized primary clarifier sludge was utilized as the filler pigment in a full scale paper machine trial. S. D. Warren's Number Eight Paper Machine was utilized. It is a typical Fourdrinier with a size press and MF (machine finish) stack. This machine is capable of producing paper 57 inches wide at a rate of 340 feet per minute. It normally produces thirteen tons per day.

The paper, Publisher's English Finish Offset, was made in three basis weights, 45 pound, 50 pound, and 70 pound (per 3300 ft²). No mechanical problems were encountered with the paper machine while producing paper utilizing the recovered pigment as filler.

Printing Trials

Paper produced utilizing recovered pigment as filler was print tested on commercial presses.

Livermore and Knight Company, commercial printers located in Providence, Rhode Island, printed 1000 sheets of 50 pound (per 3300 ft²) Demonstration Grant paper. The paper

had been cut to sheet dimensions of 52" x 76" (the largest sheet size normally printed). Two presses were utilized. One side was printed on a 77" Miehle offset press in two-color (black and green) at 4000 impressions per hour. Twenty four hours later the other side was printed on a 77" Harris four-color press at a rate of 2300 impressions per hours.

The Print Testing Department of the S. D. Warren Company printed 30,000 sheets with black ink on both sides. A 26" Harris offset press was utilized.

No mechanical problems were encountered using Demonstration Grant paper on any of the three commercial printing presses.

SECTION V

RESULTS AND DISCUSSION

Evaluations were conducted during and at the completion of each of the experimental phases previously discussed.

During the sludge dilution, degritting and dewatering phase samples were obtained for analysis so that sludge quality could be determined. S. D. Warren Research Laboratory personnel performed the testing. No sand or grit was found in any of the centricleaner accepts samples under visual microscopic observation at 20 power or by photomicroscopy at 45 power. Results of quantitative analysis of Batch Number Eight and additional details may be found by referring to Appendix A "Clarifier Sludge Trial Solids Analysis." This batch was representative of the trial. Laboratory report "Abrasion Tests of Calcined Sludge" included as Appendix "B" compares results obtained from calcined sludge with the Hi Opaque clay normally used in the papermaking process. The abrasiveness of sludge dried and calcined (incinerated) in the laboratory at 1500°F. was obtained after the sludge recovery trial and before calcining by the Fuller Company. The value, .0178, was satisfactory.

Since these evaluations showed the degrittied sludge to be acceptable, 30,000 pounds were shipped to the Fuller Company for incineration. Incineration was done in the Fuller Company's pilot 3' x 30' rotary kiln. The incineration was to be accomplished at 1500°F.-1600°F. However, the Fuller Company, in an effort to improve brightness characteristics, and without consulting S. D. Warren personnel, increased the calcination temperature to 1900°F.-2100°F. The resulting product was very abrasive in nature. Upon learning that the Fuller Company had calcined at 1900°F., S. D. Warren personnel calcined at 1900°F. under controlled laboratory conditions. This was done so that a determination of the effects of temperature on the abrasive quality could again be obtained. The results are in Appendix B and

show that calcining at higher temperature causes abrasion to be 2.71 times as great as at 1500°F. The test of the Fuller calcined clay showed it to be 3.56 times as abrasive.

An additional problem was also encountered. Although the original proposal was to recover three tons of pigment, only one ton of product was obtained. This was due to the fact that the LOI (loss on ignition) was higher than expected and because dust losses were considerably higher than anticipated in the range of 30 percent.

Procedures followed during the incineration trial, details of operation, results and conclusions are presented in Appendix C. This work was done by the Research Department of the Fuller Company. Although it was realized that the calcined product was too abrasive, hope was sustained that pulverizing would improve this characteristic.

Degritted, incinerated sludge was pulverized with a Bauer Brothers Hurricane Pulverizer-Classified. This operation was successful from an operational standpoint, however the abrasiveness was improved only slightly, and not to a satisfactory level.

Appendix D, "Hurricane Milling of Calcined Sludge," defines the operating conditions and conclusions of the pulverization trial. Brightness of the product was satisfactory at 81-82. Evaluations of the experimental work completed to this point showed that the quality of the product obtained was not satisfactory for use in a paper machine trial because of high abrasiveness. This was a very regrettable circumstance, because the process otherwise looked very promising. The cause of the high abrasiveness was traced directly to incineration at too high a temperature. Discussions were held with the Fuller Company and it was determined that, with minimal modifications the incineration could indeed be done at 1500°F.

Because of our desire to continue this project to completion, and armed with information that pinpointed the cause

of the problem encountered, and a solution to that problem, it was decided to repeat the work done to date.

Therefore more sludge was degrittied, dewatered and barreled for shipment and 65,500 pounds were shipped for incineration so that a greater quantity of product could be obtained.

The incineration trial was modified in the following manner: Two passes through the 3' x 30' rotary kiln were utilized. The first pass was at low temperature (1200°F.) and high feed rate to pre-dry the sludge. The second pass was at 1500°F. and a low feed rate to accomplish the incineration.

The incineration portion of this trial was witnessed by a representative of the S. D. Warren Company who performed abrasion tests at the site during the trial. The on site abrasion testing enabled Fuller Company personnel to adjust kiln operating conditions so that a product of acceptable abrasiveness, while maintaining brightness, could be obtained. Actual results of the abrasion tests can be found in Appendix E. Details of the rotary kiln operating conditions during the second drying and incineration entitled "Paper Mill Sludge Calcination" is included as Appendix F.

Calcination was again followed by pulverizing in the Bauer Brothers Hurricane Pulverizer-Classified. A report of this work is included here as Appendix G. It should be noted that a Fisher particle size averaging 0.83 microns was obtained. This indicates a product of low abrasiveness. Evaluation of the second calcination product at S. D. Warren showed an abrasiveness of .0185 and brightness of 83-84, both of which are acceptable.

Degrittied, calcined, pulverized sludge was evaluated by the Research Laboratory to determine whether it should be used as a filler pigment or in a coating application. Appendix H "Evaluation of Calcined Sludge as a Filler Pigment" concludes that the pigment appears to have desirable properties

as a filler pigment. Dirt was found to be at a minimum and abrasiveness and brightness were acceptable. Chemical assay showed the following composition; 14.3% as CaO, 9.2% as TiO₂ and 76.5% clay. Analysis was done in accordance with TAPPI suggested method T673, Quantitative Determination of Mineral Filler and Mineral Coating of Paper, Vol. 49, No. 11, November 1966.

Difficulty was experienced utilizing the calcined sludge for use in blade coating applications. Results utilizing sludge for this purpose were only moderately encouraging. In addition the sludge was tried as a coating pigment for conversion coated grades of paper. It was concluded that coatings containing sludge pigment show lower brightness and gloss values than normal coating pigments. High pH levels in coating slurries also presented problems. The recommendation was that other uses for calcined sludge be explored.

It was decided that the pigment recovered from the incineration of degritted primary clarifier sludge would be used as a filler pigment in a full scale paper machine trial.

No serious "runnability" problems were encountered during the trial although high alkalinity caused poor retention. S. D. Warren print test results for all basis weights were rated as "FAIR", on a scale extending from POOR, FAIR-, FAIR, FAIR+, GOOD. Thus it was felt that the paper was shippable as first quality. The paper produced, Publisher's English Offset Finish, met at least minimum specifications in all tests required for that paper. "Demonstration Grant - Paper Machine Trial" a report documenting general trial procedures, paper testing criteria, results and conclusions is included here as Appendix I. Specific test results of the paper's physical and printing characteristics in comparison to a blank, of normally produced paper, are summarized.

The paper produced using recovered pigment as filler was print tested by Livermore and Knight, commercial printers

located in Providence, Rhode Island. The four-color offset print trial of 50 pound paper (3300 ft²) was a success with no problems encountered. S. D. Warren print testing department printed the 70 pound paper in a full scale test. The sheets delivered a good flat load with ink drying and binding to the surface very well. The paper performed well during the test and also in a later bindery operation. Appendix J summarizes the results of the "Print Trial - Demonstration Grant."

Thus it has been shown that S. D. Warren primary clarifier underflow sludge can be successfully degrittied, incinerated, and pulverized. The resulting pigment can be introduced back into the papermaking process and incorporated as a filler material. The paper produced will meet at least minimum specifications for certain grades of paper and perform well under commercial printing conditions.

SECTION VI

FULL SCALE RECOVERY SYSTEM

If a full scale pigment recovery system were built at this plant it would be sized to process 40 tons per day (dry basis) of primary treatment sludge which would be dewatered to 30% solids.

Based on the results of this study such a system would consist basically of a sludge degritting system, a sludge shredder, a rotary kiln, a pigment pulverizer-classifier and a pigment storage and distribution system. (See Figure 3 for a schematic of such a system.)

The basic equipment breakdown would be as follows:

- 1 - 10,000 gallon sludge dilution tank
- 1 - Sludge solids controller
- 18 - Centricleaners sized to handle 2.5 MGD @ 0.5% solids
- 2 - Vacuum filters
- 1 - Sludge shredder
- 1 - Rotary kiln 6' 6" x 160' long
- 1 - 50,000 gallon oil storage tank and distribution system
- 1 - Pulverizer-Classified sized to handle one ton per hour feed
- 1 - 80 ton per day bulk storage system
- 1 - Slurry tank

All necessary conveyors, instrumentation, buildings, etc.

The system described has been estimated to cost approximately \$1,500,000 with an annual operating cost in the neighborhood of \$175,000 per year. (See cost estimate included in Appendix K for details.)

On a production basis of 24 hours per day, 360 days per year and at a rate of 7,500 tons per year of recoverable pigment the estimated cost of the recovered pigment would be \$71 per ton. However, part of this cost would be defrayed because of the savings associated with elimination

FULL SCALE PIGMENT RECOVERY SYSTEM

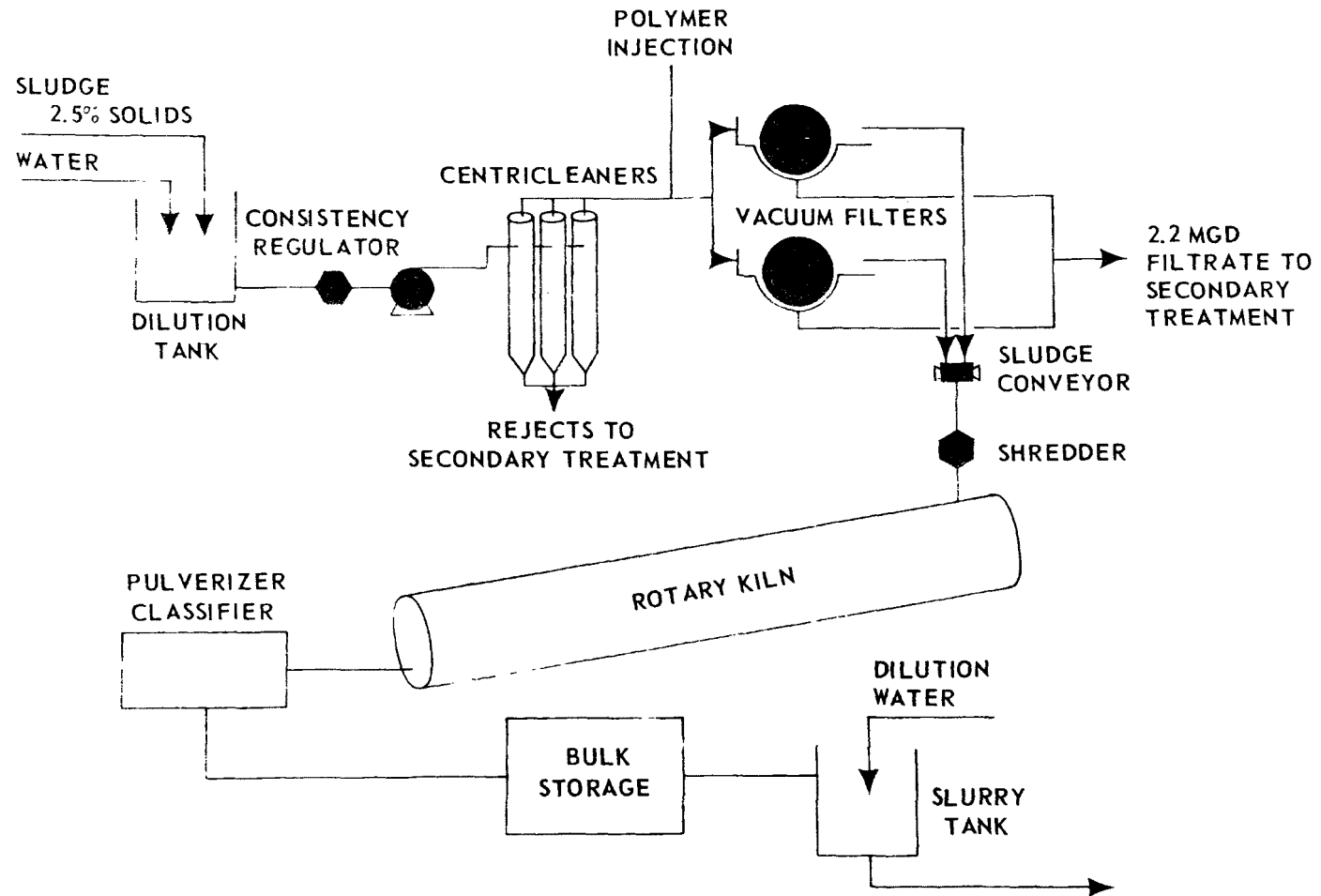


FIGURE 3

of the present method of sludge disposal. This would amount to approximately \$21 per ton saving or effectively a \$50 per ton cost for the recovered pigment.

Since the delivered cost of virgin filler pigment is \$38 per ton, it is readily apparent that recovery of the same quality pigment at \$50 per ton is not economical.

However, the concept of full scale pigment recovery should not be discarded because of the potential value of this pigment as a replacement for some of the more expensive coating pigments. Admittedly, the results of the laboratory evaluations on the pigment reclaimed during this trial were not very encouraging. Nevertheless, it should be pointed out that it may be possible to either re-precipitate the troublesome calcium oxide present in the pigment or to remove the calcium oxide by washing with clean water. In either case the product that would result may well be of use as a coating pigment of higher value and a full scale pigment recovery system of this type would then become more economical.

SECTION VII

ACKNOWLEDGEMENTS

The Project Director, Dr. Richard P. Labrecque, wishes to acknowledge the assistance received from the following S. D. Warren Company personnel:

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SECTION VIII

GLOSSARY

Abrasive - degree to which a substance would cause wearing by friction.

Accepts - product reclaimed, as opposed to rejects.

Broke - repulped waste process paper.

Calcine - to heat to a high temperature but without fusing in order to effect useful changes.

Centricleaner - device which utilizes centrifugal force to separate undesirable particles such as sand.

Coating Pigment - a pigment suitable for addition principally to the surface of paper.

Degrit - remove unwanted abrasive components.

Dewater - to remove excess water leaving solids.

Filler Pigment - a pigment added throughout the internal paper structure.

Flocculating Polymer - chemicals which will cause particles to agglomerate.

GE Brightness - (General Electric brightness) measure of reflective properties.

Incinerate - burn to ashes, i.e. removal of organics.

Loss on Ignition - (LOI) weight of a substance lost during incineration.

Pulverize - to reduce to very small particles.

Rotary Kiln - heated enclosure, revolving on its axis, used for processing a substance by burning.

Runnability - capability of the paper machine to continuously run paper of acceptable quality.

Solids Content - matter remaining after removal of the liquid phase of a solution.

SECTION IX
APPENDICES

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APPENDIX A

CLARIFIER SLUDGE TRIAL SOLIDS ANALYSIS

A request was submitted to analyze clarifier sludge solids and coordinate other research activity as abrasion testing and photo documentation. "Feed" samples directly from the clarifier and "accepts" versus "rejects" samples from trials using a Bauer Centricleaner were submitted to determine differences in solids. Differences were determined by screening, filtration, microscopic examination and chemical examination. Identification of particles with particular emphasis on grit or sand particles was carried out.

Summary

No hard grit and/or sand was detected in any of the submitted samples.

The Bauer Centricleaner appeared to separate on the basis of particle size rejecting the larger particles of a given trial as opposed to separating on the basis of mass.

Retained, screened material was primarily fiber and coating chips. Black and brown specks, etc. were a minimal fraction.

More black and brown specks or grains were noted in the "rejects" samples than in the "feed" or "accepts" samples.

Upon examination, the black and brown specks or grains proved to be oil, grease, tar, coal, bark, melamine-like resin, leafs, tobacco, and other particles that would powder or break easily as compared to sand.

Coating chips were present in three forms: hard platelets of varied sizes, in a semi-gelled state and in a complete gelled state. Treating these materials with HCl caused disintegration and CO_2 evolution.

One sludge trial was measured quantitatively for sieve sizing, total and suspended solids, ash, TiO_2 and CaCO_3 .

Photomicrographs were taken of the screened samples to document the visual data.

A composite sampling of all the trials showed a similar distribution of fiber, coating chips, black and brown particles and gelled coating as the above trials.

Discussion

The first trial of September 23, 1970 consisted of samples from the Bauer Centricleaner using various discharges to determine best operation method for this device. 80, 120, 200, and 325 mesh screens were used in a stack to separate 100 mls. of starting sample. In turn, these separates were examined using 15X microscopy:

Submerged discharge with 4 psi backpressure

Feed sample - Mainly fiber mass with few coating chips. 325 screen was plugged with small fiber ends, vessels and coating gel. Very few black or brown specks noted.

Accepts sample - A large presence of fiber with few coating chips on 325 screen. Very few other specks noted.

Rejects sample - An abundance of fiber with coating chips. Also presence of black and brown specks of leaves and tar-like substance.

Submerged discharge

Feed sample - A fiber mass with coating chips suspended within. Some large black particle-like grease. 325 screen plugged with fiber ends, vessels and coating gel.

Accepts sample - A fair amount of fiber with some brown leaf-like material. 325 screen had slight fiber and coating presence.

Rejects sample - Large quantity of fiber with coating chips being suspended within. Also black and brown specks seen throughout suspension.

Free fall discharge

Feed sample

- Some fiber mass with few coating chips and splinters. 325 screen had little fiber and coating gel.

Accepts sample

- A fiber mass with a few brown leaf-like specks. 325 screen had fiber ends and vessels in small quantity.

Rejects sample

- Abundant fiber and coating chip presence with some very large black grains like coal or rust.

None of the above samples showed presence of sand or grit. This trial shows the influent as a constantly changing media from the "feed sample" information listed above.

The next trial of September 25, 1970 was B-8 (batch 8) samples taken at 4:00 a.m. These samples were treated as the previous trial but using 200 mls. of sample. A quantitative measure of the solids retained on screens, dried vs. suspended solids and ash with TiO_2 and CaCO_3 were performed:

B-8 Feed sample

- Small quantity of fiber with coating chips. Few specks but some large brown resin chunks and black chunks of grease or oil. 325 screen had short fiber and coating gel with few specks or coating chips.

B-8 Accepts sample

- Fair quantity of fiber with few specks. 325 screen had short fiber ends or vessels with few specks.

B-8 Rejects sample

- Large quantity of coating chips suspended in fiber mass. Also large brown resin chunks and bark presence. 325 screen had small coating chips in fiber ends with appearance of some black and brown specks scattered throughout.

When picked apart, the black specks noted in this trial appeared as coagulated grease or oil or possibly tar. Pieces of coal were apparently present also. Photomicrographs were made from the screenings.

B-8 Quantitative Compilation

	<u>Feed</u>	<u>Accepts</u>	<u>Rejects</u>
Suspended Solids gms./100 mls.	0.5304	0.4689	1.2807
Retained Screened Solids gms./100 mls.			
80 mesh	0.1798	0.1706	1.1881
120 mesh	0.0053	0.0026	0.0103
200 mesh	0.0127	0.0029	0.0505
+325 mesh	<u>0.1004</u>	<u>0.0319</u>	<u>0.1884</u>
Total retained solids	0.2982	0.2080	1.4373
Total Dried Solids gms./100 mls.	0.5950	0.5455	1.9680
Screened Solids as % of Total Dried Solids	50.10%	38.11%	73.03%
Screened Solubles as % of Total Dried Solids	49.89%	61.89%	26.97%
Ash as % from Total Dried Solids	50.09%	54.12%	50.81%
TiO ₂ as % from Total Dried Solids	5.49%	5.95%	2.64%
CaCO ₃ as % from Total Dried Solids	9.81%	-	11.55%

The above screened samples were retained on filter paper for photomicroscopy at X45. Since the samples were relatively large for the 80 and 325 mesh screen sample, a reflected light source was used here. The 120 and 200 mesh screen samples were small enough to allow use of transmitted light. Samples of mill sand, a filter paper blank and a calibration measure were photographed also.

Upon completion of the sludge trial batch samples were made into a composite of feed, accepts and rejects. The composite samples were made from the settled solids thus eliminating extra water. Therefore, only a microscopic examination was performed using 100 mls. of this sample solution for screening through 80, 200, and 325 mesh sieves:

- Composite Feed Sample - Primarily fiber with coating chips suspended throughout. Some black and few brown specks being present. 325 screen had small coating chips with fiber ends or vessels and few specks being present.
- Composite Accepts Sample - Mainly fiber with some coating chips and few specks noted. 325 screen had coating chips and gel in some fiber and few specks.
- Composite Rejects Sample - Primarily fiber with coating chips with some black and brown specks. 325 screen had coating chips with black and brown specks.

In this sample series, there were small brown lumps that would powder like cocoa lumps when pressed. Photomicrographs were made of the above screened separates.

APPENDIX B
ABRASION TESTS OF CALCINED SLUDGE

Abrasion tests were run on samples of sludge collected from the barrels which were shipped to Traylor Company for calcining.

Abrasiveness was determined by the copper disc method which is essentially the New Jersey Zinc Company procedure as derived in Tappi 43 No. 7 230A-232A (July, 1960).

Results

<u>Pigment</u>	<u>Loss In Weight of Copper Disc.</u>
Sludge dried and calcined in Lab at 1500°F.	.0178 grams
Sludge calcined at Traylor 1900°F.	.0634 grams
Sludge calcined at Lab at 1900°F.	.0482 grams
Hi Opaque clay	.0118 grams

Calcining temperature is again found to be critical in controlling the abrasiveness of the sludge.

APPENDIX C
PAPER MILL SLUDGE CALCINATION

Object

The test described in this report was made for the following purposes:

To determine the proper burning conditions and produce a pigment having maximum brightness and minimum abrasiveness.

To produce enough material for the S. D. Warren Company to evaluate the quality of the product.

To obtain design data.

Introduction

A test program was run on four consecutive days: October 5, 6, 7, and 8, 1970.

Some previous work was performed by the Fuller Research Department for S. D. Warren Company in April of 1969 and August of 1965 on similar waste sludges to determine the feasibility of producing a suitable clay pigment. This program is a scale up similar to the 1' x 12' laboratory kiln test made in April of 1969.

Operating Conditions

A pug mill was used in this test program to prepare the wet feed prior to its entering the kiln. The wet "as received" feed material fed to the pug mill was in the form of rolls ranging in size from 6" to 12" long with a diameter of approximately 3". Some of the rolls were up to 18" long. The product from the pug mill ranged in size from 1/4" to 1". The particular high speed shredding machine that had been considered initially to prepare the kiln feed was found to be inadequate and tended to pack and pulp the material.

The kiln slope was kept at 3/8" per foot throughout the test, and the speeds and retention times used are listed below:

<u>Kiln R.P.M.</u>	<u>Retention Time</u>	(Based on the U.S. Bureau of Mines standard formula)
1.00	73 Min.	
1.70	49.5 Min.	
1.75	44.5 Min.	
1.81	43.2 Min.	

The maximum feed rate attained in the test program was 503 lbs/hr. The indicated fuel consumption is 3.26 million BTU per ton of feed.

Material Tested

On October 1, 1970, 55 gallon drums (32,200 lbs. gross) of sludge was received. The drums varied in moisture from 67.0% to 74.0%.

The feed material was in the form of wet rolls of clay impregnated paper. The rolls ranged in size from 6" to 1' long and were approximately 3" in diameter; a few of the rolls were up to 18" long and some were flattened out by compaction in the drum.

Product Evaluation

No standard equipment was available to detect brightness or abrasiveness. The products were visually observed for whiteness and hardness and was judged by rubbing the material between one's fingers. Bulk densities were taken on a sample from each drum.

The physical appearance of the product was white to a yellow tint in color and was relatively soft to the touch insofar as it would crumble easily when handled. The individual pieces ranged in size from 3/4 inch to fines.

The quantity of material sent to S. D. Warren Company for evaluation was found to be unsatisfactory.

Material Balance

<u>Material In</u>	<u>Pounds</u>
Wet feed to kiln	29,240
Product	3,500
<u>Material Out</u>	
Kiln product	2,121
Kiln discharge after shut down	164
Kiln drop out material (222# at 12% H ₂ O and 50% L.O.I.)	97.8
Material recovered from dust collector and duct work	931
Stack losses when not using dust collector	185
	<hr/> 3,498.8

3500 pounds
-3498.8 pounds
1.2 pounds unaccountable

Details of Operation

The "as received" material was hand fed to the pug mill. The feed material, which was matted rolls of wet clay impregnated paper approximately 6" to 1' long by 3" in diameter, was chopped and shredded down to pieces ranging in size from 1" to 1/4" pieces. The pug mill discharged directly into a 5" I.D. feed pipe at the back end of the kiln. A feed sample was taken from each drum for moisture and L.O.I. analysis. The kiln discharge was checked periodically for whiteness and texture. Each product drum was sampled for bulk densities and visual inspection.

10-5-70

The test was started using the natural draft system through the exhaust stack. The run was started with an

approximate feed rate of 460 lb/hr and a kiln speed of .9 to 1.0 RPM. The hot zone temperature was gradually increased to 2000°F. and held at this range. After adjusting temperature and positioning the burner flame over the kiln load, a product which contained 3/4" modules down to fines was produced. The product was soft and white in color with no dark cores in the center. The feed discharge from the pug mill ranged in size from 1" pieces to 1/4" pieces and was fairly steady in rate of discharge. The 5" dia. feed pipe plugged occasionally; however, this was eliminated by having an air purge line with a light flow of air going into the feed pipe. The material ignited and flamed in the first 12' of the kiln from the feed end.

10-6-70

At 0800 hrs the kiln speed was increased in order to reduce the heavy dropout from the back of the kiln. The kiln speed was gradually increased to 1.70 RPM and the dropout was all but eliminated. The product was not affected by the speed change and remained soft and white. At 1600 hrs the kiln speed was increased to 1.81 RPM and the feed rate was also increased. These conditions were held until midnight, at which time it was noticed that the product color had a yellow tint.

10-7-70

The hot zone temperature was adjusted slightly with no apparent effect on the yellow tint of the product. It was also noticed that the free flame zone was slowly moving forward toward the hot zone. At 0800 hrs the feed rate was cut back slightly and the kiln speed was reduced to 1.75 RPM. The product color was slowly improving, however, the free flame zone continued to move forward. With the pressure drop across the exhaust system gradually increasing, it was decided to exhaust through the baghouse dust collector. By 1230 hrs the system was switched over. The kiln speed was reduced to 1.70 RPM, the free flame zone was moving back and the product was losing the yellow tint. This was continued until midnight. The product was white and soft throughout this period.

10-8-70

The dust collector was not functioning properly, and at midnight the system was again switched over to natural draft. The dropout material from the previous days running was returned as feed. The product from this material had a yellow tint to it also. At 0530 hrs the feed was exhausted and the test terminated.

Heat Balance

Theoretical

Heat required to evaporated water:

$$\begin{array}{lcl} (70\%) (503\text{Lb/hr.}) (1122 \text{ BTU/Lb.}) & = & \\ 394,944 \text{ BTU/hr.} & = & 1.535 \text{ M. BTU/} \\ & & \text{ton Feed} \end{array}$$

Heat required to raise solids to temperature:

$$\begin{array}{lcl} (151\text{Lb solids/hr.}) (0.25) (1950.70) & = & \\ 71,440 \text{ BTU/hr.} & = & 0.283 \text{ M. BTU/} \\ & & \text{ton Feed} \end{array}$$

Required heat assuming no heat recovery
from the internal carbon

$$\begin{array}{lcl} & = & 1.818 \text{ M. BTU/} \\ & & \text{ton Feed} \end{array}$$

Efficiency of the kiln is 40%

$$\begin{array}{lcl} \text{Input required} & = & \frac{1.818}{40} = 4.52 \text{ M. BTU/} \\ & & \text{ton Feed} \end{array}$$

Heat from the internal carbon:

$$\begin{array}{lcl} (151\text{Lb dry solids}) (15\%C) & = & 22.7 \text{ LbC/hr.} \\ (22.7\text{LbC/hr.}) (14000 \text{ BTU/Lb.}) & = & 320,000 \text{ BTU/hr.} \end{array} \quad \begin{array}{l} - 1.26 \text{ M.} \\ \text{BTU/ton} \\ \text{Feed} \end{array}$$

$$\begin{array}{l} 4.52 \text{ M. BTU/ton Feed} - 1.26 \text{ M. BTU/ton Feed} = \\ \text{Required Heat} = 3.26 \text{ M. BTU/ton Feed} \end{array}$$

Experimental

Feed = 503 Lb/hr.

Average fuel per hour = 11.8 gal. = 1.54 M. BTU/hr.

Fuel consumption per ton of feed =

$$\frac{(1.54) (2000)}{(503)} = 5.98 \text{ M. BTU/ton Feed}$$

Scale up factor for commercial kilns = 1.95

$$\frac{5.98 \text{ M. BTU/hr.}}{1.95 \text{ Factor}} = 3.07 \text{ M. BTU/ton Feed}$$

Equipment

Equipment in this test program consisted of the following:

Kiln

The 3' x 30' Kiln is lined with 70% alumina brick 4 1/2 inches thick. It has a variable speed range between 0.8 and 4.2 RPM and a slope control between horizontal and 3/4" per ft. The exhaust end of the kiln passes through a brick fallout chamber, and then can either go to a bag collector or to atmosphere through a natural draft system. Temperatures in the kiln hot zone are measured optically and temperatures at the feed end of the kiln are measured with a thermocouple. Viewports are located in the firing hood. The kiln is oil fired (#2 fuel oil). The kiln was fed with a pug mill.

Pug Mill

The pug mill is an 8' long unit with variable speed drive. It consists of a single shaft chopping blade arrangement. Additional blades were added to the last 3' of the unit for this test to insure satisfactory shredding of the material.

TABLE I
 PROPERTIES OF DOMESTIC #2 FUEL OIL

API Gravity No.	33.3
Heating Value:	139,000 Btu's/gallon gross
	130,400 Btu's/gallon net
Flash Point	154 ^o F.
Distillation:	10% at 425 ^o F.
	50% at 505 ^o F.
	90% at 580 ^o F.
End Point	630 ^o F.
Ash	0.001%

CHEMICAL ANALYSES

Carbon	87%
Hydrogen	12.5%
Sulfur	0.5%
Oxygen	0.1%
Nitrogen	0.1%

TABLE II
FEED CHARACTERISTICS

<u>Feed Drum No.</u>	<u>Net Wt.</u>	<u>Cum. Wt.</u>	<u>H₂O %</u>	<u>Dry Wt.</u>	<u>LOI %</u>	<u>Potential Product Wt.</u>	<u>Cum. Wt.</u>
1	415	415	71.0	120.4	62.01	45.7	
2	364	779	74.20	93.9	62.03	35.6	81.3
3	459	1238	68.98	142.4	59.37	57.9	139.2
4	439	1677	69.43	134.2	58.93	55.1	194.3
5	433	2110	68.34	137.1	57.16	58.7	253.0
6	435	2545	69.13	134.3	56.55	58.4	311.4
7	436	2981	68.69	136.5	52.50	64.8	376.2
8	430	3411	70.95	124.9	61.24	48.4	424.6
9	432	3843	68.54	135.9	57.10	58.3	482.9
10	447	4290	69.75	135.2	64.16	48.5	531.4
11	430	4720	68.72	134.5	56.73	58.2	589.6
12	430	5150	69.59	130.8	61.56	50.2	639.8
13	460	5610	68.66	144.2	61.98	54.8	694.6
14	477	6087	69.40	145.9	65.37	50.5	745.1
15	477	6554	72.90	129.3	63.05	47.8	792.9
16	433	6987	70.07	129.6	65.01	45.3	838.2
17	396	7383	68.90	123.2	62.54	46.1	884.3
18	442	7825	69.08	136.7	57.57	58.0	942.3
19	416	8241	71.12	120.1	63.52	43.8	986.1
20	450	8691	70.17	135.1	61.59	51.9	1038.0
21	469	9160	71.26	134.8	58.74	55.6	1093.6
22	461	9521	69.87	138.9	58.97	57.0	1150.6
23	462	9983	72.01	125.1	62.64	46.7	1197.3
24	451	10434	69.37	138.1	56.72	59.8	1257.1
25	415	10849	69.03	128.5	62.37	48.4	1305.5
26	415	11264	72.24	115.2	68.47	36.3	1341.8
27	456	11710	71.16	131.5	63.25	48.3	1390.1
28	441	12151	69.38	135.0	57.93	56.8	1446.9
29	434	12585	69.78	131.2	58.16	54.9	1501.8
30	440	13025	71.89	123.7	63.21	45.5	1547.3
31	460	13485	70.23	136.9	60.96	53.4	1600.7
32	450	13935	69.73	136.2	64.64	48.2	1648.9

FEED CHARACTERISTICS
(continued)

Feed Drum No.	Net Wt.	Cum. Wt.	H ₂ O %	Dry Wt.	LOI %	Potential Product Wt.	Cum. Wt.
33	406	14341	69.96	121.9	65.24	42.4	1691.3
34	435	14775	69.75	131.6	58.03	55.2	1746.5
35	434	15209	69.96	130.4	63.59	47.5	1794.0
36	439	15648	68.91	136.5	61.08	53.1	1847.1
37	409	16057	71.38	117.1	62.37	44.1	1891.2
38	380	16437	70.02	113.9	62.97	42.2	1933.4
39	473	16910	67.95	151.6	62.86	56.3	1989.7
40	433	17343	68.90	134.6	62.29	50.8	2040.5
41	347	17690	69.15	107.0	57.29	45.7	2086.2
42	455	18145	71.47	129.8	67.23	42.5	2128.7
43	481	18626	72.03	134.5	64.26	48.1	2176.8
44	434	19060	71.05	125.6	58.13	52.6	2229.4
45	391	19451	70.29	116.2	64.87	40.8	2270.2
46	382	19833	67.68	123.5	58.77	50.9	2321.1
47	426	20259	69.02	131.9	61.70	50.5	2371.6
48	343	20602	68.96	106.5	59.79	42.8	2414.4
49	366	20968	68.12	116.7	57.61	49.5	2463.9
50	393	21361	70.25	116.9	58.66	48.3	2512.2
51	334	21694	72.05	93.4	60.07	37.3	2549.4
52	374	22068	67.42	121.8	56.05	53.5	2602.9
53	426	22484	67.85	136.9	56.11	60.1	2663.0
54	443	22927	68.30	140.4	56.50	62.6	2725.6
55	411	23338	68.16	130.9	58.62	54.2	2779.8
56	436	23774	71.54	124.1	61.42	47.9	2827.7
57	376	24150	69.55	114.5	55.25	51.2	2878.9
58	438	24588	69.68	132.8	61.87	50.6	2929.5
59	373	24961	73.14	100.2	60.56	39.5	2969.0
60	391	25352	69.12	120.7	57.63	51.1	3020.1
61	401	25753	70.75	117.3	60.34	46.5	3066.6
62	426	26179	71.94	119.5	55.96	52.6	3119.2
63	463	26642	69.65	140.5	57.67	59.5	3178.7
64	391	27033	69.55	119.1	59.35	48.4	3227.1

FEED CHARACTERISTICS
(continued)

Feed Drum No.	Net Wt.	Cum. Wt.	H ₂ O %	Dry Wt.	LOI %	Potential Product Wt.	Cum. Wt.
65	384	27417	70.38	113.7	55.15	51.0	3278.1
66	399	27816	68.61	125.2	54.45	57.0	3335.1
67	481	28297	67.47	156.4	64.25	55.9	3391.0
68	431	28728	68.85	134.3	62.89	49.8	3440.8
69	411	29139	71.81	115.9	58.01	48.7	3489.5
70	401	29540	70.86	116.9	58.79	48.2	3537.7

TABLE III
PRODUCT BULK DENSITY

<u>Product</u> <u>Drum No.</u>	<u>Date</u>	<u>Time</u>	<u>Bulk Density</u>
2	10-5-70	1704-2047 Hr.	15.7 lb./CF.
3	"	2047-0105 "	15.1 "
4	10-6-71	0105-0454 "	15.6 "
5	"	0454-0835 "	16.0 "
6	"	0835-1135 "	15.7 "
7	"	1135-1515 "	16.5 "
8	"	1515-1815 "	17.2 "
9	"	1815-2040 "	17.7 "
10	"	2040-2315 "	15.9 "
11	10-7-70	2315-0150 "	18.3 "
12	"	0150-0410 "	18.3 "
13	"	0410-0635 "	17.1 "
14	"	0635-0905 "	16.9 "
15	"	0905-1150 "	18.4 "
16	"	1150-1435 "	16.8 "
17	"	1435-1740 "	16.8 "
18	"	1740-2015 "	17.8 "
19	"	2015-2320 "	13.6 "
20 (1st Pt)	10-8-70	2320-0200 "	17.3 "
20 (2nd Pt)	"	2320-0200 "	26.8 "
21	"	0200-0400 "	21.3 "

APPENDIX D

HURRICANE MILLING OF CALCINED SLUDGE

Material

01 - Calcined sludge - 15 to 24 pounds per cubic foot and a brightness of 76.4.

Objective

To demonstrate the ability of the Hurricane Pulverizer-Classifer to grind sludge to 99.9% minus a 325 mesh screen, and to upgrade by impurity extraction.

Summary &

Conclusions

This calcined sludge was harder to grind than the previous materials tested.

While no trouble was encountered in grinding to 99.9%, minus 325 mesh the overall fineness of the material was not the same. On previous test work an average Fisher Sub Sieve of .55 to .92 Microns was obtained. The lowest obtainable on this test was 1.5 Microns.

The material was highly contaminated with iron slag, scale and bolts.

The material had to be run through a magnetic grate to remove the metal.

If the material had been in better condition a better overall grinding job would have been done on the pulverizer.

Procedure

The Bauer Model No. 724 Hurricane was used for grinding and extracting impurities. The machine set-up is shown on Table No. I and the Laboratory data is shown on Table No. II.

Water wash screen analyses were taken at 10 pounds of pressure.

The processed material was sent to S. D. Warren for their evaluation.

TABLE I

October 20, 1970

Run No.	No. 1 ROTOR	No. 2 ROTOR	No. 3 ROTOR	No. 4 ROTOR	No. 5 ROTOR	INNER DISC. DIAM. (INCHES) AND No.	No. FINGERS IN CLASS- IFIER
W-1	21"	21"	Blank	21"	21"	5/21	24
W-2	"	"	"	"	"	"	"
W-3	"	"	"	"	"	"	"
W-4	"	"	"	"	"	"	"
W-5	"	"	"	"	"	"	"
W-6	"	"	"	"	"	"	30
- - - - -							
Run No.	FRONT DAMPER OPENING INCHES	BACK DAMPER OPENING INCHES	SKIM- MER OPENING INCHES	FEED SCREW PITCH AND RATIO	UPPER FAN DIAM. INCHES	BAFFLE I.D. INCHES	
W-1	1	1	4 x 2	5 x 4 12:60	28	18	
W-2	1	1	"	"	"	"	
W-3	1/2	1/2	"	"	"	"	
W-4	"	"	"	"	"	"	
W-5	"	"	"	"	"	"	
W-6	"	"	"	"	"	"	

NOTES: Two Angle Iron Deflector Added to Classifier Shelf.

TABLE II

October 20, 1970

RUN NO.	MACH. MAT'L. NO.	R.P.M.	LBS. FED	TIME (MINS.)	LBS/ HR.	H.P. USED	STACK MANO- METER. IN. OF H ₂ O
W-1	01	724	3080	95	2.7	2100	75 -1 1/2
W-2	"	"	"	103	3.0	2060	" -1 1/2
W-3	"	"	"	95	2.7	2100	" -1
W-4	"	"	"	100	2.4	2400	" -1
W-5	"	"	"	740	17.1	2600	" -1 1/2
W-6	"	"	"	881	31.0	1710	66 -2

RUN NO.	POUNDS EXTRACTED	SPECIFIC GRAVITY	FISHER PARTICAL SIZE	% - 325 MESH	BRIGHTNESS
W-1	<u>7.7%</u> 7.3	2.58	2.1	100.0	
W-2	<u>9.3%</u> 9.6	"	1.52	99.97	82.5
W-3	<u>6.1%</u> 5.8	"	1.85	99.99	81.5
W-4	<u>11.0%</u> 11.0	"	1.70	99.99	81.0
W-5	<u>22.0%</u> 147	"	1.57	99.99	83.3
W-6	<u>13.6%</u> 120	"	1.75	99.99	82.3

APPENDIX E

RESULTS OF ABRASION TESTS
DONE AT THE FULLER COMPANY

The fuller Company calcined degrittred, S. D. Warren Company sludge on January 6 through 10, 1971. Abrasiveness tests were performed during the first three days of the trial to define the kiln conditions under which a product with acceptable abrasiveness (and hopefully brightness) characteristics could be obtained. Abrasiveness was determined by the copper disc method which is essentially the New Jersey Zinc Company procedure as derived in TAPPI 43 No. 7 230A-232A (July, 1960). Samples of pigments normally used at the S. D. Warren Company were taken and the abrasiveness determined on site so that a direct comparison, under identical conditions, could be made with the calcined sludge.

Following is a summary of the results of the abrasion tests. Samples preceded by a capital S are Fuller incinerated sludge samples. Note that abrasiveness improved as the trial progressed. High Opaque, Filler Clay and Albacar are the S. D. Warren samples. The abrasiveness values on a sample of sludge calcined and determined in the S. D. Warren laboratory at 1500^oF. averaged .0182.

Summary of Abrasion Values

Date	Time	Sample	Abrasion	Remarks
1/6	1530	High Opaque	.0104	SDW Lab = .0118
	1620	Filler Clay	.0030	SDW Lab = .0025
	1700	S 1400	.0365	
	1780	High Opaque	.0099	
1/7	0815	Albacar	.0036	
	0930	S 0800	.0435	
	1015	S 0700	.0292	
	1100	High Opaque	.0037	Wrinkle in Felt
	1125	S 1000	.0328	
	1145	Filler Clay	.0041	
	1400	S 1100	.0357	
	1430	High Opaque	.0090	
	1500	S 1400	.0229	Torn Felt
	1600	S 1400	.0273	Rerun
	1715	S 1600	.0213	
1/8	0900	S 0800	.0180	
	1000	Filler Clay	.0037	
	1115	S 1000	.0208	
	1400	S 1200	.0207	

NOTE: S is for sample, number denotes hour of day obtained.

APPENDIX F
PAPER MILL SLUDGE CALCINATION

Object

The tests described in this report were made for the following purposes:

To determine the proper burning condition and produce a pigment having maximum brightness and minimum abrasiveness.

To produce enough material for the S. D. Warren Company to evaluate the quality of the product.

To a limited extent to obtain design data.

Introduction

In the continuing investigation by the S. D. Warren Company, a fourth series of tests was conducted in the Fuller Company's Research 3' x 30' rotary kiln on the calcination of paper plant waste sludge. The previous work performed by the Fuller Research Department was accomplished in August of 1965, April of 1969 and in November of 1970, on similar waste sludges to determine the feasibility of producing a useable product from these waste materials. The last of these tests, in November of 1970, was conducted in our 3' x 30' rotary kiln. All tests prior to that time were conducted in a small 1' x 12' laboratory kiln.

In the latest test it was determined that the excessive temperature required in the single pass system in order to obtain the required brightness, produced an abrasive product which did not meet the requirements for its end use. In order to produce a quantity of material at the suitable abrasiveness level, the present test was arranged for. Since the test prior to the test in November indicated good abrasiveness levels at operating temperatures in the order of 1500 to 1550^oF., it was determined that this would have to be the limiting temperature for any new attempts.

Based on the last 3' x 30' test where it was determined that in a single pass system at 1550°F. the required brightness could not be obtained, it was decided that in order for this new program to be successful, it would be necessary to utilize a two-pass system to obtain the required retention time at the lower temperature. The first pass was made at a short retention time limiting the upper temperature to 1200°F. This was done to remove the bulk of the water present in the initial feed material. The second pass would take the product from the first pass, with its 10 to 20% moisture, and calcine it at the retention time required at 1500-1550°F. to obtain the necessary abrasive qualities and whiteness.

Recommendations & Conclusions

Based on the information obtained from the test program, the following conclusions and recommendations are made:

As in the last test program using this 3' x 30' kiln, it was found that a pug mill could be used to prepare the wet feed prior to entering the kiln. The product from the pug mill ranged in size from 1/4" to 1" in size.

The kiln slope was kept at 3/8" per foot throughout the test. The speeds were varied between the drying phase and the calcining phase with the drying phase operating at the highest speed and the calcining phase at the slowest speed.

The drying phase took the 72% moisture sludge down to under 20% moisture at a temperature of 1200°F. with a feed rate of approximately 950 lbs/hr. The indicated loss of material during this drying stage was approximately 0.38%.

The physical appearance of the product from the drying phase was a grayish to black charred surface material which seemed to be dry, at least on the surface portions. The recovered material from the drying step contained an average of 16.6% moisture.

The second pass through the kiln gave us an overall product which met the specifications for abrasiveness and seemed to have a good brightness appearance to the naked eye.

The rate fed to the unit during the calcination test was in the order of 200 lbs/hr. This material was determined to have a moisture content on the average of 16.6% and a loss on ignition on an average of 60.6%.

The calcining tests, due to the re-introduction of a dry feed to kiln gave a higher loss of material than indicated in the previous programs. Approximately 40% of the material was blown out of the test unit, a majority of which is believed to come immediately from the feed pipe. Of the material that was lost, about 18.7% was found in the fallout chamber immediately off the back end of the kiln and 17.7% was blown through to the exhaust stack.

The overall losses during the test as operated, came to 42.55%. This includes the 40.10% in the calcining stage and the 2.45% in the drying phase. This figure is an approximate one due to the errors involved in the handling and weighing of the material three or four times during the test.

It is recommended that a system be designed that would do the required job in a single pass system. This would require a kiln with a larger length to diameter ratio than found in the 3' x 30' kiln used in this test program. It is felt that a system utilizing a single kiln would produce the same quality product as found by passing the material twice, at a much lower dust loading.

It is recommended that the dust catches from the system, which should be less than 30% based on our previous work, be returned to the pug mill system with the new wet feed reducing the total moisture content of the feed and yet allowing the wet material to carry the fine fraction back into the system.

The dust that was caught during the course of the test program was found to be a partially burnt material, gray to black in color still containing about 50% of its initial loss of ignition, but being almost entirely dry.

Material Tested

On December 17, 1970, 140 (55 gallon) drums, having a gross weight of 66,260 pounds was received. Composite samples of material taken during the course of the test program indicated that the moisture levels varied between 70 to 74% moisture. The feed materials were in the form of wet rolls of clay and laminated paper or fiber. The rolls ranged in size from 6" to 1' long and were approximately 3" in diameter. A few other rolls were up to 18" long and some were flattened out by impaction in the drums.

Product Evaluation

All evaluation during the course of the test was conducted by the S. D. Warren Company. Evaluations were primarily a test to determine the abrasiveness of the end product. As far as the brightness was concerned, this was visually observed by all parties involved and the best possible brightness obtained.

Material Balance

Input	Pounds
New weight (as received)	62,500
Average moisture 72.7%	
New weight (dry basis)	17,062
Average LOI at 1550 ^o F. - 60.6%	
Net weight (calcine basis)	6,722
Feed handling losses (450 lb. gross)	
dry basis	<u>51</u>
	6,671

Output Drying	Pounds
Drying (Pass No. 1) recovered	19,773.00
Average moisture 16.6%	
Recovery dry basis discharge	<u>16,489.68</u>
Shut Down Recovery	
After shut down	183 lb.
Fall out	<u>325 lb.</u>
	Dry basis <u>423.00</u>
Kiln discharge	16,489.68
Fall out	325.00
Kiln discharge after shut down	<u>183.00</u>
Total recovered (Dry Basis)	<u>16,997.68</u>
Unaccounted loss - 17,062.00 - 16,997.68 = 64.32 = 0.38%	
Output Calcining	Pounds
(second pass)	
Total feed to unit	19,773.00
Dry Basis	<u>6,507.03</u>
Recovered Kiln discharge	3,887.00
Shut down material	242.00
Fall out	<u>1,331.00</u> = 18.7%
Dust collector losses 6507.03 - 5350.00 = 1157.03 = 17.7%	
Total loss from production 6507.03 - 3887.00 = 2620.03	
= <u>40.1%</u>	
Total Processing Losses	
Feed dry basis	6,671.00
Dryer recovery	<u>6,507.03</u>
Dryer loss	163.97 = 2.45%
Calciner Recovery	<u>3,887.00</u>
Calciner loss	2,620.03 = <u>40.10%</u>
Total loss	<u>42.55%</u>

Details of Operation

During the week of December 30, 1970, an attempt was made to dry the wet paper sludge material in a parallel flow rotary dryer. This proved to be somewhat successful, however, the size of the unit, an 18" x 12' dryer limited its capacity to under 400 pounds per hour for drying purposes. It was then decided that a similar try would be made by passing the wet material through the 3' x 30' lined rotary kiln at 1200°F. and short retention time. This was accomplished in two days, December 30 and 31, 1970. A rate of 950 lbs/hr. was obtained in the unit giving an end product at 1200°F. of 15 to 20% moisture from its initial 74% moisture content. Starting on January 4, 1971, the remaining 110 drums of wet material left after the first initial drying attempt were fed through the dryer on a 24 hour basis. The entire work took until the morning of January 6, 1971. The total recovery for the drying step came to almost 20,000 pounds.

The kiln was immediately adjusted to give a long retention time and temperatures of 1550°F. and the partially dry product re-introduced to the system starting at approximately 1000 hours on January 6, 1971. In order to guarantee that the materials do not exceed the required 1500 to 1550°F., two thermocouple probes were installed directly into the kiln load at the hottest possible point. These were then used for controlling temperature for the entire program. Adjustments were started in the feed rate to the kiln at 1200 hours on January 6. The first initial product from the unit had a gray cast to it. At 1830 hours on January 6, it was decided that the color problem was due primarily to a lack of oxygen towards the back end of the kiln and also the low back end temperatures. At that point, the primary air fan to the burner system was put on to increase total air flow through the system. By 2100 hours, the first product, which indicated good color, was produced.

High abrasive levels were obtained, so on the morning of January 7, a decision was made to reduce the burning zone

temperature to 1500^oF. This required a reduction in the feed rate in order to maintain color. On January 7, by 1100 hours, indications were that the abrasive level requirements were being met, and that the color seemed to be stable indicating a good brightness. The kiln was being run at its lowest possible speed which was in this case 0.48 RPM giving a total retention time of approximately 3 hours within the kiln. The kiln produced approximately 35 pounds an hour for the remainder of the program. All attempts to increase this capacity gave an indication of an off-white color returning. This was being done by a visual inspection.

Since the figures for abrasiveness seemed to stabilize at an acceptable level, it was decided to maintain these conditions for the entire run. The conditions were held stable through January 10, 1971 at which time the run was terminated at 1930 hours. Upon completion of the run, the fall-out chamber was opened and checked for the first time. It was determined that if this was done during the course of the run, it would upset the burning conditions. At this time, approximately 1100 or 1200 pounds of material was removed from the fall-out chamber. This material is believed to have been blown back immediately from the feed pipe due to the re-introduction of the material into the feed end gas stream in the dry state. This had a tendency to increase the overall loss out of the system which did not report out as product. Total product recovery proved to be around 60% of the initial starting material. All products obtained during the course of the run, including hourly samples, were sent to the Bauer Brothers Company.

Equipment

Equipment in this test program consisted of the following: 3' x 30' kiln, which is lined with 70% alumina brick 4 1/2 inches thick. It has a variable speed range between 0.48 and 4 RPM, and a slope control between horizontal and 3/4" per foot. The exhaust end gases of the kiln pass through a brick fall-out chamber and then to a bag collector or to atmosphere when using natural draft. Temperatures in the kiln hot zone were measured optically and by direct

thermocouple probes within the load. View ports are located in the firing hood. The kiln is fired with the #2 fuel oil.

Pug Mill

The pug mill used in this program was an 8' long unit with variable speed drive. It consists of a single shaft chopping blade arrangement. Additional blades were added to the last 3' of the unit for this test to insure satisfactory shredding of the feed material. The pug mill was hand fed from the drums.

PROPERTIES OF DOMESTIC #2 FUEL OIL

API Gravity No.	33.3
Heating Value:	139,000 Btu's/gallon gross
	130,400 Btu's/gallon net
Flash Point	154°F.
Distillation:	10% at 425°F.
	50% at 505°F.
	90% at 580°F.
End Point	630°F.
Ash	0.001%

CHEMICAL ANALYSES

Carbon	87%
Hydrogen	12.5%
Sulfur	0.5%
Oxygen	0.1%
Nitrogen	0.1%

APPENDIX G
HURRICANE MILLING OF CALCINED SLUDGE

Material

01 - Light calcined sludge as received; 17.6 lbs per cubic foot and a brightness of 84.8.

02 - Grey calcined sludge as received; 18.0 lbs per cubic foot and a brightness of 50.9.

Objective

To demonstrate the ability of the Hurricane Pulverizer-Classififier to grind sludge to 99.9% minus a 325 mesh screen, and to upgrade by impurity extraction.

Summary & Conclusions

This material ground to average .80 micron compared to 1.7 micron in the last test.

The material was increased in brightness by 2 points in Run W2.

The two types of materials were kept separate and marked when shipped.

The Pulverizer can be highly recommended for grinding this calcined sludge.

Procedure

The Bauer Model No. 724 Hurricane was used for grinding and extracting impurities. The machine set-up is shown on Table No. I and the laboratory data is shown on Table No. II.

Wet wash screen analyses were taken at 10 pounds of pressure.

The processed material was sent to S. D. Warren for further evaluation.

TABLE I

January 25, 1971

Run No.	No. 1 ROTOR	No. 2 ROTOR	No. 3 ROTOR	No. 4 ROTOR	No. 5 ROTOR	INNER DISC. DIAM. (INCHES) AND No.	No. FINGERS IN CLASS- IFIER
W-1	21"	21"	Blank	21"	21"	5/21	24
W-2	"	"	"	"	"	"	"
W-3	"	"	"	"	"	"	"
- - - - -							
Run No.	FRONT DAMPER OPENING INCHES	BACK DAMPER OPENING INCHES	SKIM- MER OPENING INCHES	FEED SCREW PITCH AND RATIO	UPPER FAN DIAM. INCHES	BAFFLE I.D. INCHES	
W-1	1/2"	1/2"	4 x 2	5 x 4 12:18	28	18	
W-2	5/8"	5/8"	"	"	"	"	
W-3	5/8"	5/8"	"	"	"	"	

NOTE: TWO ANGLE IRON DEFLECTOR USED ON CLASSIFIER SHELF.

TABLE II

January 25, 1971

MAT'L NO.	MACH. R.P.M.	LBS. FED	TIME (MINS)	LBS. PER HR.	H.P. USED	OUTLET AIR TEMP. °F.	STACK MAN- OMETER. IN. OF H ₂ O
01	724	3080	120	4.5	1600	66	150 -2
01	"	"	2945	90.0	1960	75	160 -2
02	"	"	651	21.0	1860	75	160 -2
- - - - -							
MAT'L	POUNDS EX- TRACTED	SPECIFIC GRAVITY	FISHER PARTICAL SIZE	BRIGHTNESS	% -325 MESH		
01	<u>3.7%</u> 4.5	2.58	0.81	84.6	99.996		
01	<u>3.6%</u> 105	"	0.80	86.8	99.996		
02	<u>3.7%</u> 24	"	0.88	70.6	99.950		

NOTE: Equipment operated to extract rejects.

APPENDIX H

EVALUATION OF CALCINED SLUDGE AS A FILLER PIGMENT

Introduction

The Government Grant project concerns a possible solution to mill sludge disposal which entails calcining at a controlled temperature to burn out organic matter without causing abrasiveness and grinding to produce a pigment for mill reuse. This report covers a handsheet evaluation as filler pigment of a sample from a considerable quantity of such material calcined by Traylor and ground by Bauer. An evaluation of the same pigment, designated W-2 (2/12/71), for coating applications is underway. These combined studies should indicate the best possible mill reuse application for this pigment, which will then be tried in a mill run.

Summary

A pigment sample was dispersed at 15% in water under low shear conditions. It did not disperse very well and left a number of soft aggregates which would not pass a 120 mesh screen. There was no appreciable grit detected and dispersion should be much improved with higher shear conditions.

Abrasiveness against copper was determined by the New Jersey Zinc Method as described in TAPPI, Vol 43, No. 7, July, 1960, Pgs. 230A-232A. Weight loss averaged 18.5 mgs. which is in the same range as Hi Opaque clay. This latter material, although testing several times more abrasive than filler clay, has been in use at Cumberland Mills for several years with no apparent ill effects.

The W-2 dry pigment brightness determination is 83.4 and chemical assay is 14.3% as CaO, 9.2% TiO₂ and 76.5% clay.

For evaluation of physical and optical properties of W-2 as a loading pigment, four sets of 50 lb. handsheets, of 20 sheets per set, were made on the Noble Wood recirculating mold.

The furnish used was 60% Penobscot Bleached Hardwood Kraft, 40% Pictou Bleached Softwood Kraft, cobeaten in the Cycle beater to a Canadian Standard Freeness of 370. Preslurried W-2 pigment at levels of 12 and 28% based on fiber was added to the thick stock while similar levels of Columbia Filler Clay were used for controls. Fifteen lbs per ton of cooked Sta-Lok 333 cationic starch was added at the sheet mold as a retention aid and pH of the system was maintained in the 7.5 - 9.0 range.

The last five sheets of each set were tested for physical and optical properties and these results appear in Table I and Graphs I & II.

Conclusion

The W-2 pigment appears to have desirable properties as a filler pigment, particularly for its opacifying power. Slurrying may prove to be a minor problem - with the available mill equipment - and brightness, although somewhat superior to filler clay, is not very good. W-2 tends to increase bulk in the sheet and retains well. It would seem to be well-suited for grades where TiO_2 is currently used for opacity but brightness is not of top importance.

TABLE I

	W-2 Pigment		Filler Clay	
	12%	28%	12%	28%
Basis Weight 25 x 38, 500	50.4	49.5	50.0	49.1
Bulk Mils/Sheet	6.3	6.5	6.2	6.0
Bulk Factor cc/gm.	2.15	2.25	2.13	2.10
B + L Opacity (^C 89 @ 50 lb)	90.8	95.2	87.9	90.4
Kubelka-Munk S x 10 ²	8.1	11.0	7.1	8.4
K x 10 ⁴	12.6	18.6	9.5	11.4
Alinco Brightness	79.4	79.3	79.1	78.3
Mullen Burst Dial	24.0	13.0	25.0	15.0
% Mullen	47.0	26.1	49.9	30.7
% Ash In Sheet	8.5	20.2	9.6	18.6
% Filler In Sheet*	10.2	24.3	11.2	21.6
% Pond to Wire Retention	94.8	70.8	70.4	71.9

*Ash Factors: W-2 = 0.83

Columbia Filler Clay = 0.86

APPENDIX I
DEMONSTRATION GRANT - PAPER MACHINE TRIAL

Purpose

The purpose of this report is to describe the procedures and results of the paper machine trial using calcined sludge as a filler pigment.

Procedures of the Trial

Pigment Slurry

The pigment was slurried in the Cowles dissolver with water at 20% solids. Lab work indicated that the sludge filler was difficult to disperse. As a result a fifteen minute treatment in the Cowles was used to slurry the sludge. All batches in the Cowles showed soft lumps on a 120 mesh sieve. The high pH of the sludge caused the white water pH to jump from 8 to 10.6 in an hour. As a result froth became more noticeable. Alum addition was used to correct this problem.

Furnish Preparation

The pigment slurry was added to the pulp mixture in the beaters in proportions shown below.

Furnishes: English Finish

Basis Weight	45-lb	45-lb	70-lb	50-lb	50-lb
	Blank	T	T	T	T*
	99621	99828	99830	99829	99829
Hardwood(%)	30	30	30	30	30
Broke (%)	70	70	70	70	70
Clay (%)	19.6	--	6.4	6.4	--
Titanium Dioxide	2.5	--	--	--	--
Sludge Filler (%)	--	19.6	14	16.8	22.4
Starch (gpm)	1.0	1.0	1.0	1.0	1.0
Size (gpm)	0.4	0.4	0.4	0.4	0.4
Alum (gpm)	--	0.25	0.25	0.25	0.25

*Low Brightness

Sludge

Size Press: 5.2 to 6.2% of PG 280 solids.

Paper Machine Running

The trial was started at 9 a.m. Previous to that time the paper machine had been making 45 pound (per 3300 ft.²) Publishers English Finish Offset, which was used as a blank for trial comparison. The trial was started on the same grade. After two rolls of the 45 pound were made, the basis weight was changed to 70 pound. Three rolls of the 70 pound were made and the basis weight was changed to 50 pound. Total running time of the entire run was 15 1/2 hours - from 9 a.m. on 3/31/71 to 12:30 a.m. on 4/1/71. A summary of the weight of paper produced follows:

<u>Grade</u>	<u>Order No.</u>	<u>Basis Weight² per 3300 ft.</u>	<u>Number of Rolls</u>	<u>Total Wgt. Made</u>
Pub.E.F.	99828	45 lb.	2	3600 lbs.
" " "	99830	70 lb.	3	4400 lbs.
" " "	99829	50 lb.	5	5980 lbs.

Testing

During the paper machine run the Quality Control Department tested the paper as it normally does to assist the Paper Machine Department in making the paper to the desired specifications. The Research Department also tested the paper and took samples of the wet end of the paper machine to determine pigment retention.

Paper Machine Department Test Results

Filler retention: 45-lb. Blank - 59.8% without alum
45-lb. Trial - 22.5% without alum
45-lb. Trial - 55.0% with alum
70-lb. Trial - 70.6% with alum

Print Ratings: 45-lb. Trial - Fair
70-lb. Trial - Fair
50-lb. Trial - Fair
45-lb. Blank - Fair Plus

	<u>Wax</u>	<u>Ash</u>
Wax - Ash Tests: 45-lb. Blank -	14	16
45-lb. Trial -	18	12
70-lb. Trial -	13	17

	<u>Wax</u>	<u>Ash</u>
50-lb. Trial -	13	16
45-lb. Blank -	13	16

Research Department Test Results

<u>Sample</u>	<u>Reel</u>	<u>Basis Wgt.</u>	<u>Mullen</u>	<u>Tear W/A</u>	<u>G.E. Bright.</u>	<u>Opacity</u>
45-lb. Blank	2	44.55	23	37/31	75.76	92.44/92.6*
45-lb. Trial	1	43.45	27	31/33	76.24	90.74/91.3*
45-lb. Trial	2	39.52	20	28/33	76.56	92.52/94.3*
70-lb. Trial	1	68.05	31	61/70	77.24	97.98
70-lb. Trial	2	70.13	35	70/72	77.38	98.02
70-lb. Trial	3	65.10	32	61/68	76.62	97.74
50-lb. Trial	1	47.40	24	34/31	74.78	94.28/95.3**
50-lb. Trial	2	47.44	25	40/49	73.86	94.76/95.7**
50-lb. Trial	3	48.08	245	38/45	73.14	94.62/95.0**

* Corrected to 45-lb. basis weight

** Corrected to 50-lb. basis weight

	<u>SX10²</u>	<u>KX10⁴</u>	<u>Fold W/A</u>	<u>MI Size</u>	<u>Gurley Density</u>	<u>Bulk Factor</u>
45-lb. Blank	9.10	23.8	36/26	33	10.2	1.08
45-lb. Trial	8.31	23.2	117/28	28	61.9	1.10
45-lb. Trial	10.31	26.0	99/31	10.9	73.5	1.32
70-lb. Trial	10.09	25.2	42/14	26.5	29.3	1.26
70-lb. Trial	8.78	19.1	53/25	20.8	41.8	1.32
70-lb. Trial	9.56	25.3	46/34	59.1	51.6	1.21

50-lb.						
Trial	9.50	2.95	69/29	14.0	87.3	1.12
50-lb.						
Trial	9.38	32.9	105/31	6.6	55.3	1.19
50-lb.						
Trial	9.19	34.8	57/38	18.1	64.1	1.11

One of the critical tests on a trial of this sort is the Quality Control Print test. This involves printing 100 sheets (17" x 22") of the paper on a commercial sized Miehle offset printing press which is housed in the Print Testing Department. Samples of almost all printing papers produced at the Westbrook mill are tested by this technique on a 24-hour basis. After the paper is printed the printer rates the printability of the paper as Good, Fair+, Fair, Fair-, and Poor. If the rating is poor, the paper is rejected for shipping as first quality; Fair- is a borderline condition; and Fair, Fair+, and Good are shippable in first quality.

Results

Generally, the results of this run were very good. The paper met at least the minimum specification in all the tests, and in some cases was better than spec. The major problem of the run was the high pH of the pigment slurry, which caused poor retention at the start. The addition of alum reduced the pH to normal and the retention improved.

The opacifying power of the calcined pigment was good enough to produce the desired opacity at lower than normal ash levels. Therefore, on the 70 pound and part of the 50 pound clay was added to meet the ash spec. Since clay is less expensive than fiber it is advantageous for us to run the ash level as high as possible and still meet the strength specifications of the product, even though the opacity is over spec.

The low brightness calcined sludge produced the desired shade properties without any dye being required. This means that this pigment would be adequate for low

brightness papers, but inadequate for high brightness papers.

Conclusions

The Demonstration Grant calcined pigment can be used as a filler for making some grades of printing papers. The high pH of the slurried pigment will require more care and control than our normal pigment additions. This is not a major deterrent to the use of the calcined pigment, however.

APPENDIX J
PRINT TRIAL - DEMONSTRATION GRANT

Purpose

The purpose of this report is to present the results of the commercial print test on the Demonstration Grant Paper.

Discussion

In order to get a reasonable evaluation of the Publishers English Finish made on #8 Paper Machine under the Demonstration Grant Project, the Sales Department arranged for 1000 sheets of the 50 pound paper to be printed at Livermore and Knight Company in Providence, R. I.

The form being used was a biology book for D. C. Heath Publishers. Bookman Offset (50 pound) was the paper being used to make the book and the trial paper was printed at the trial-end of the run. On May 11, one side was printed on a 77" Miehle offset press, two-color (black and green), 4000 impressions per hour. The paper ran very well on the press, produced an excellent print, and the blankets and press were clean after printing. The other side was printed on a 77" Harris four-color press at a rate of 2300 impressions per hour. The paper again ran very well.

In addition the Print-Testing Department at S. D. Warren printed the Supplement to "Warren's Standard" on May 24 and 25, 1971, and the following is a summary of press conditions.

Press: Harris 26" x 27" LUS
Sheet Size: 17 1/2" x 22 1/2" grain long
Number of Pages: 12
Finished Size: 8 1/2" x 11" booklet
Offset Blanket: Dayco Black #8312
Ink: General Printing Ink, Publication Black NC66-1580
Ink Tack: 12.8
Fountain Solution: Lith-KemKO Etch. 14⁰ Baum Gum
 Arabic, Isopropyl Alcohol
pH of Fountain Solution: 3.5
Printing Plates: Warren Fotogold 12 Pt.

Folder: Baum 23" x 32"

Number of Sheets Printed: 30,000

Bindery: Northeast Bindery, 335 Forest Ave., Portland

Conclusions

This commercial printing trial of the Demonstration Grant Paper was a success with no problems.

The paper performed satisfactorily at S. D. Warren on both the printing press and bindery operations. Some static electricity was noted, but not enough to give production problems. The blanket showed a slight filler buildup after 10,000 impressions in the non-image area. Felt hairs and clay lumps were also noted. None of the above created print interference.

The sheets delivered a good flat load with the ink drying and binding to the surface very well. From the Print-Testing Department's point of view the sheet showed good runnability for single color work.

APPENDIX K

FULL SCALE SYSTEM

Cost estimates from the Fuller Company and the Bauer Brothers Company detail quite accurately the major components needed for a 40 tpd full scale sludge pigment recovery system. In addition to those costs the following should be added:

	<u>Material</u>	<u>Mill Labor</u>	<u>Sub-Contract</u>	<u>Total</u>
Pumping Sta. for Dilution Water	10,000	3,000	7,000	20,000
Mix Tank, Agitator & Controls	15,000	3,000	3,000	21,000
Pumps & Piping	17,000	5,000	15,000	37,000
Centricleaners	42,000	1,000	7,000	50,000
Vacuum Filters	78,000	10,000	12,000	100,000
Sludge Conveyors	25,000	2,500	2,500	30,000
Pigment Storage & Distribution Building	140,000	20,000	40,000	200,000
	<u>100,000</u>	<u>15,000</u>	<u>100,000</u>	<u>215,000</u>
	\$427,000	\$59,500	\$186,500	\$673,000
		Sub Total		\$673,000
		Freight		5,000
		Contingency @ 15%		<u>102,000</u>
		Total		\$780,000

Bauer Bros. submitted a quotation covering the No. 724-Pulverizer System that will handle 1-Ton per hour of feed.

This quotation covers all of the grinding, collecting equipment, motors and starters. In addition to this equipment, a hopper ahead of the pulverizer and piping and wiring to and from the starter panel are required.

Construction prices vary from one area to another so installation costs are not given.

Transportation charges are \$5.00 per hundred.

The total weight on the No. 724-Pulverizer System would be approximately 9000 lbs.

Operating requirements of the System
After the equipment is setup and operating, it would only require a touring operator for periodic checking.

The machine can be torn down and rebuilt within four (4) hours after the maintenance people become familiar with the machine, and if a hoist is installed for removing the heavy parts.

Parts cost on calcined clay has been running 30 to 50 cents per ton.

No fuel required on the Pulverizer.

Electrical requirements covered in the quote.

Following are the load weights on the various items quoted:

No. 724-Pulverizer with motor - 4430 lbs.

No. 25-Exhauster with motor - 570 lbs.

Starter - 400 lbs.

84FK-40 Flex-Kleen Collector - 3600 lbs.

Quotation

One Model No. 724 Bauer Hurricane Pulverizer of welded carbon steel construction. Equipped with whizzer classifier and integral high pressure fan. With high alloy replaceable rotor blades, classifier fingers with carbide face. The mill housing to be lined with Ni-hard liners and the classifier housing section to be lined with M-alloy. Equipped with a screw feeder with a 1/2 H.P., D.C. variable speed motor, feed hopper, drive chain and sprockets, circulating oil pump with 1/4 H.P. TENV motor and four gallon reservoir. Including Dyna-V-Belts and Sheaves.

Price

\$15,875.00

One 75 H.P., Type K, 1800 RPM, 3 phase, 60 cycle, 460 volt, vertical Motor, dripproof design, with special shaft and sliding base for belt drive.

Price \$ 1,284.00

One Integrated Starter and Automatic Control System consisting of a NEMA-12 enclosure containing a mainline circuit breaker, a main motor starter, (cross-the-line), a lube pump motor starter, feed drive solid state D.C. converter, circuit protection device, wired complete with sequence interlock and ready for mounting. Also included is an operator's station (for remote mounting) consisting of a percent of load indicating ammeter (main motor) start-stop stations for lub pump, main motor and feeder motor, feeder drive speed adjustment control mounted in a NEMA-12 enclosure for 460 volts, 3 phase, 60 cycle service.

Price \$ 2,520.00

One Special Feed Hopper with magnetic Grate Bars, installed in a Drawer type frame for easy cleaning and inspection.

Price \$ 650.00

One Model 84FK-40 Dust Collector, carbon steel construction including 60° hopper cone, quick opening side and top access doors, inside walkway, compressed air header, solenoid valves, internal air piping, 11 oz. dacron filter bags, mild steel bag cages, type 304 s/s bag clamps, solid state and mechanical stepping switch relay timer, prewired and magnehelic pressure gage, air pressure gage.

Price (F.O.B., Chicago, Illinois) \$ 3,640.00

One Model No. C-8" Rotary Airlock with 1/2 H.P., T.E.F.C. Gearhead motor, complete with chain drive

guard and mounting plate. For lower end of Dust Filter Cone.

Price \$ 995.00

One Model No. 25 Exhauster with Belts and Sheaves, including a 5 H.P. motor, 1800 RPM, 460 volts, 3 phase, 60 cycle open motor.

Price \$ 667.00

Total Price \$25,631.00

NOTES: The above prices are F.O.B., Springfield, Ohio, except where otherwise noted, and are firm for 60 days.

These prices do not include any taxes which, if applicable, are to be paid by the Purchaser.

Service Supervision will be supplied at the rate of \$100.00 per diem, plus Travel and Living Expenses, however, on New Equipment Start-Up, the \$100.00 per diem charge will be waived for the first day. (1)

The price of motors and other electrical equipment are subject to escalation based on the motor manufacturers' discounts at the time an order is received.

TERMS: Net 30 days from date of invoice.

DELIVERY: Estimated within 3 to 4 months.

The Fuller Company submitted budgetary information covering a rotary kiln and associated equipment as follows.

Based on the information obtained during the various tests run on S. D. Warren material in their Research Department, it is estimated that the equipment for the 40 TPD application would require a feed at 70% moisture

amounting to 133 TPD and the material or product exiting from the kiln discharge would be approximately 16 TPD.

The equipment offered would consist of a rotary kiln, and this kiln would be so equipped with a suitable chain section so that in effect the kiln becomes a combined dryer and kiln.

It is estimated that the fuel requirements per ton of material fed to the kiln as feed would be approximately 3,260,000 BTU's and basing this figure on the material discharged from the kiln the thermal requirements per ton of material or product discharged from the kiln would then become approximately 23,500,000 BTU's.

40 TPD

For this application, we would suggest the use of a rotary kiln, 6' 6" diameter x 160'0" long. This kiln would be on a slope of 3/8" per foot and have a retention time of approximately 240 minutes. The kiln would be equipped with the necessary drive and auxiliary drive, feed end housing and discharge end fire hood. The kiln would be equipped internally with a suitable chain section.

Also included in this package would be the supply of a Peabody Mechanically Atomized Type Burner with a maximum heat release of 24,000,000 BTU's/hour. This burner would be of the water cooled type and would be equipped with a suitable Duplex Pump and Heater Set which would utilize steam for the preheating of Bunker C oil. The pump and heater set would have a maximum capacity of 4 GPM. The burner pipe itself would be equipped with a suitable primary air fan and damper for controlling the volume of air to the burner. The burner package would also include a suitable flame failure device for insurance purposes. Also included in this package would be the necessary ducting to connect the primary air fan to the burner pipe. It should be noted, however, that this

SELECTED WATER RESOURCES ABSTRACTS INPUT TRANSACTION FORM		1. Report No.	2.	3. Accession No. <div style="font-size: 2em; font-weight: bold; text-align: center;">W</div>
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7. Author(s) Labrecque, R. P. and Weymouth, T. E.		8. Performing Organization Report No.		
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16. Abstract <p>A process was developed and tested in a full scale trial wherein the pigment present in waste sludge was reclaimed. Sludge resulting from the primary treatment of white waters was diluted to less than 0.75% solids, centricleaned, dewatered to 30% solids, shredded, dried, and burned in a rotary kiln. The ash that resulted - the pigment - was then pulverized and used as filler pigment in the papermaking process.</p> <p>A pigment of acceptable abrasiveness and a GE brightness of 84-85% could be produced provided that the sludge was centricleaned and the temperature in the kiln kept below 1600°F.</p> <p>A system capable of processing 40 dry tons per day primary treatment sludge would produce reusable filler pigment at a net cost of \$50 per ton. Compared to the delivered cost of virgin filler clay (\$38 per ton) it can be seen that full scale pigment recovery utilizing this system is not economically justifiable at this time.</p>				
17a. Descriptors Material Recovery Wastes, Pulp and Paper Wastes, Sludge*, Incineration*, Pigment Recovery*.				
17b. Identifiers Centricleaning Sludge, Paper from Sludge, Pulverization, Burning, Solid Wastes, Sludge Treatment.				
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