OF ASH FROM PAPERMILL SLUDGES



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INVESTIGATION OF REUSE POTENTIAL OF ASH FROM PAPERMILL SLUDGES

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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory - Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

"Investigation of Reuse Potential of Ash From Papermill Sludges" is a product of the above efforts. Two techniques for recovery of filler from fine papermill high ash sludges, screening and wet oxidation, were evaluated for their technical feasibility. The alternative of screening the sludge and using the material passing through the screen as recovered filler was investigated in cooperation with two mills. The wet oxidation scheme appears technically feasible. The screening alternative would probably have to incorporate a bleaching step before it would be deemed acceptable. The technical feasibility of oxidative bleaching was demonstrated.

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ABSTRACT

Two techniques for recovery of filler from fine papermill high ash sludges, screening and wet oxidation, were evaluated for their technical feasibility. The alternative of screening the sludge and using the material passing through the screen as recovered filler was investigated in cooperation with two mills. The screening for recovery of filler was conducted at the mill Then the material was shipped to Western Michigan University where selected grades incorporating the recovered filler The paper manufactured was shipped to Rocheswere manufactured. ter Institute of Technology where it was printed on their web offset press. The grades simulated were found lacking only in that the brightness of the paper was from four to seven points lower than grades made with virgin filler. The wet oxidation alternative was evaluated in a similar manner with a cooperating mill. The wet oxidized, recovered fillers only slightly lowered the brightness of the sheet simulated and gave an increase in opacity in exchange. The wet oxidation scheme appears technically feasible. The screening alternative would probably have to incorporate a bleaching step before it would be deemed accept-The technical feasibility of oxidative bleaching was able. demonstrated.

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SECTION I

CONCLUSIONS

Fractionation of the high ash sludge from the primary clarifier of a fine paper manufacturing operation by use of a vibrating screen results in a reusable high filler content fraction, which passes through the screen, and a high organic material content fraction, which is retained on the screen. The high organic content fraction becomes amenable to incineration, should circumstances dictate. Fifty to seventy-five percent of the sludge may be reclaimed in the high ash fraction as possible reusable material and would result in a parallel decrease in the mass of sludge requiring disposal.

In recovery of fillers in the paper manufacturing process, selection of screen mesh and feed rate are specific to each mill due to the varying nature of the sludges and the tolerance of the mill's grade structure to contamination introduced with the recovered materials. In the course of this study, a mill manufacturing coating base stock opted for a 100-mesh screen whereas the mill making uncoated fine paper opted for a 230-mesh screen. Less organic contamination is associated with the use of a finer screen mesh. However, a smaller amount of material is recovered.

Based upon pilot work, the screened, recovered filler did not affect the physical properties of the sheet made in this study when used to partially replace virgin filler. The highest level of replacement observed was 83 percent of the filler clay, constituting 68 percent of the total filler. The screened, recovered filler did not (a) affect the printing properties of the sheet in the grades examined, (b) increase the dirt count of the sheets or (c) adversely affect the filler retention characteristics of the pilot paper machine. Moreover, the recovered materials have abrasive properties comparable to normal filler grade clay utilized in the paper industry.

Utilization of screened, recovered filler led to an increase in opacity of the sheets. The 50-lb (74 gm/m²) coated offset grade gained 3.6 points in the base stock and 1.8 points in the final coated product. The 50-lb (74 gm/m²) uncoated offset sheet gained 1.2 points and the 30-lb sheet (44 gm/m²), 0.4 points.

Utilization of screened, recovered filler, however, did adversely affect the brightness of the sheets. For the 50-1b (74 gm/m²) coated offset grade, this amounted to 7 points in the coating base sheets; 4 points in the final coated sheet. For the uncoated offset sheet, it amounted to 4.6 points for the 50-1b (74 gm/m²) sheet and 0.8 of a point for the 30-1b (44 gm/m²) sheet. All sheets reflected deteriorating brightness, with differences in magnitude attributable to utilization of (a) different levels of addition of the recovered filler and (d) different blends of filler materials. Subsequent study indicates that oxidative bleaching techniques have potential for brightening screened, recovered filler.

Wet oxidation produces a reusable filler material from high ash sludges. Fillers recovered by wet oxidation did not (a) harm the physical properties of the sheet, (b) increase the dirt count of the sheet, or (c) adversely affect the printing properties of the sheet at addition levels employed in this study. Futhermore, tests of the wet oxidized, recovered filler indicated it to be no more abrasive than normal filler clay.

The wet oxidized, recovered filler from the sludge evaluated imparted 2.6 points lower brightness to the sheet but gave an increased 4.9 points of opacity. From the standpoint of product brightness, wet oxidation would have to be considered superior to separation by screening. However, the wet oxidized, recovered filler from the sludge evaluated has a buffer effect and consumed several times as much acid in controlling the pH to 4.5 during the machine trial as did No. 2 coating clay when it was used as filler. In comparison with No. 2 coating clay, titration of the wet oxidized filler from pH 7 to pH 4.5 required 48 times the quantity of sulfuric acid.

The screening alternative looked more promising than wet oxidation for the conventional mill (nondeinking) but would not be feasible for the deinking mill due to the low brightness of the recovered product. The wet oxidation alternative would appear workable on either type of sludge but involves a good deal higher capital investment cost than the screening.

The wet oxidized, recovered filler was preliminarily evaluated as a coating pigment. The evaluation showed that this material would be unsuitable as a coating pigment due to its heterogeneous nature, lower brightness, particle size distribution, and dispersion difficulties.

SECTION II

RECOMMENDATIONS

This study has addressed the technical feasibility for recovery of filler materials from high ash sludges and subsequent reuse in the paper manufacturing process. Though separation techniques employed herein involved screening and wet oxidation, efforts were not made to optimize either. As a consequence alternative screening devices besides the Sweco vibrating screen, as well as alternative wet oxidation processes, should be evaluated for their relative merits as a means for filler recovery from clarifier underflow.

Remaining to be quantified are the operational and economic implications of filler recovery on a larger scale continuous basis. Alternative means for filler recovery warrant further evaluation on a continuously operating pilot plant scale at an actual mill site. Elements of such further study would appropriately include such issues as how the variable nature of the clarifier underflow would affect properties of the recovered filler and how much equilization capacity would be required to dampen variation of those properties. It could also address the issue of system dependability and the effect of recovered filler addition to a full scale paper machine system. Cost analysis would appropriately follow to determine the economic feasibility of filler recovery by screening or wet oxidation in an overall residuals management program.

SECTION III

INTRODUCTION

Among the solids which are accumulated in the treatment of wastewaters of pulp and paper origin are those lost from the papermaking process and subsequently separated during primary clarification. Land disposal is the largest single method practiced in the pulp and paper industry for disposal of these Sludges with ash contents exceeding 50 percent are commonly associated with the manufacture of (a) deinked pulp and paper and (b) integrated and nonintegrated fine papers. absence of by-product recovery opportunities, land disposal remains the only feasible method of disposing of these sludges. Of the mills which must deal with the disposal of high ash sludges, more than 100 are located in or adjacent to metropolitan areas where land available for solid waste disposal is limited. Combined with declining land availability in restricting the practice of land disposal are declining public acceptance and increasingly stringent regulation. Thus, an impetus for development of by-product approaches is clearly suggested.

This problem was identified as early as 1951 when the Kalamazoo River Improvement Company (a group of six paper companies in southwestern Michigan practicing deinking) initiated work under the direction of Dr. A. H. Nadelman to develop alternative solutions to the problem (1). Two approaches were pursued: the first to convert sludge into a product which could be reused on site in the papermaking operation, and the second to study the possibility of other industrial uses of the sludge. Two processes for sludge modification were investigated and included: (a) drying and powdering and (b) calcining and ball milling the sludge.

The calcined product was of adequate brightness for reuse in the papermaking process, though agglomerated. As a consequence, it was necessary to pulverize the reclaimed material with a ball mill. Though promising from the standpoint of appearance, the final product, when evaluated for abrasiveness with the Valley abrasion tester (2), was found to be more than 10 times as abrasive as commercial clay, thus down-grading its desirability as a paper filler material. The material was also evaluated for use as a filler pigment in rubber products and asphalt tile and found to function satisfactorily. However, for those

applications the cost of calcining and ball milling made the recovered fillers more expensive than virgin filler.

Attention was, therefore, given to other techniques for recovering the filler, including electrophoretic separation and flotation. Electrophoresis seemed an unlikely alternative since the cost of electrical power needed to do the separation was more than the value of the product produced. Flotation, however, was shown to have some potential for making a partial separation, but experiments were not carried far enough to reach a definite conclusion.

In early 1962, S. D. Warren Company began development work in the field of sludge incineration and arrived at a viable system for their fine paper mills which they felt would economically dispose of their primary high ash sludge while generating from it a usable paper filler material. A full scale system, which was constructed and operated under an EPA demonstration grant, is shown in Figure 1. It consisted of (a) a centricleaner system to remove grit, (b) vacuum filters to dewater the sludge, (c) a shredder to break up the cake, (d) a rotary kiln to incinerate the sludge and (e) a pulverizer classifier to break up the incinerated product (3). It was observed that if the kiln temperature was below 1500°F (816°C), the product did not achieve the desired brightness. However, if the kiln temperature exceeded 1600°F (871°C), the product was exceedingly abrasive, even after pulverization. By maintaining kiln temperatures between 1500 to 1550°F (816 to 843°C) with a 90-minute detention time, a product could be obtained which had a G.E. brightness of 84 to 85 percent. Moreover, it was only two to five times as abrasive as normal filler clay. Full scale paper machine trials employing the recovered filler were subsequently conducted, and the paper was commercially printed with no difficulties encountered. Based on 1971 prices, the recovered filler was estimated to cost \$50/ton to produce. In comparison, virgin filler cost only \$38/ ton and was not plaqued with the abrasive properties. However, the costs associated with solid waste disposal at specific mill sites would warrant inclusion in the economic balance. Because of the abrasive properties of fillers recovered by incineration, alternative techniques for recovery of filler material from high ash sludge continued to warrant investigation, again with the aim of providing a product which could be reused at the mill as a paper filler material. As a result, in 1973 the National Council of the Paper Industry for Air and Stream Improvement (NCASI) studied four separation techniques under laboratory conditions to attempt recovery of filler from high ash sludge. These included (a) foam flotation, (b) centrifugal separation (centricleaning), (c) screening and (d) wet oxidation. capability of each was judged on the basis of (a) the degree of separation (proportion of the recovered product which is filler), (b) recovery (the proportion of the original filler in the sludge which is recovered) and (c) brightness of the final product in

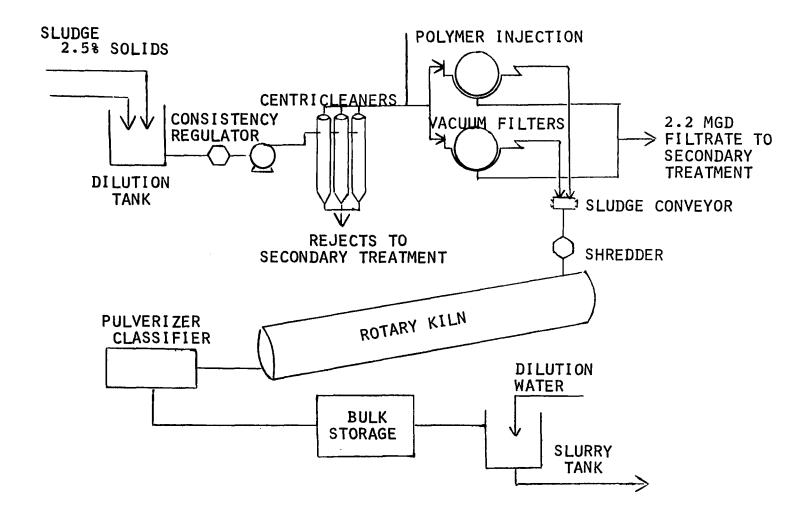


Figure 1. Full scale pigment recovery system.

comparison to the original sludge. Appendix A contains the results of these tests as well as details of the experiments. high ash sludge samples were studied: one from a fine paper mill practicing deinking and one from a mill not deinking, which will be termed a conventional mill. The recovered fillers were evaluated for abrasiveness by a modification of the flat disc method (4,5) and found to be nonabrasive. Based upon this evaluation, the screening and wet oxidation techniques showed most promise. Further demonstration of the acceptability of filler materials recovered by these techniques for reuse in the paper manufacturing process required pilot plant scale trials. To that end, it was proposed to evaluate on pilot scale paper manufacturing equipment the technical feasibility of reusing filler materials recovered by the screening of sludges from two fine paper mills and the wet oxidation of a third sludge generated at a fine paper mill employing deinking. In the course of doing so, the screening alternative was evaluated for both coated and uncoated grades of web offset printing paper, whereas the wet oxidation alternative was evaluated only for uncoated web offset printing paper.

SECTION IV

EVALUATION OF SCREENING AS A RECOVERY

ALTERNATIVE FOR A MILL MANUFACTURING COATED FINE PAPER

EXPERIMENTAL DESIGN

The overall experimental design involves simulating on a model paper machine production of grades of paper similar to those produced at cooperative mills, using both virgin and reclaimed fillers, and testing the papers to distinguish property changes due to reclaimed filler content. In evaluating the potential use of recovered fillers in the manufacture of coated fine paper, a cooperative mill chose from its grade structure a high volume grade felt to be capable of accepting recovered filler as part of its furnish. In the normal production of this particular grade, all of the filler normally employed in the sheet enters in the coated broke. Thus, recovered filler addition would increase the ash content of the grade.

Utilizing virgin material provided by the mill, manufacture of coated fine paper was simulated on a 24-in. (0.6 m) pilot fourdrinier paper machine at Western Michigan University (WMU) at the normal ash level of 5 percent, as well as a higher 8 percent level representative of the ash content associated with projected use of recovered fillers. These sheets were compared with a similarly manufactured sheet containing a combination of virgin filler at the 5 percent level and sufficient recovered filler to increase the ash content to 8 percent, thereby simulating the way in which the mill would utilize the recovered filler.

The rolls of paper manufactured were shipped to the cooperating mill to be coated and supercalendered. The coated paper was subsequently evaluated for its physical and optical properties and shipped to Rochester Institute of Technology for printing on a commercial four-color web offset press.

SCREENING PROCEDURE

The screening was accomplished at the research and development center of the cooperating mill using a 30 in. (0.76 m) diameter Sweco vibrating screen. The primary clarifier sludge

was trucked from the mill in drums, diluted to 2 percent, and screened using a 100-mesh sieve.

Mill personnel had previously conducted studies varying the mesh size from 325 mesh to 100 mesh. Initial examination of the recovered fillers for brightness and dirt speck content suggested that the separation effected by the 100-mesh screen would result in an acceptable quality material for the intended end use. Apparently a significant proportion of the dirt and grit were preferentially retained on the screen with the more highly fibrous organic fraction, allowing a more selective passage of filler components through the sieve.

Furthermore, the 100-mesh screen had the added advantages of (a) allowing a higher throughput rate, 2 to 3 gpm (7.6 to 11.4~l/m) and (b) recovering a larger fraction of the clarifier underflow as reusable material. The recovered fraction, which passed through the screen, amounted to 50 to 75 percent of the total sludge. Seventy to eighty-five percent of the possible filler was recovered. The recovered filler was thickened by settling and decanted to approximately 5 to 8 percent solids. In addition, it was treated with a biocide, 125~mg/l Dowicide G, to prevent bacterial degradation during subsequent shipment to Western Michigan University. There it was refrigerated upon arrival.

PILOT PAPER MACHINE TRIAL

The major raw materials for the pilot machine trial were provided by the cooperating mill. The groundwood pulp was dewatered on a large vacuum screen to approximately 18 percent solids and packed in polyethylene lined 55-gallon (210 l) drums for shipment. Pulp degradation was prevented by addition of Rx-28. The softwood kraft pulp was shipped in dry lap form to Western Michigan University. The starch (Cato 16) and No. 2 coating clay were shipped in dry form in 100-lb (45.4 Kg) bags. Coating clay was utilized because in the mill the ash content in the grade comes from addition of coated broke.

A run with a 1-day machine trial was devoted to each of three conditions designed to simulate (a) the usual manner in which the selected grade was produced in the mill, (b) the way the grade would be manufactured if recovered fillers were to be incorporated and (c) a control where the recovered filler was replaced by virgin filler.

In each case, the furnish was held constant throughout the run at 45 percent groundwood and 55 percent softwood kraft. The softwood kraft was refined from 660 Canadian Standard Freeness to 600 Canadian Standard Freeness (CSF) in a beater. The pressure was then taken off the bed, and the groundwood was added

and reslushed. The starch was cooked at 200°F (93°C) for 30 minutes at 6 percent solids and diluted to 3 percent solids for metering to the machine. The clay recovered filler, and starch were metered in continuously to a mix tank just before the headbox.

Distinctions between the various trials merit mention. During the first trial, the starch was metered in to give 1.75 percent starch. The No. 2 coating clay was slurried at 70 percent solids, diluted to 15 percent solids, and metered in at a rate to give 5 percent ash in the sheet. In the second trial, the starch feed rate was increased to give 2.25 percent starch, but the virgin clay feed rate remained at the previous day's level. The recovered filler, at 3 percent consistency, was metered in at a sufficient rate to result in a final ash level in the sheet of 8 percent. In the final trial on the third day, the starch level was left fixed at the previous day's level. However, recovered filler was not used and the feed rate of virgin filler was increased to yield an ash content of 8 percent. The recovered filler was replaced exactly with virgin filler.

Throughout the trials the basis weight was held constant at $45\ lb/3300\ ft^2$ (66.6 gm/m²). The alum level was maintained at 30 lb/ton (15 Kg/metric ton). All water used in the trial was deionized. Hardness was added back to give 200 grains (3420 mg/l). The headbox temperature was maintained at 95°F (35°C), and headbox pH was adjusted continuously with sulfuric acid to give a pH of 4.5 to 4.7. The sheet was not internally sized nor was a size press used. Three nips were used in the calender stack. The dandy roll was not used. The final sheet moisture was controlled to 3 percent \pm 0.5 percent.

During the run, the basis weight and moisture were controlled by the machine's Accu-ray system. Four times during each day's run 30-ft (9.1 m) paper samples were collected and stored in a constant temperature and humidity control testing laboratory for later evaluation. Concurrently control tests for basis weight, caliper, ash, and Scott Bond were conducted. The collected paper samples were subsequently cut every 2 feet (0.6 m) for a length of 20 feet (6.1 m) to provide 10 segments for testing each of the physical, optical and printing properties of interest. For each test, a strip from each of the 10 samples The 20-ft (6.1 m) length was selected on the was evaluated. basis of the basis weight fluctuation cycle which the model paper machine exhibits. In addition, 8 minutes prior to the taking of the paper samples, water samples were collected from the headbox, first tray white water and total wet end overflow. Water sample analyses, in conjunction with those conducted on the paper samples, were used to calculate retention values.

COATING TRIAL

Each day three to four 30-in. (0.76 m) diameter rolls of paper 24 in. (0.6 m) wide were made. These rolls were wrapped and shipped to the cooperating company for coating. Upon arrival, the rolls from each trial were rewound into large master rolls and trimmed to a 20-in. (0.5 m) width for use on the pilot blade coater. All rolls were coated using the coating formulation commercially applied on the simulated grade. It consisted of 100 parts dry weight of clay, and 15 parts dry weight of calcium stearate. The pilot blade coater was operated at a speed of 1000 fpm (305 m/min). The coated paper was supercalendered to meet a gloss specification of 50 ± 2. Samples of the coated paper were taken for testing of the physical and optical proper-The rolls of paper were then rewound into two composite rolls containing paper from each of the three test conditions. These rolls were wrapped and shipped to Rochester Institute of Technology for further assessment of printing properties.

PRINTING TRIAL

The composite rolls consisted of a leader of paper manufactured commercially by the mill, followed by segments from each of the three test conditions. In doing so, the rationale was to get the press set up and running on the commercial paper and then print at the same conditions the pilot paper machine manufactured paper. The printing was accomplished with a GOSS Commercial 30-in. (0.76 m), four-color perfecting press equipped with a brush dampening system. The four colors run in sequence were cyan (tack 14.2), magenta (tack 12.0), yellow (tack 10.0) and black (tack 8.2). The press speed was controlled at 600 fpm (183 m/min), and the temperature of the web out of the drier ranged from 200 to 350°F (93 to 177°C). There appeared to be little difference in the runability of the different components of the composite rolls. The printed samples were evaluated both by printing experts from the cooperating company and by Dr. E.W. Rayford of Western Michigan University. Dr. Rayford's comments on the printed samples are also included in Appendix B. He was asked to evaluate a series of number-coded samples on ink holdout, strikethrough, printing smoothness, pick resistance, fiber rise, sharpness and showthrough on a scale of 1 to 5: 1 - poor, 2 - fair, 3 - average, 4 - good, 5 - excellent. Results can be found on Table 6.

LABORATORY PHYSICAL, OPTICAL AND PRINTING TESTS

The paper was evaluated using methods commonly practiced in the paper industry. Following is a listing of the various tests performed and the method used for each. With the one exception, they are either Technical Association of Pulp and Paper Industry Standard Methods (6) or Useful Methods (7).

Basis Weight	T410
Caliper	T411
Tensile	T494
Stretch	T457
Sheffield Porosity	UM524
Sheffield Smoothness	UM518
Mullen	T403
Tear	T414
Fold	T511
Brightness	T452
Opacity	T425
Gloss	T480
IGT	Т499
K & N Ink	UM553
Wax Pick	T459
Dirt Specks	Т437
Hiding Power	(described below)

Hiding power is a control test which is used to monitor printing showthrough. The sample of interest is solidly printed on one side using No. 4 black printing ink (I.P.I.) at a constant speed of 0.4 m/sec by means of the IGT printability tester AC2. The sample is allowed to dry three hours and the brightness of the nonprinted side is taken. The brightness of a nonprinted sample is also measured, and the corresponding percent reduction in brightness due to the printing on the opposite side is calculated as:

HP = 100(1 - Original Opposite Side Printed Brightness Original Brightness)

SECTION V

DISCUSSION OF RESULTS FROM EVALUATION OF SCREENING AS AN ALTERNATIVE FOR FILLER RECOVERY FROM HIGH ASH SLUDGE FOR REUSE IN COATED FINE PAPER

SCREENING

Characteristics of the screening process and the recovered material is shown in Table 1. The pilot scale screening trials conducted at the cooperating mill achieved filler recoveries (ignition loss compensated ash recoveries) on the order of 70 to 85 percent; whereas, the laboratory results on a conventional sludge (nondekining) suggested potential filler recovery of 91.5 This difference in recovery is probably due to the percent. difference in composition of the two sludges evaluated. cooperating mill's sludge had a high groundwood content, making it very hydrous. Possibly it was harder to separate the fillers from the hydrous groundwood particles. The conventional sludge studied under laboratory conditions did not contain any groundwood. It would follow that the differing composition and properties of primary clarifier sludges among individual mills would require screening trials on a site-specific basis to determine their filler recovery potential.

PHYSICAL AND OPTICAL PROPERTIES OF COATING BASE SHEET

Table 2 lists the physical and optical properties of the uncoated base sheet for the three trial conditions. For comparison, the mill-manufactured base sheet properties are also listed. The column labeled "5% Control" represents simulation of the mill-manufactured grade. Comparing these two on a physical and optical properties basis, one would conclude that they are significantly different from one another on many of the properties.

The column labeled "8% Control" was to simulate the effect of increasing the ash content to the level of the sheet associated with reuse of the recovered filler. A comparison of results of the 5 and 8 percent virgin filler additions indicates increasing the ash had a negligible effect on the physical and optical properties.

Table 1. SCREENING CHARACTERISTICS FOR FILLER RECOVERY FOR REUSE IN COATED FINE PAPER

Parameter	Mill site data
Feed consistency (%)	2
Feed ash content (%)	38.2
Feed rate (gpm)/(1/min)	2-3/8-11
Screen diameter (in.)/(cm)	30/76
Mesh	100
Recovered ash content (%)	54.5
Purity (%)	64.1
Total solids recovered (%)	50-75
Filler recovery (%)	70-85
Brightness (% elrepho)	56.7
Solids loading rate (lb solids/min ft ²)/(Kg/min m ²)	.071 lb/min ft ² / .35 Kg/m ² min

Note: For comparison, brightness of No. 2 filler clay is 80 to 82.

Table 2. PHYSICAL AND OPTICAL PROPERTIES OF COATING BASE SHEET

			r==	
				Mill
			Recov-	manuf.
	5%	88	ered	base
Properties	Control	Control	filler	sheet
Basis weight 2 2.	47.0/69.6	45.2/66.9	45.8/67.8	46.3/68.5
$(1b/3300 \text{ ft}^2)/(gm/m^2)$			_	
Ash (%)	4.4	8.0	8.6	3.7
Caliper (.001 in.)/(mm)	5.0/0.13			
Opacity (TAPPI)	90.3	89.7	93.3	90.6
Felt brightness	68.4	70.4	62.8 a	72.5
(% elrepho)				
Wire brightness	68.6	70.4	63.4 ^a	73.4
(% elrepho)		[
Smoothness (Sheffield)	171	171	177	97
Porosity (HOP)	120	131	96	81
MD Tensile (Kg)	9.6	8.3	8.5	11.0
CD Tensile (Kg)	5.1	4.7	4.4	3.9
Mullen (PSI)/(kPa)	20.7/143	16.8/116	16.6/114	18.6/128
Mullen ply bond (PSI)/	172/1180	168/1160	175/1200	185/1270
(kPa)				
MD Tear (g)	62	62	55 a	46
CD Tear (g)	75	75	65 a	62
MD Fold (number)	157	111	84	157
CD Fold (number)	25	17	15	18
MD Stretch (%)	1.72	1.71	1.74	1.5
CD Stretch (%)	3.6	3.4	3.4	2.4
MD TEA $(Kg(m)/m_2^2)$	3.4	2.9	3.1	3.3
CD TEA $(Kg(m)/m^2)$	4.4	3.8	3.7	2.2
Dirt specks	554	413	428	384
First pass retention (%)	64.2	65.4	66.0	_
Overall retention (%)	79.0	76.9	76.8	

Significantly lower than 8% control sample at 99.5% confidence as indicated by a t test.

The column designated "Recovered filler" reflects the properties of the sheet made employing recovered filler to increase the ash content, thus simulating the way it would be utilized in the mill. The best basis for direct comparison with this sheet is the 8 percent control. The boxed values in the recovered filler column are statistically lower than the 8 percent control values. The apparent small loss in tear is not of practical consequence; however, the drop in brightness of the product is. The drop of 7.6 points in felt side brightness and 7.0 points in wire side brightness is of real concern. This loss in brightness caused by the recovered filler suggested the necessity for bleaching of the recovered filler prior to reuse and prompted the additional investigation of that option, discussed later in this report. It should be noted also that the brightness of all of the sheets made at Western Michigan University are lower than the products made at the mill. This was due to the preserved .groundwood discoloring during storage prior to use.

However, loss of brightness was accompanied by the gain of 3.6 points in opacity. The gain in opacity may be related to the presence of other filler materials besides clay in the recovered filler material. It is interesting to note that the dirt speck count did not significantly increase when utilizing the recovered filler, although all values are very high due to the groundwood content of the product.

The first pass and overall retention values, also reported in Table 2, demonstrate that utilization of recovered filler does not appear to have changed the retention characteristics on the machine.

COATING

The physical properties of the coated offset sheets are summarized in Table 3. There does not appear to be any significant deterioration in the physical properties of the sheet due to either the addition of the recovered filler or increase in ash content. Unlike results reported for the uncoated sheet, a difference in tear between the sheet containing the recovered filler and that containing exclusively virgin filler was not apparent. The result reported on the coated sheet was substantiated by additional tests, inclusive of mill testing, and is felt to be the more reliable.

The optical properties of the coated offset sheet are summarized in Table 4. The brightness of the sheet containing the recovered filler is 4 points lower than the 8 percent control, and its opacity is 1.8 points higher. Thus, the loss in brightness of 7.0 to 7.6 points in the base sheet is dampened in the coated product. However, it remains questionable whether the

Table 3. COATED OFFSET SHEET PHYSICAL PROPERTIES Summary

			Recov-	Mill
	5%	88	ered	manuf.
Physical properties	Control Control	Control	filler	sheet
2				
Coat wt (1b/3300 ft ²)/	6.15/	6.17/	6.05/	6.10/
(gm/m ²)	9.10	9.13	8.95	9.03
σ	0.10/	0.13/	0.16/	0.58/
	0.15	0.19	0.24	0.86
Caliper (.001 in)/(mm)	3.79/	3.73/	3.66/	3.55/
	0.096	0.095	0.093	0.090
σ	0.11/	0.13/	0.12/	0.11/
	0.003	0.003	0.003	0.003
MD Tensile (Kg)	11.95	10.0	11.1	33.9
σ	0.61	0.90	0.46	0.61
CD Tensile (Kg)	5.91	5.90	5.61	5.07
σ -	0.29	0.23	0.26	0.32
MD TEA $(Kg(m)/m^2)$	4.04	3.41	3.31	4.22
σ,	0.53	0.77	0.41	0.44
CD TEA (Kg(m)/m ²)	3.84	3.97	4.25	2.77
σ	0.72	0.46	0.46	0.36
MC Stretch (%)	1.75	1.50	1.61	1.57
AC DETECTION (0)	0.16	0.22	0.15	0.10
CD Stretch (%)	2.9	2.9	3.3	2.4
σ	ี ก	0.25	0.27	0.23
Porosity (Sheffield)	28.0 a	33.8	31.8	33.9
-	0.6	0.9	2.3	0.6
σ Porosity (HOP)	216	204	215	182
-	22	33	20	25
σ Smoothness (Sheffield)	53.2	40.1	48.7	49.7
	7.8	6.5	5.6	12.2
σ - Marillan (DCT) (/IsDa)		19.6/	19.4/	20.1/
Mullen (PSI)/(kPa)	22.5/		134	138
	155	135		1
σ	2.2/	1.8/	1.8/	1.6/
_	15.4	12.5	12.1	10.7
MD Tear (gm)	57.8	59.6	57.7	49.8
σ	4.2	3.8	5.3	6.2
CD Tear (gm)	72.9	73.8	69.2	62.0
σ	3.3	5.2	3.7	3.0
MD MIT fold (number)	254	184	201	186
σ	83	76	58	62
CD MIT fold (number)	40.8	29.1	26.7	25.2
σ	11.3	11.5	13.0	9.51

a Significantly different from recovered filler sample at 99.5% confidence as indicated by a t test.

Table 4. COATED OFFSET SHEET OPTICAL PROPERTIES Summary

	1 = = -			
Out to the second	5%	8%	Recovered	
Optical properties	Control	Control	filler	CPI
Brightness (% elrepho)	72.2	73.3	69.3 a	73.7
σ	0.21	0.13	0.22	0.16
Opacity (TAPPI)	94.3	94.6	96.4 a	94.7
σ	0.31	0.29	0.26	0.20
Gloss (75°)	49.2	48.5	47.5	50.6
σ .	5.3	2.2	3.2	2.7

indicates significantly different from control at 99.5 confidence level as indicated by a t test.

Table 5. COATED OFFSET SHEET LABORATORY PRINTING TESTS

Laboratory printing	5%	88	Recovered	
tests	Control	Control	filler	CPI
IGT Pick (#6 ink)	92	88	88	94
σ	12.0	8.1	5.1	5.5
K & N ink (% reduction)	19.6	20.2	20.4	22.4
σ	1.3	1.5	1.9	3.2
Hiding power	0.98	0.98	0.99	0.97
σ	0.015	0.005	0.004	0.008
Fiber rise W/F B = blisters (0 = poorest, 4 = best)				
285°	1-0	1-0	1-0	1-0
255°	1-0	1-0	2-0	2-1
230°	3-1	3-1	3-1	3-1

mill could, under present marketing conditions, tolerate this degree of brightness loss.

The laboratory printing tests for the coated sheet are given in Table 5, and there appears to be no significant difference in the laboratory printing properties. The printing trial conducted at the Rochester Institute of Technology upon rolls of coated paper composited from those produced under the various manufacturing conditions allowed determination under uniform printing conditions of whether the type of paper affected print quality. Four signatures selected at random for each condition were submitted to a printing specialist for his independent evaluation. He was asked to grade each signature on a scale of 1 to 5 for 7 printing properties. The scale was: 1 - poor, 2 - fair, 3 - average, 4 - good, 5 - excellent. The printing properties he was asked to judge were gloss, ink holdout, strikethrough, printing smoothness, pick resistance, fiber rise, ink spread and showthrough. The specialist was not aware of the origin of the samples or purpose of the evaluation until after the evaluation was completed. His detailed comments are given in Appendix B. The results of his independent evaluations are tabulated in Table 6.

All three test conditions from the pilot paper machine trial were rated as essentially identical. Thus, it appears that the recovered filler does not affect printability significantly. The mill-manufactured paper was graded lower on the properties of ink holdout, printing smoothness and showthrough but superior in its lack of fiber puffing.

Printing experts at the cooperating mill examined the printing quality and reached the similar conclusion that there was little difference among the three conditions from the pilot trial at WMU. However, a difference between these and the commercially produced paper was cited. This difference was mostly due to the color difference between the papers: the commercial paper was a more blue-white; whereas, the pilot plant paper had a cream color.

V.

Table 6. COATED OFFSET SHEET PRINTING RESULTS

		(Gloss)		Printing	Pick	Fiber	Ink			
_		Ink	Strike-	(smoothness)		Rise	spread	Show-	ĺ	Aver-
S	ample	holdout	through	mottle	tance	(puffing)	(sharpness)	through	Total	age
Recov- ered Filler	8R	4	4	4	4	4	4	4	28	27.5
	8R	4	4	4	4	4	4	4	28	
	8R	4	4	4	4	4	4	4	28	
	8R	2	4	4	4	4	4	4	26	
8% Control	8V	4	4	4	4	4	4	4	28	27.75
	8V	4	4	4	4	4	4	4	28	
	8V	3	4	4	4	4	4	4	27	
	87	4	4	4	4	4	4	4	28	
5% Control	5V	4	4	4	4	4	4	4	28	27.75
	57	4	4	4	4	4	4	4	28	
	5V	4	4	4	4	4	3	4	27 ~	
	5V	4	44	4	4	4	4	4	28	
Mill Manufac- tured Paper	CM	3	4	3	4	3	3	3	23	24.75
	CM	3	4	3	4	5	4	3	26	
	CM	3	4	3	4	5	4	3	26	
	CW-P	2	4	3	4	5	2	3	24	

1. Poor 2. Fair 3. Average 4. Good 5. Excellent

SECTION VI

EVALUATION OF SCREENING AS A RECOVERY ALTERNATIVE FOR A MILL MANUFACTURING UNCOATED FINE PAPER

EXPERIMENTAL DESIGN

A mill selected from its grade structure two grades which were high volume items and would be likely candidates for plans to utilize the recovered filler. It was felt that a heavy grade, which had a basis weight of 50 lb/3300 ft² (74 gm/m²), would probably be able to utilize the recovered filler with little In addition a 30 lb/3300 ft² (44 gm/m²) grade was difficulty. chosen as a more critical test of the recovered filler in recognition of long range industry trend toward lighter weight fine papers. It was intended that the recovered filler be tested as a partial replacement for virgin filler clay. The mill had such a diverse grade structure, some products of which the recovered filler would not be suitable for, that no single virgin fillerrecovered filler blend condition emerged as likely to be principally utilized. However, practical experimental conditions suggested that recovered filler clay be added directly to the beater during the model machine run and that virgin clay be continuously metered in at a rate necessary to maintain the desired The screening was conducted at the mill site and the ash level. recovered filler shipped to WMU where the two grades were simulated on the pilot paper machine. The grades were simulated using virgin filler exclusively and utilizing recovered filler in combination with virgin clay. Properties of the paper produced were evaluated by NCASI personnel. The rolls produced were evaluated by NCASI personnel. The rolls produced were subsequently rewound at the cooperating mill before shipment to Rochester Institute of Technology for printing evaluation on a four-color commercial web offset press.

SCREENING PROCEDURE

The screening was accomplished on site at the cooperating mill utilizing an unused 30-in. (0.76 m) Sweco vibrating screen. Primary sludge at 10 to 12 percent consistency was hauled from the site of the primary clarifier in half-full 55-gallon (210 l) drums, diluted at the screen installation to 5 to 6 percent

using a drum pump and mixed manually. The screen was equipped with a 230-mesh stainless steel screen and fed at a rate of 3 to 5 gpm (11.4 to 18.9 ℓ/\min). At the rate of 5 gpm (18.9 ℓ/\min), the screen became flooded and one occasionally had to stop to allow the screen to clear itself.

The solids content of the material passing the screen, as recovered filler, was 3 to 3.5 percent solids and amounted to approximately 50 percent of the total sludge fed the screen. Considering only the ash fraction as being the component of interest, 65 to 67 percent of the total ash or filler was recovered. The material passing through the screen was 88 percent filler and only 21 percent organic matter, as calculated from an ash measurement, compensating for 15 percent ignition loss. This material passing through the screen was predominantly filler.

The screening was conducted over a 1-week period by both mill and NCASI personnel. The recovered filler was allowed to settle, decanted, consolidated to a minimum number of drums and shipped by truck to WMU. At the conclusion of each day's screening and again prior to shipment to WMU, the drums of recovered filler were treated with 500 ml of formaldehyde to prevent bacterial decomposition. Upon arrival at WMU, the drums were all adjusted to 6 percent consistency by addition of city water and treated with an additional 500 ml of formaldehyde per drum.

PILOT PAPER MACHINE TRIAL

The two mill grades simulated during the WMU pilot paper machine trial were a 30 lb/3300 ft 2 (44 gm/m 2) Bible-type paper and a 50-lb (74 gm/m 2) offset sheet. Two days of machine time were devoted to each grade. The first day the grade was produced using virgin filler clay; the second day screened, recovered filler was substituted for a portion of virgin material.

The furnish for the 30-1b (44 gm/m²) sheet consisted of 40 percent Canadian bleached softwood kraft, 20 percent bleached hardwood kraft, 20 percent semi-bleached softwood kraft and 20 percent broke, which was supplied by the cooperating mill. The pulp was refined in the beater to 250 to 300 Canadian Standard Freeness. Added to the beater after refining, expressed as percent of 0.D. pulp weight, were 5 percent titanium dioxide, 12 percent filler clay and 0.8 percent alum. In addition, dyes provided by the cooperating mill were added to tint the paper a cream color. The pH was controlled on the machine to 4.8 to 5.0 with sulfuric acid. The sheet was sized by continuous addition of Neuphor emulsified rosin size at a rate equal to 2 lb/ton of paper (1 Kg/metric ton). The size was added to a mixing chamber just prior to the headbox. The dandy roll was used and

three nips were used in the calender stack. The sheet was also externally starch-sized in the size press. The starch, Clinton 753B, had been previously batch cooked at 10 percent for use in the size press. The size press temperature was held at 140° to 150°F (60 to 66°C), and the pickup rate was about 65 lb/ton of product (32 Kg/metric ton).

The initial headbox temperature selected was 110°F (43°C), and a retention aid (Dow CP7) was fed at a rate of 1/2 lb/ton (0.25 Kg/metric ton). These two parameters had to be modified during the first hour of running the machine due to bad sheet formation. The sheet was overflocculated and blotchy, so the retention aid was stopped and the temperature dropped to approximately 70°F (21°C). This solved the formation problem. Additional filler clay, at 15 percent solids consistency, was metered into the system to maintain the ash content in the sheet at a desired 15 percent level. An additional 9 percent clay was metered in this manner. In the absence of a retention aid, the achievement of such a level in a lightweight grade sheet with the prescribed furnish on the WMU pilot machine required a 9 percent trim clay addition.

During the second day of running, recovered filler material was substituted for the virgin clay added to the beater. However, use of virgin filler was continued for the supplemental trim clay. The magnitude of trim clay addition was such that filler materials constituted 57 percent of the clay or 46 percent of the total filler contained in the 30-lb (44 gm/m²) sheet.

The furnish for the 50-lb (74 gm/m²) sheet consisted of 40 percent southern bleached softwood kraft, 27 percent southern bleached hardwood kraft and 33 percent broke supplied by the mill. The pulp was refined in a beater to 250 to 300 Canadian Standard Freeness. Three percent titanium dioxide, 14 percent filler clay, 0.8 percent alum, expressed as weight of 0.D. pulp, were added after refining; dye also was added and mixed in to give the sheet a bluish-white cast.

The pH of the furnish to the machine was controlled continuously with sulfuric acid to 4.6. The sheet was internally sized with Neuphor emulsified rosin size at a level of 2 lb/ton (1 Kg/metric ton), and a retention aid (Dow CP7) was added to the mix tank prior to the headbox at a level of 2 lb/ton (1 Kg/metric ton). Three nips were taken in the calender stack, and the dandy roll was utilized. The sheet was externally sized with starch through use of the size press, which was operated under conditions similar to those associated with the lightweight sheet. The starch used was the same as for the 30-lb (44 gm/m²) grade and was prepared in a similar manner. The pickup rate was approximately 50 lb/ton of product (25 Kg/metric ton). Final sheet moisture was controlled to 4 percent.

During the first day's run of the 50-lb (74 gm/m²) sheet no trim clay was added, but during the second day's run, the ash level of the sheet was maintained by adding a 15 percent slurry of filler clay to the mix chamber prior to the headbox. This addition represented 2.35 percent of the filler added with the final sheet ash controlled to 15 percent ± 1. During the second day's run on the 50-lb (74 gm/m²) sheet, the filler added in the beater was replaced by screened, recovered filler. Thus, during the run containing the recovered filler, 83 percent of the clay was recovered filler and 68 percent of the total filler in the sheet was recovered filler. A daily sampling routine of collecting four sets of paper and white water samples per day was followed similar to that described in the coated fine paper model machine run.

The rolls of paper from the four 1-day machine trials were wrapped and shipped to the cooperating mill, where they were rewound into four composite rolls, two of which were of the lightweight grade and two of the heavy. Each composite roll was half made with virgin filler and half with recovered filler. Rewinding problems were encountered with the lightweight paper which consisted of a series of breaks due to pin holes and dirt in the paper. However, these problems were observed in both sheets made with and without recovered fillers and as a result, could not be attributed to the presence of reclaimed material. The composite rolls were then shipped to Rochester Institute of Technology for printing.

LABORATORY PHYSICAL, OPTICAL AND PRINTING TESTS

The procedures used are in common practice in the paper industry and are described in detail in the section on testing procedures for the coated fine paper evaluation, Section IV of this report.

PRINTING TRIAL

The printing was accomplished using a GOSS Commercial 38-in., four-color perfecting press equipped with a brush-dampening system. The four colors used were yellow, magenta, cyan and black, and they were applied in that order. The printing speed was controlled at 520 to 540 fpm (158 to 165 m/min) and the web temperature leaving the dryers varied from 220°F to 290°F (104 to 143°C). The hot air temperature varied from 400°F to 490°F (204 to 254°C).

The press was made ready using a roll of 50 lb/3300 ft² (74 gm/m²) mill-manufactured paper. When the press exhibited satisfactory printing conditions, it was run for an additional 3000 impressions before a composite roll, consisting of equal proportions of the 50-lb (74 gm/m²) control sheet and the 50-lb

(74 gm/m²) recovered filler sheet was spliced in and printed. Two breaks occurred during the run of the roll of mill-manufactured paper and one each during the sections of composite roll containing the control and recovered filler.

A roll of 35 lb/3300 ft² (52 gm/m²) mill-manufactured paper was run next as a made-ready roll for printing of a composite roll of the 30-lb (44 gm/m²) paper manufactured on the pilot paper machine. The cooperating mill did not have an extra roll of the 30-lb (44 gm/m²) grade which simulated in the pilot trial so the 35-lb (52 gm/m²) roll was substituted. The 35-lb (52 gm/m²), mill-made paper left a white deposit on the yellow blanket, and two breaks were experienced during the printing of this roll. Three breaks occurred in the subsequent printing of the 30-lb (44 gm/m²) composite roll, two during the course of running the section containing only the virgin filler and one during the segment containing recovered filler. The second composite roll of 30-lb (44 gm/m²) paper was run next, and only one break, during the portion containing the recovered filler, was encountered.

The final roll printed was the second 50-1b (74 gm/m²) basis weight composite roll, and it ran without any breaks.

The frequency of breaks was undoubtedly detrimental to quality of the printed sheets. In the course of the trial, the occurrence of breaks diminished as the press crew gained experience in the printing of the grades of paper under study. Furthermore, to prevent breaks, sheet tension had been progressively decreased over duration of the trial.

Each roll or segment of a roll was printed with greater than 3500 impressions. Plate wear was observed at the end of the printing trial after only 60,000 impressions but it is not possible to say whether this was due to the paper or the stopstart nature of the press operation.

After the press had reached steady state, the different papers printed were sampled every 500th impression by taking 10 signatures. These signatures were packaged and shipped to Western Michigan University for evaluation. From these signatures, five samples from each condition were selected at random and submitted to an impartial printing expert at Western Michigan University for evaluation based upon seven printing properties. These included ink holdout, strikethrough, showthrough, printing smoothness, ink spread, printing sharpness and pick resistance. The samples were judged on a scale of 1 - poor, 2 - fair, 3 - average, 4 - good, 5 - excellent.

In addition to the samples from each manufacturing condition, a set of sequential samples were submitted for evaluation of whether plate wear significantly affected printing sharpness as the run progressed.

SECTION VII

DISCUSSION OF RESULTS

The cooperating mill in this segment of the study was one whose sludge had been evaluated in the initial laboratory test Table 7 is a comparison of the screening results obtained under laboratory and field conditions. The purity of the screened product decreased only slightly, while the percentage of the filler which was recovered decreased significantly. In the laboratory study it was found at a constant solids loading rate to the screen that variation in sludge consistency over a range from 0.1 to 3 percent did not affect the recovery efficiency. However, it is conceivable that such a trend would not extend to the 6 percent consistency utilized in the field evaluation. Going further, a comparison of respective mass loading rates, expressed as pounds of dry solids/minute per ft2, indicates that the screen under field conditions was loaded at least an order of magnitude more heavily than under laboratory condi-This likely contributed to the lower filler recovery efficiency and corresponds with the field observation that the screen at times appeared overloaded and took on a flooded appearance.

The high feed rate to the screen was chosen under field conditions in order to complete the screening operation in sufficient time to permit the previously scheduled installation of new equipment by the host mill. The product recovered, however, provides a more critical test of the recovered filler's potential usefulness since it is less pure than the material recovered under laboratory conditions.

In the 50-1b (74 gm/m²) sheet, reclaimed filler materials constituted 87 percent of the filler clay, which corresponds to 68 percent of the total filler in the sheet. In the 30-1b (44 gm/m²) sheet, 57 percent of the filler clay was replaced with recovered filler, representing 46 percent of the total filler. The lower percentage of total filler replaced is due to the addition of titanium dioxide to each sheet. Its level of addition was not altered by substitution of filler grade clays with recovered filler. The 30-1b (44 gm/m²) sheet contained a higher percentage of titanium dioxide and exhibited less brightness loss.

Table 7. COMPARISON OF LABORATORY SCREENING VS MILL SITE SCREENING OF CONVENTIONAL HIGH ASH SLUDGE

Parameter	Laboratory data	Mill site data
- 42 4110 002	1	
Original sludge ash content (%)	-	55
Consistency of feed (%)	0.1-3.0	5-6
Total solids recovered (%)	-	50
Filler recovered (%)	98	65-67
Purity of product (% of recovered material which is filler)	92	88
Feed rate to screen (gpm)/(1/m)	0.2 to 4.8/ 0.6 to 18.0	3 to 5/ 11 to 19
Screen diameter (in.)/(m)	18/0.46	30/0.76
Screen mesh	230,325	230
Solids loading rate to screen (1b solids/min ft2)/Kg/min m2)	0.023/0.112	0.25-0.51/ 1.22-2.49
Abrasiveness (mg)	-	4.2
Brightness (% elrepho)	_	66.5

The first pass and overall retention values experienced during the pilot machine runs are summarized in Table 8. It would appear from the data that the retention conditions were not significantly influenced by the substitution of recovered filler clay for virgin filler clay in either of the grades simulated.

Table 8. UNCOATED OFFSET MODEL MACHINE TRIAL RETENTION VALUES

Sheet produced	First pass,	Overall,
30-lb (44 gm/m^2) control sheet	29.5	67.0
30-lb (44 gm/m ²) recovered filler sheet	28.3	65.0
50-lb (74 gm/m^2) control sheet	51.0	76.5
50-lb (74 gm/m ²) recovered filler sheet	54.0	77.0

The physical properties of the sheets made during the pilot machine run are listed in Table 9 along with the samples of mill-manufactured paper which were utilized during the printing trial. In comparing the control sheet to that containing recovered filler, the figures boxed in the recovered filler column are statistically significantly different from the control. However, none of the differences are of sufficient magnitude to be of practical concern. Thus, the use of recovered filler does not seem to have an adverse effect on the physical properties. Special note should be taken that the dirt speck level of the sheets containing the recovered filler did not significantly increase.

The optical properties of the sheets are summarized in Table 10, and it appears that the brightness is adversely affected by the addition of the recovered filler. The 30-lb (44 gm/m²) sheet lost 0.8 points and the 50-lb (74 gm/m²) sheet lost 4.6 points. The 50-lb (74 gm/m²) sheet with the recovered filler gained 1.2 points in opacity. It is doubtful whether a mill could tolerate 4.5 points loss in brightness. As a consequence, if the recovered filler is to be utilized, it would have to be used in a smaller percentage or would have to be bleached prior to use. Possible techniques for bleaching are discussed later in this report.

The laboratory printing test results are summarized in Table 11. No differences were noted between the control samples and samples containing the recovered filler. In general, the

Table 9. UNCOATED OFFSET SHEET PHYSICAL PROPERTIES

			Paper s	ample		
•		30-lb 2	35-Ib	<u> </u>	50-1b	50-1b
Test	30-1b	30-16 (44 gm/m ²)	(52 gm/m^2)	50-1b	(74 gm/m^2)	(74 gm/m^2)
	(44 gm/m ²)	Recovered	Mill	(74 gm/m^2)	Recovered	Mill
	Control	filler	manuf.	Control	filler	manuf.
Basis wt $(\frac{1}{2}b/3300 \text{ ft}^2)/$	20.07	29.7 ^a /	25 67	50.67	50.27	50 0/
(m /=2)	30.8/	[<u>29.7</u>] /	35.6/	50.6/	50.3/	58.8/
(gm/m²)	45.6	44.0	52.7	74.9	74.4	87.0
σ	0.6/	0.6/	0.3/	0.3/	0.3/	0.21/
· · · · · · · · · · · · · · · · · ·	0.9	0.9	0.4	0.4	0.4	0.3
Caliper (.001 in.)/(mm)	2.46/	2.35a/	3.19/	3.80/	3.84/	3.88/
	0.062	0.060	0.081	0.097	0.098	0.099
σ	0.04/	0.04/	0.07/	0.05/	0.05/	-
	0.001	0.001	0.002	0.001	0.001	
Porosity (SHeffield)	161	147	84.5	122	100 a	94.8
σ	16	13	11.5	13	7	10
Smoothness (Sheffield)	167	163	168	196	193	220
σ	11	8.5	7.7	11	12	19
MD Tear (gm)	26.6	24.4	37.1	67.4	66.0	47.4
σ	3.0	2.0	2.1	3.3	3.0	1.8
CD Tear (gm)	30.4	31.0	52.6	81.6	74.4	54.4
σ	4.1	2.0	4.1	5.5	3.6	1.6
Mullen (PSI)/(kPa)	11.5/	10.7/	13.7/	16.4/	16.3/	19.4/
	79.2	73.7	94.4	113.0	112.3	133.7
σ	1.5/	1.4/	1.3/	1.3/	1.5/	3.3/
	10.3	9.6	9.0	9.0	10.3	22.7
MD Fold (%)	8.1	7.7	26.7	17.4	14.0	7.6
σ	3.0	3.5	8.9	4.5	3.6	1.8
CD Fold (%)	5.5	4.4	10.8	8.2	8.3	5.3
σ	1.5	0.9	3.7	2.3	1.9	1.5

Statistically different from the control at 99.5% confidence level as indicated by a t test.

Table 9. UNCOATED OFFSET SHEET PHYSICAL PROPERTIES (Continued)

	Paper sample						
		30-1b 2	35-1b ₂		50-lb ₂	50-1b ₂	
Test	30-1b 3	(44 gm/m^2)	35-1b (52 gm/m ²)	50-1b 2	50-1b (74 gm/m ²)	(74 gm/m ²	
	30-1b (44 gm/m ²)	Recovered		(74 gm/m^2)	Recovered	Mill	
	Control	filler	manuf.	Control	filler	manuf.	
Sizing (Hercules-sec)	8.9	10.6	157	95.2	83.7	148	
σ (πουσαμού 200)	0.5	3.0	58	45	25	36	
Dirt Specks	5.94	16.1	25.6	11.8	12.3	23.7	
σ	1.9	7.9	5.8	4.2	4.3	8.3	
MD Tensile (Kg)	6.2	6.3	8.6	8.2	8.5	12.9	
σ	0.6	0.9	0.6	0.9	0.8	1.1	
CD Tensile (Kg)	3.17	3.16	4.3	4.4	4.2	4.72	
σ	0.4	0.3	0.3	0.7	0.5	0.2	
MD TEA (Kg(m)/m ²)	1.89	2.38	1.9	6.3	6.8	3.5	
σ.	0.4	0.6	0.4	1.2	1.8	0.5	
CD TEA (Kg(m)/m ²)	4.32	4.32	2.3	3.7	4.5	3.09	
σ	0.8	0.7	0.3	0.7	1.0	0.6	
MD Stretch (%)	1.5	1.5	1.0	1.8	1.9	0.9	
σ	0.2	$\frac{0.2}{3.4}$ a	0.1	0.2	0.2	0.1	
CD Stretch (%)	4.3	3.4	1.9	4.6	4.9	1.9	
σ	0.5	0.6	0.2	0.8	0.8	0.3	
Ash (%)	15.3	15.5	14.6	16.3	16.5	16.4	

Statistically different from the control at 99.5% confidence level as indicated by a t test.

31

σ

σ

Opacity (TAPPI)

30-1b (44 gm/m^2) 35-1b (52 gm/m²)50-1b (74 gm/m^2) 50-1b₂ $\begin{array}{c} 30-1b \\ (44 \text{ gm/m}^2) \end{array}$ (74 gm/m^2) 50-1b (74 gm/m^2) Recovered Optical Recovered Mill Mill Control properties filler manuf. Control filler manuf. 81.6 a 77.8 a Brightness (G.E.) 78.6 78.3 77.7 86.2 0.33 0.25 0.30 0.22 0.42 0.30

85.6

1.1

94.3 a

0.6

91.1

0.5

93.1

0.6

UNCOATED OFFSET SHEET OPTICAL PROPERTIES

81.4

1.3

Table 10.

81.0

1.0

а Statistically different from control at 99.5 confidence level as indicated by a t test.

Table 11. UNCOATED OFFSET SHEET LABORATORY PRINTING PROPERTIES

	Paper sample							
Test	30-1b (44 gm/m ²)		35-1b (52 gm/m ²) Mill	50-1b (74 gm/m ²)		Mill		
	Control	filler	manuf.	Control	filler	manuf.		
Wax pick: Wire side Felt side	14 16	14 16	10 20	14 14	14 14	16 11		
Hiding power	76.1	75.9	89.4	91.9	93.0	95.1		
σ	1.3	2.0	0.2	0.4	0.7	-		
IGT Pick (cm/sec)	129.8	129.3	115.0	100.9	106.5	129.0		
σ	20	15	7.9	11	8.5	14		
Velocity viscosity product (x10 ⁺⁴ gm/sec ²)	10.7	10.6	9.5	8.3	8.8	10.6		
σ	1.7	1.2	0.6	0.9	0.72	1.2		
K & N Ink (% reduction)	50.8	53.0	52.9	54.2	53.3	55.0		
σσ	1.6	1.7	2.0	1.0	0.9	2.1		

breaks previously cited hampered the printing with the consequence that overall printing quality achieved was not very good.

Though the printing expert was unaware of which samples were associated with which grades of paper, it was obviously easy to distinguish the heavy basis weight sheets. His detailed comments are given in Appendix C. Table 12 summarizes the results of his evaluation. Each trial condition had five signatures evaluated. For the 50-1b (74 gm/m²) grade, the sheets containing recovered filler were judged superior to the control on ink holdout, strikethrough, and printing sharpness. It compared very well to the mill-manufactured paper. For the 30-1b (44 gm/ m²) grade, the sheet containing the recovered filler rated poorer than the control on printing smoothness and sharpness but they differ by only 1 point in their overall rating. Both the control and the sheet containing the recovered filler rated below the mill-manufactured paper but this was probably due more to the basis weight difference between the 30- and 35-lb (44 and 52 gm/ m²) sheets than to any other factor.

Considering all of the printing results as a whole, it would appear that the recovered filler addition was not a prime factor in affecting printing properties. It was the opinion of the printing expert that varying press conditions were the prime factor, even though the trial design was set up to minimize this.

During the printing trial, plate wear was observed and to see if this significantly influenced the printing characteristics, a set of sequential samples arranged in the order in which the printing was done was submitted to the printing expert for evaluation. These samples consisted of an early signature and a late signature from the printing of each individual paper condition. From an examination of the results shown in Table 13, it is apparent that although the printing sharpness did vary throughout the run, there is not a trend to relate it to plate wear.

Table 12. EVALUATION OF WEB OFFSET PRINTED SAMPLES

Sample	Ink holdout	Strike- through	Showthrough	Printing smoothness (mottle	Ink spread	Printing sharpness	Pick resistance	Total average
50-1b (74 gm/m ²) Mill manuf.	4 4 4 3 4 3.8	4 4 4 3 4 3.8	2 2 3 2 2 2	4 4 4 4 4 4 4 4	4 4 4 4 5 4.2	Lt. ink 4	5 5 5 5 5 5	27.0
35-1b	4 4 4 4 3 3.8	4 4 4 4 3 3.8	4 4 4 4	4 4	4 3-4 4	4 4 4 2-3 4 3.8	5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	28.2
50-1b (74 gm/m ²) Control	3 3 3 3 3	3 3 3 3 3	3 3 2 3 2.8	4 4 4 4 4	4 4 3 4 3 4 2-3 3.4	2 2 2 2-3 3 2.4	5 5 5 5 5	23.6
35-1b (52 gm/m ²) Mill manuf.	3 3 3 3 3 3 3 2 2	3 2 2 3 2.6	3 3 3 3 3 2 3 2 8 2 1 2 3 2 0	4 4 4 4 4 4 3 4 3 2 3.2	4 4 3 3.6	2 2 2 3 2 2.2	5 5 5 5 5 5 5 5	21.2
30~lb 44 gm/m ²) ecovered filler	1 1 1 1 1 1	3 3 3 3 3 3 3 2 2 3 2 6 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	1 1 1	3 3 4 3.2	4 4 3 3 3 4 3 4 3 4 3	4 4 4 2-3 4 3.8 2 2 2-3 3 2.4 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2		16.6
30-lb (44 gm/m ²) (Control R	1 1 1 1	1 1 1 1 1	1 1 1 1 1	4 3 4 4 3.6	4 4 3 2 3.4	3 3 2 2 2 2.6	5 5 5 5 5 5 5 5	17.6

Grading Scale: 1 - poor, 2 - fair, 3 - average, 4 - good 5 - excellent

Table 13. PRINTING SHARPNESS OF SEQUENTIAL SAMPLES

				
Sample	High-	Middle-	Shadow	Average
Number	lights	tones	areas	
Al	3	1	1	2
A2	4	4	4	4
Bl	4	3	4 3	3-4
B2	4 3	2	2	2-3
Cl	1 2	2		1
C2	2	2	1 3	2-3
Dl	1	1	1	1
D2	2	1	1	1
E1	2 2	3	2	2
E2	3	3	2	2
F1	1	2 2 1 3 3	2 1	1
F2	2	3	2	2
Gl	1	1	1,	1
G2		1	2	1
Hl	1 2 2 2 2	1	1	2
H2	2	1	1	1-2
11	2	1	2	2
12	1	2	2	2
J1	4	ī	ī	1
J2	1 2 3	3	2	2-3
K1	3	2	2	2
K2	3	3	2	3

Grading scale: 1 - poor, 2 - fair, 3 - average, 4 - good, 5 - excellent

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SECTION VIII

BLEACHING OF SCREENED, RECOVERED FILLER

The chief drawback to the use of the screened, recovered filler appears to be its low brightness. This phenomena was observed in all three grades of paper manufactured incorporating the screened, recovered filler. There are a variety of bleaching techniques which might be of value in restoring the brightness of the recovered filler. Sodium hydrosulfite is commonly used in the clay industry to bleach its clays. As a result, it was evaluated over a variety of conditions on the screened sludge used in the manufacture of the uncoated offset sheets. The temperature was varied from 150 to 200°F (66 to 93°C). The concentration of Na₂S₂O₄ was varied from 5 to 20 percent, and the EDTA level from 0.5 to 2 percent. The best condition appeared to be 1 percent EDTA and 15 percent sodium hydrosulfite for 1 hour at 180°F (82°C), but this resulted in only a 3-point gain in bright-This 3-point gain would only increase the brightness from 70 to 73 and probably would not be enough to overcome the degradation of final sheet brightness.

A variety of other bleaching techniques common to the paper industry were also evaluated under severe bleaching conditions on two high ash sludges from conventional mills which were not deinking. These techniques included borohydride, chlorination, hypochlorite, chlorine-water system at pH 5, chlorine dioxide and peroxide. The bleaching conditions are summarized in Table 14, and the results given in Table 15. It appears that all of the oxidative techniques have the ability to brighten the sludge, with peroxide showing the greatest capacity. Thus, bleaching may provide an alternative to the brightness loss difficulty. Economic comparison of the various options warrants further evaluation.

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Table. 14. CONDITIONS FOR BLEACHING

		Types of Bleaching						
Experimental parameters	Boro- hydride	Chlori- nation	Hypo- chlorite		Chlorine- H ₂ O pH 5	Peroxide		
Consistency	2% SS	2% SS	2% SS	2% SS	2% SS	2% SS		
рн	10	1-2	11-12	3.5-6.0	5	10-11		
Time	100 min	1 hr	2 hrs	5 hrs	1 hr	l hr		
Temperature	room	room	room	160°F(71°C)	room	160°F(71°C)		
% Chemical	2	6.59% avCl	10% avCl	1.2	10% avCl	1.5% of 100% chem- ical		
Additional chem- ical						5% Na ₂ SiO ₃ 1% Mg SO ₄		
Post treatment		Thiosul- fate	Thiosul- fate	Thiosul- fate	Thiosul- fate	SO ₂ wash		

Table 15. BLEACHING RESULTS

	,	
Туре	Brightness,	% Elrepho
of	A	В
bleaching	Sludge	Sludge
No bleaching	70.5	50.0
Borohydride	70.0	52.0
Chlorination	74.2	67.4
Hypochlorite	75.9	56.2
Chlorine - H ₂ O pH 5	77.5	64.4
Chlorine Doxide	76.8	55.6
Peroxide	77.3	70.9

Note: 1. Sludge A originated at the same mill from which sludges used in the uncoated fine paper evaluation were taken.

2. Sludge B originated at a small fine paper/specialty mill. Originally low brightness of the sludge is attributable to excessive clarifier residence time.

SECTION IX

PLAUSIBLE SCREENING SYSTEM TO RECOVER FILLER

If a mill were to employ screening to recover filler from their high ash sludge as a sludge volume reduction technique, the flow schematic might be the one shown in Figure 2. The primary clarifier underflow is fed through a consistency regulator to a screening device. Sludge components which pass the screen are sent to a mix tank with sufficient residence time to dampen recovered filler quality variations. The mix tank might also function as a tank in which to do any bleaching, if necessary. From the mix tank the material would flow to the mill for addition at the usual point where filler is added in the system, such as a blend chest.

There are many facets in designing a filler recovery system to which the previously discussed trials and data do not speak. However, if one were to make recommendations from the data contained in this report, as far as the consistency fed the screen, about 3 percent would be a good figure on which to control.

As far as screen types are concerned, the Sweco vibrating screens which were used in these studies worked well, but there are other alternatives commercially available which might be considered. A trial was attempted using the Sweco concentrator rather than their normal flat vibrating screen and it gave very poor recovery efficiencies when tested. The screen retained most of the filler as well as the fiber.

The mesh of the screen to be employed depends on how much contamination the grades run can tolerate. A 320-mesh screen gives a very pure product. Employing a more open mesh would result in more contamination but a greater throughput rate and associated reduction in required screening surface area. Life of the screen is a factor of consideration as well.

Explicit size and design criteria for the equalization chamber is elusive. Equalization required would be dependent upon the mill's grade structure and frequency of grade changes. The more changes and wider the variety, the longer will have to be the equalization chamber to dampen these out.

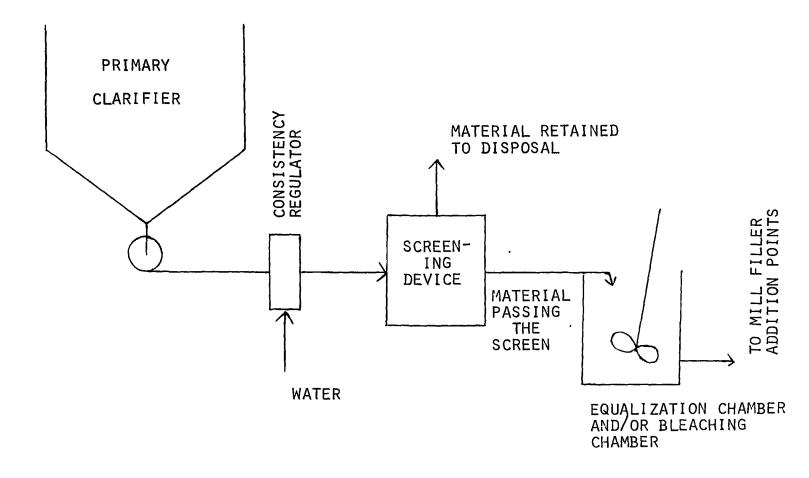


Figure 2. Diagram of possible scheme for mill installation.

For a small specialties mill, the screening of the primary clarifier sludge might not be the route to follow. There it would likely be preferable to go back into the mill, catch the losses close to the point of origin, and reuse them immediately. For example, one source of filler loss to the sewer is from the cleaner rejects. One specialty mill is currently using 325-mesh Sweco vibrating screen to recover the filler from this source by taking the material passing through the screen and sending it back to the headbox fan pump (8). No adverse effect on product quality has been observed. However, it is important not to send the screen accepts back to the primary cleaner intake. Otherwise, the cleaner system will be overloaded with filler. This scheme has a particularly good return on investment where TiO2 is being used as filler. It is a good illustration of isolating the loss at the source point and immediately reusing it.

MILL EXPERIENCES INCORPORATING PRIMARY CLARIFIER HIGH ASH SLUDGE INTO A BLEACHING SEQUENCE

Case I (9)

A fine paper mill producing 485 tpd (440 metric tpd) of a mix of bond, offset, book publishing and reprographic papers, when confronted with the 1974 pulp shortage, decided to reactivate a portion of a dormant three-stage bleach plant to process 100 tpd (91 metric tpd) of semi-bleached hardwood pulp which had been manufactured at another location. The pulp would be bleached in a hypochlorite stage to achieve an 84 to 86 brightness. At this same time it was decided to reclaim the 10 tpd (9.1 metric tpd) of primary clarifier sludge which the mill produced by blending it with the semi-bleached pulp prior to the thickener preceding the hypochlorite stage. The bleaching conditions used in the hypochlorite stage were 10 percent stock consistency, 2-hour detention time, calcium hypochlorite to give 3 percent available chlorine, 0.5 percent sulfamic acid, 120°F (49°C) and pH 7 to 8. These bleaching conditions worked fine on the pulp alone, but on the sludge pulp combination it was found to give a brightness of less than 80 G.E. Upon investigation in the laboratory it was found that the primary clarifier sludge by itself could not be bleached above 55 G.E. brightness. A strict blending of 10 tons (9.1 metric tons) of 55 brightness sludge with 100 tpd (91 metric tpd) of 85 brightness pulp should give an 82 brightness product which would have been acceptable, but this additive relationship did not hold, and it was found experimentally that only 5 tpd (4.5 metric tpd) of sludge could be utilized if the brightness level were to be maintained.

A second difficulty with the sludge was that the ash fraction was retained poorly on the thickener so a fair portion of it returned via the thickener filtrate overflow to the clarifier, thus producing a dead load in the loop between the thickener

and the clarifier. A third difficulty which the sludge caused was plugging of the centricleaners which followed the hypochlorite stage. After the hypochlorite stage, the stock is diluted to 1 percent, sent through three stages of centricleaners, pumped to a vacuum washer and then pumped to the paper mill. The rejects from the third stage centricleaners are sent over a Bauer Hydrasieve and trucked to a land fill. This acts as the purge of material from the system. The filtrate from the hydrasieve was sent back to the clarifier. The mill lived with the above difficulties and utilized about half of its primary clarifier sludge from July through November of 1974. At this time the operations were suspended because the extra bleached pulp capacity was not needed.

After this experience an economic analysis indicated that it would be advantageous to continue recovery of the clarifier sludge even if fully bleached pulp or broke were used as a carrier instead of semibleached pulp. It must be kept in mind that there were no capital investment costs, only operating costs of utilizing existing equipment which was available in the inactive bleach plant.

Before pursuing this, however, efforts were made to trace down and alleviate some of the problem areas. The primary clarifier was fed by total mill effluent, storm sewers, roof drains, septic tank leachings, boiler house sewers, and backwash from the water treatment plant sand filters. By daily monitoring of laboratory bleached brightness of the clarifier sludge, it was observed that when storm runoff was present, poor brightness of the bleached sludge resulted. When the intake water was very turbid, a lot of filter backwash was in the primary clarifier sludge and this caused lower brightness. This amounted to 1 ton (0.9 metric ton) of silt in 10 tons (9.1 metric tons) of sludge where normally there would be 500 lbs (227 Kg) of filter backwash in 10 tons (9.1 metric tons) of sludge. The mill negotiated with the state for permission to place the filter backwash back in the water intake basin, and this feed stream was taken out of the primary clarifier. This resulted in an average increase of 10 G.E. points in sludge bleachability moving from an average of 55 to an average of 65.

The storm sewer runoff was still a problem, but it was determined that it would be a problem only 25 percent of the time and moving the coal piles and rerouting storm runoffs so they would not drain to the clarifier would bring this sufficiently under control.

Laboratory studies showed that addition of a chelating agent before bleaching could increase the brightness an additional 3 points. In practice it would be too costly to do this on a full scale, but it was felt that perhaps iron in the boiler

blow-down water was the source of this and an alternate way to dispose of this stream is being sought.

To solve the loss of filler on the first washer it was decided to apply the sludge through a drilled pipe to the surface of the washer after the pulp mat was formed. This brought the solids loss problem under control.

To solve the centricleaner plugging problem it was thought that placing an 1/8-in. (0.32 cm) opening vibrating-type Johnson screen prior to the clarifier sludge surge tank and placing a pressure-type centriscreen prior to the centricleaners would alleviate this problem. These are possible future refinements of their existing system.

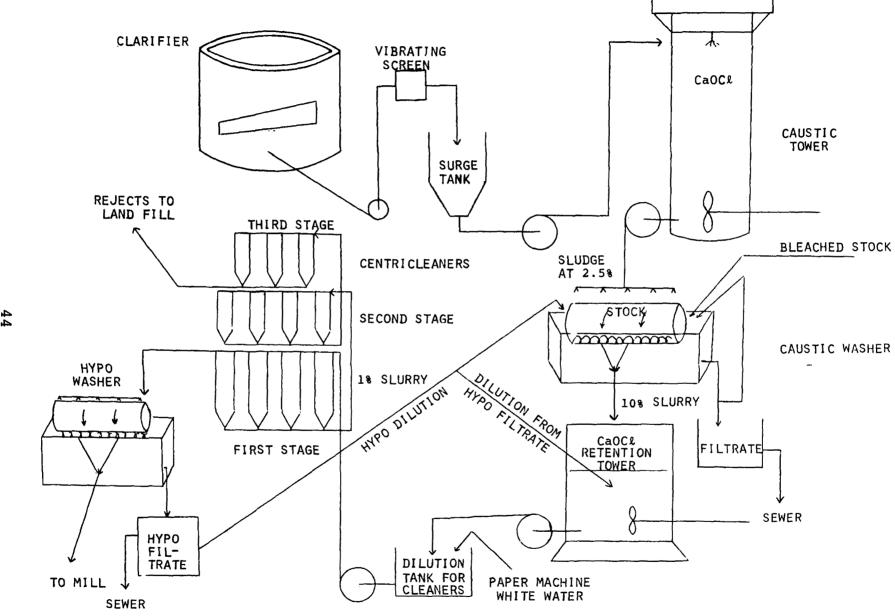
The sludge bleaching recovery system illustrated in Figure 3 was operated for most of the month of June. Forty tpd (36.4 metric tpd) of fully bleached pulp was fed to a thickener prior to the hypochlorite retention tower. This thickener had been the caustic washer of the inactive bleach plant. Ten tpd (9.1 metric tpd) of sludge were fed into the chlorination tower of the inactive bleach plant and treated with calcium hypochlorite to a level of 3 percent available chlorine. The chlorination tower was run as a short retention, completely mixed system at ambient temperature. The sludge was fed through showers to the face of the caustic washer and then into the hypochlorite tower with the pulp. Calcium hypochlorite, to give 0.5 percent available chlorine on the pulp, was added to the hypochlorite tower. The stock consistency in the tower was 10 percent; 4 hours detention time was utilized at a 120°F (49°C) temperature and a pH of 7 to 8.

The stock-sludge mixture was diluted with papermill white water and sent to the centricleaners. After the centricleaners, it was thickened on a vacuum washer and sent to the paper mill for utilization. The centricleaners still had plugging problems but the solids loss problem and the brightness problem were brought under control. The sludge processed in this fashion did not appear to cause any problems when utilized in the mill. Further modification of the system to include screening devices to solve the centricleaner plugging problem is anticipated.

This system or some alternate scheme will be in use in the near future at this mill to recover their primary clarifier sludge.

Case II (10)

A pulp and paper mill producing 500 tpd (455 metric tpd) of fine paper (book, bond, offset, ledger, duplicator, reprographic, etc.) and 650 tpd (590 metric tpd) of bleached soda pulp is reusing its primary clarifier sludge by sending it back



Case I primary clarifier sludge utilization bleaching system. Figure 3.

through the bleach plant. This mill has a separate sewer system for the pulp mill and bleach plant areas. The sewer stream, whose solids are reclaimed, consists of the papermill sewer, wet lap machine sewer, storm sewer, and flexographic printing operation sewer. These streams are fed through a 1/2-in. (1.3 cm) bar screen, to a 1/8-in. (0.32 cm) bar screen and then into a 60-mesh sidehill screen, which reclaims much of the pulp solids. The filtrate passing the screen is sent to two highly over-loaded settling primary clarifiers, whose overflow goes to the city sewage treatment plant. The clarifier underflow is combined with the solids recovered from the sidehill screen and sent to a chest where they are blended with mill colored broke. The combined clarifier underflow and sidehill screen accepts consist of approximately 50 percent ash which contains titanium dioxide, talc, clay, and hydrated silica. This blend is then shipped to the brown stock surge tank prior to the chlorination stage. approximate proportion of material processed through the CEHD bleach plant is 600 tpd (546 metric tpd) virgin pulp, 15 tpd (13.6 metric tpd) clarifier and sidehill screen sludge and 10 tpd (9.1 metric tpd) colored broke. The approximate bleaching conditions employed are given in Table 16. No overt change in the bleaching chemical demand was noticed when the clarifier sludge was introduced. The mill had been reclaiming the sidehill screen-captured material for approximately 20 years and has been utilizing the clarifier underflow in this manner for a year and a half.

The only problem which has been noted is an increase in the dirt count in the final paper product during periods of heavy rainfall. Most of the dirt is usually taken out in the Radiclone system in the bleach plant, but in heavy rainfall periods this purge is insufficient. The stock out of the bleach plant is used to manufacture all of the grades commonly produced by the mill. The only grade in which it caused difficulty was a perforator base paper grade which had a low ash requirement. Since ash was already present in the pulp coming from the bleach plant, virgin pulp had to be substituted.

Table 16. BLEACHING CONDITIONS FOR MILL RECLAIMING CLARIFIER SLUDGE BY PROCESSING THROUGH BLEACH PLANT

Conditions	С	E	Н	D
Detention time (min)	27	17.1	81.5	326
Consistency (%)	3	8	8	8
Temperature (°F)/(C°)	90/32	140-150/60-66	120/49	160/71
Нд	2.0-2.5	10.0-11.0	8.5-9.5	3.0-3.5
Chemical (wt % 0f O.D. pulp)	3% avg cl	1% ' NaOH	3% avg cl CaOCl	0.5% avg cl ClO ₂

635 tpd (580 metric tpd) through plant

SECTION X

EVALUATION OF FILLER RECOVERED BY WET OXIDATION

EXPERIMENTAL DESIGN

The overall experimental design involved the cooperating fine paper mill practicing deinking, picking from its grade structure a high volume 50 lb/3300 ft² (74 gm/m²) uncoated offset sheet which was to be simulated on the pilot paper machine at Western Michigan University. This grade was to be manufactured using virgin filler material and then made with the filler completely replaced with wet oxidized, recovered filler. The wet oxidized, recovered filler was obtained by the mill's having shipped drums of their dewatered clarifier sludge to a commercial wet oxidation equipment supplier (Zimpro, Inc.) for processing. The paper produced was tested for its physical and optical properties, rewound at the cooperating mill, and shipped to a nearby commercial printer for a printing evaluation.

WET OXIDATION PROCEDURE

Dewatered primary clarifier sludge was placed in 55-gallon (210 l) drums by the cooperating mill and shipped to Zimpro, Inc. in Rothschild, Wisconsin for wet oxidation. The material was reslurried and processed through their pilot plant at a temperature of 600°F (316°C) and a retention time of one hour. resulted in a pressure in the reaction chamber of approximately 3000 psi (21000 kPa). The solids fraction, after being wet oxidized, appeared very similar to virgin filler. The liquid fraction had a distinct yellowish tint to it. As a result, the solids fraction was washed by allowing it to settle, decanting the liquid and reslurrying in tap water. This process was repeated several times to ensure removal of the yellow supernatant. The material was concentrated to 15 percent solids and shipped to Western Michigan University in plastic-lined, 55-gallon (210 &) drums.

The wet oxidation condition of one hour at 600°F (316°C) is as severe an oxidation condition as the processor had ever observed. At the time of the trial, they had developed experience with high ash sludges from five different plants. To obtain a satisfactory high-brightness product the temperature

conditions generally ranged from 482°F to 600°F (250°C to 316°C). The conditions needed to achieve a satisfactory product is something which will have to be determined on each individual sludge. Again, the sludge utilized in this study was the most difficult case that had been encountered, requiring the most intensive of conditions to achieve high brightness.

PILOT PAPER MACHINE TRIALS

The pilot paper machine at Western Michigan University was utilized to make a 50 lb/3300 ft 2 (74 gm/m 2) sheet from a furnish consisting of 40 percent bleached softwood kraft and 60 percent bleached hardwood kraft refined to 300 Canadian Standard Freeness with a Claflin refiner.

The sheet was filled to a level of 15 percent ash using No. 2 coating clay on the first day of a 2-day trial, and wet oxidized, recovered filler on the second day. The sheet was rosin sized at a level of rosin addition of 8 lb/ton (4 Kg/metric ton) and alum addition of 10 lb/ton (5 Kg/metric ton). The pH on the machine was kept at 4.5 using sulfuric acid.

The headbox temperature was kept at 110°F (43°C) and a retention aid, Kato 15 cationic starch, was metered in at a mix tank prior to the headbox at a level of 10 lb/ton (5 Kg/metric ton). The dandy roll was run on the machine and three nips were taken in the calender stack.

The sheet was sized externally by use of the size press. Penford 280 ethylated starch was used at 6 to 7 percent solids and the size press temperature kept at 140°F (60°C).

The machine was run 7 hours manufacturing paper for the control sample which contained the No. 2 coating clay as filler and 6 hours manufacturing paper for the wet oxidized, recovered filler sheet.

When starting to run the recovered filler sheet, problems were encountered with pH control on the machine. The acid strength had to be increased several fold in order to compensate for the demand imposed by the wet oxidized, recovered filler. This will be explained further in the discussion of results.

During each day's run, four 30-ft (9.1 m) lengths of paper were taken for later evaluation. Samples of the headbox, first tray water, and wet end overflow were taken also in order to establish retention characteristics.

PRINTING TRIAL

The printing trial was conducted at Banta Corporation in Neenah, Wisconsin. An American Type Founders, four-color, blanket-to-blanket, perfecting offset press was utilized. The color sequence used was blue, red, yellow, black. The paper surface temperature was kept at 190°F (88°C) after the dryers. The printing speed was 700 to 800 fpm (215 to 245 m/min). The press had a cloth dampening system.

The test paper was printed at the end of a regular commercial run. The plates had 23,000 impressions on them at the start of the trial. A roll of the control paper was printed with about 2500 signatures. A roll of paper containing the wet oxidized, recovered filler was subsequently printed with approximately the same number of signatures. One hundred signatures were randomly collected from each roll and shipped back to Western Michigan Unviersity for evaluation. From these 200 signatures, 5 from each condition were randomly selected and submitted to a printing expert for evaluation of 5 printing properties: holdout, strikethrough, showthrough, smoothness and sharpness. They were evaluated on a scale of 1 - poor, 2 - fair, 3 - average, 4 - good, 5 - excellent.

LABORATORY PHYSICAL, OPTICAL AND PRINTING TESTS

The same set of tests were used as were discussed in Section IV of this report.

SECTION XI

DISCUSSION OF RESULTS FOR USE OF

WET OXIDIZED, RECOVERED FILLER AS A FILLER PIGMENT

The wet oxidized, recovered filler appears and acts very much like a normal clay slurry. Characteristics are shown in Table 17. It was handled and metered during the pilot machine trials in the same manner as a clay slurry. The only difficulty which arose was with pH control on the machine. pH control proved impossible until the acid strength was increased several To check the magnitude of this acid demand, as compared to a sample of No. 2 coating clay, a 25 ml sample of wet oxidized recovered filler slurry was titrated with .1N sulfuric acid from its pH of 7 to the pH 4.5 used in the pilot machine trials. From Figure 4 it is apparent that the recovered filler consumes about 48 times as much acid as normal No. 2 coating clay. This calculates out to 2.73×10^{-3} equivalents of acid/gm of wet oxidized, recovered filler. The acid demand of the wet oxidized, recovered filler at pH 4.5 will be an additional cost to be born. problem can be avoided by running at neutral conditions. increased acid demand may have been due to calcium carbonate being present in the wet oxidized, recovered filler.

Table 17. CHARACTERISTICS OF FILLER RECOVERED BY WET OXIDATION OF DEINKING SLUDGE

Parameter	Pilot data
Original sludge ash content (%) Oxidized sludge ash content (%) Oxidized sludge purity (%) Brightness (% elrepho) Abrasion (mg)	45.0 85.0 100.0 81.4 6.4

Table 18 gives the retention characteristics for each day of the pilot machine run. There does not appear to be a significant difference in the retention behavior of the wet oxidized, recovered filler as compared to No. 2 coating clay.

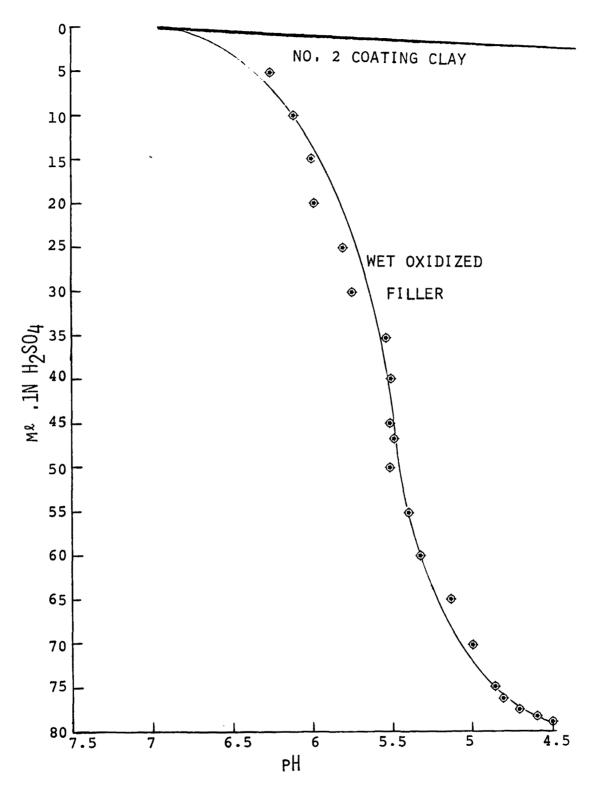


Figure 4. Acid demand of wet oxidized pigment.

Table 18. RETENTION CHARACTERISTICS OF WET OXIDIZED RECOVERED FILLER

	First pass	Overall
Control sheet	73.1	83.6
Wet oxidized recovered filler sheet	74.4	84.8

The physical properties of the sheets made during the pilot trial and a sample of the mill-manufactured paper which was being simulated are given in Table 19. Only MD TEA was significantly different from the control at the 99.5 confidence level and is not far enough out of line to be of practical concern. Note also that the dirt count did not increase significantly.

The optical properties of the sheets are listed in Table 20. The sheet containing recovered filler lost 2.6 points in brightness but gained 4.9 points in opacity. This kind of a trade of opacity for brightness could probably be tolerated. The increase in opacity may be due to the presence of titanium dioxide in the recovered filler; whereas, there was none at all present in the control sheet. Analysis of the wet oxidized, recovered filler showed it to contain about 1 percent TiO₂ which would account for about half of the opacity increase.

The laboratory printing properties are given in Table 21. The only difference between the control sheet and the sheet containing the recovered filler is in the test for hiding power where the increased opacity of the sheet containing the recovered filler manifests itself.

The paper was shipped to a commercial printer and printed on a four-color ATF perfecting web offset press. One roll of paper containing the wet oxidized, recovered filler was run as was a roll of the control. The results of the printing evaluation are presented in Table 22. The print quality was very uniform and the wet oxidized pigment sheet compared well to the control sheet, rating better than the control on showthrough. The printing expert's detailed comments are contained in Appendix D.

Table 19. WET OXIDIZED, RECOVERED FILLER UNCOATED OFFSET PAPER PHYSICAL PROPERTIES

Physical properties	Control	Recovered filler	Mill Made
Basis weight (lb/3300 ft ²)/ (gm/m ²)	51.8/76.7	51.4/76.1	46.5/68/8
Caliper (.001 in)/(mm)	1.1/1.6 3.80/0.097	1.1/1/6 3.85/0.098	
o Porosity (Sheffield)	0.0 /0.002 117 3.1	0.10/0.003 115 6.7	0.05/0.001 102 6.1
Smoothness (Sheffield)	125 15.2	121 21.2	153 27.8
MD Tensile (Kg)	10.0 0.35	9.16 0.62	9.39 0.79
CD Tensile (Kg) o MD Tensile factor	5.65 0.30 0.13	5.38 0.22 0.12	6.23 0.28 0.136
σ CD Tensile factor	0.005 0.073	0.009	0.008
σ MD Stretch (%)	0.004 1.82	0.003 1.63	0.004 1.64
σ CD Stretch (%)	0.15 3.79 0.59	0.14 4.10 0.47	0.18 4.23 0.46
MD TEA (Kg(m)/m ²)	3.55 0.36	2.88 a 0.38	2.94
CD TEA (Kg (m)/m ²)	5.02	5.24 0.73	6.32 0.95
Mullen (PSI)/(kPa)	18.3/126 1.5/10.3 58.4	17.4/120 0.95/6.5 59.3	20.5/141 2.1/14.5 48.4
MD Tear (gm) o CD Tear (gm)	5.6	4.0	3.4 49.0
σ MD MIT fold (number)	4.2 62.9	5.1 60.2	4.3
σ CD MIT fold (number)	35.1 21.5 6.4	17.1 18.9 4.6	27.5 63.8 15.7
σ Dirt specks σ	45.0 13.4	58.8 9.1	

Statistically different from the control at 99.5% confidence level as indicated by a t test.

Table 20. WET OXIDIZED, RECOVERED FILLER UNCOATED OFFSET PAPER OPTICAL PROPERTIES

Optical properties	Units	Control	Recovered filler	Mill Made
Brightness Opacity (TAPPI) o	% Elrepho	82.5 0.15 85.4 0.52	79.9 a 0.17 90.3 a 0.59	77.7 0.39 89.6 0.45

Statistically different from control at 99.5 confidence level as indicated by a t test.

Table 21. WET OXIDIZED, RECOVERED FILLER UNCOATED OFFSET PAPER PRINTING PROPERTIES

		Recovered	Mill
Lab printing properties	Control	filler	made
Wax pick σ	12.1 0.45	11.1 0.80	13.2 0.83
K & N Ink	48.4	52.3	44.4
σ	4.25	5.09	6.48
Hiding power	0.87	0.93 ^a	0.90
σ	0.028	0.007	0.014
$VVP(x 10^4) \text{ gm/sec}^2$	9.7	10.0	21.7
$\sigma (x 10^4)$	0.82	1.17	2.1
Hercules size (sec)	58.6	77.6	76.7
σ	21.2	10.9	15.2
IGT Pick (cm/sec)	118	122	264
(Polybutene oil 822 poise) σ	10.9	14.0	26

Statistically different from control at 99.5 confidence level as indicated by a t test.

Table 22. UNCOATED OFFSET SHEET PRINTING RESULTS

	Sample	(Gloss) Ink holdout	Strike- through	Printing (smoothness) mottle	Ink spread (sharpness)	Show- through	Total
Wet oxidized pigment	R 1	2	3	3	3	2	12
	R 2	2	2	3	3	2	12
	R 3	2	2	3	3	2	12
	R 4	2	2	3	3	2	12
	R 5	2	2	3	3	2	12
Control	C 6	2	2	3	3	1	11
	c 7	2	2	3	3	1	11
	C 8	2	2	3	3	1	11
	C 9	2	2	3	3	1	11
	C10	2	2	3	3	1	11

5. Excellent 4. Good 3. Average 2. Fair 1. Poor

SECTION XII

POTENTIAL OF WET OXIDIZED, RECOVERED FILLER

AS A POSSIBLE COATING PIGMENT

When the recovered filler obtained by wet oxidation was dried, repulverized using a mortar and pestle, and pressed into a pellet, it had an elrepho brightness of 81.4 percent. This is about equal to the brightness of filler grade clays but 5 to 6 points lower than conventional No. 2 coating clay. Its lower brightness would be its first drawback as a coating pigment.

The cooperating mill contracted with the Institute of Paper Chemistry to have particle size distributions run on the recovered filler. These are shown in Figure 5. Its distribution lies between that of a No. 2 coating clay and a filler clay. Seventy-four percent of the recovered clay is less than 2 μm where a No. 2 coating clay has 81 percent less than 2 μm .

The recovered fillers dispersing agent requirement was determined using a modification of TAPPI Procedure T648, using sodium hexametaphosphate. It did not readily disperse even when treated with excessive quantities of the dispersant.

Hercules viscometer rheograms taken on the wet oxidized, recovered filler at 40 percent solids showed it required about four times the shear stress to get the material to shear as compared to No. 2 coating clay at the same solids level.

The wet oxidized, recovered filler is a mixture of all of the inorganic material which was in the deinking sludge before wet oxidation. Transmission electron photomicrographs done at Western Michigan University illustrate this most readily. The recovered filler contained kaolin clay, calcium carbonate, titanium dioxide and other unidentifiable material.

It seems unlikely that this material would be suitable as a coating pigment due to its heterogeneous nature, lower brightness, particle size distribution and dispersion difficulties; but it is suitable as a filler clay substitute.

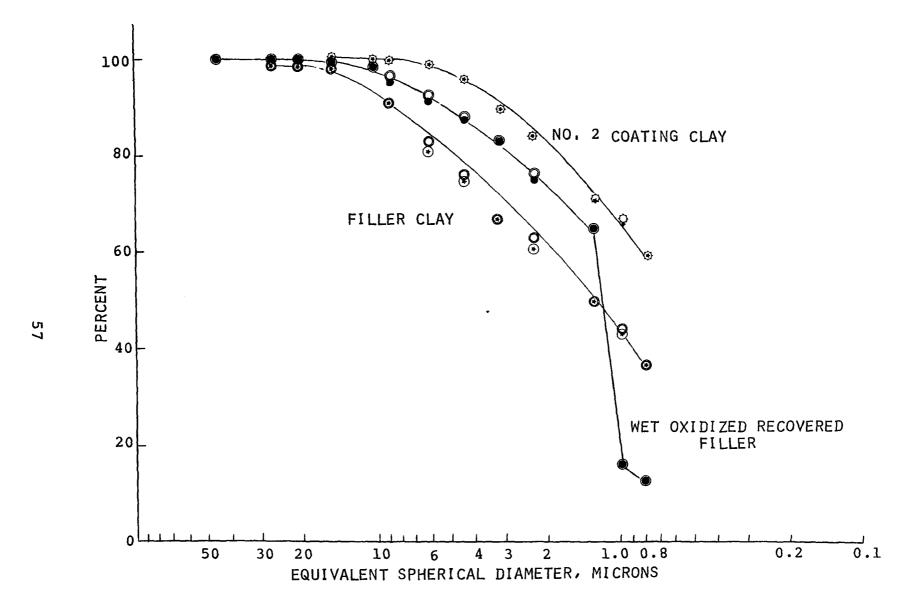


Figure 5. Particle size distribution of wet oxidized recovered filler.

SECTION XIII

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SECTION XIV

APPENDICES

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APPENDIX A

LABORATORY EVALUATION OF RECOVERY TECHNIQUES

EXPERIMENTAL PROCEDURE

The experimental design for this laboratory project was divided into three main parts. The first was to compare four methods of recovering an acceptable filler grade clay from high ash primary paper sludge. These four methods were centrifugal separation, foam flotation, screening and wet oxidation. The second part of the design was to evaluate the fillers recovered from each method. The percent filler, brightness and abrasiveness were determined in this evaluation. The final part was to make up handsheets for the two most promising techniques. These sheets were tested for their optical, strength and sizing properties.

The high ash primary sludges used in this project were from two different paper mills, both making printing and bond grades of paper. However, one mill was involved in deinking operations and the other was not. Enough of both types of sludges were stored in a refrigeration unit so that all of the recovery methods could be evaluated from the same sludge.

Recovery Methods

All of the recovery method trials were done at Western Michigan University and were run on both types of sludges.

Centricleaning--

The centrifugal separation was done using a No. 600 3-inch (7.62 cm) Bauer Centricleaner. The inlet pressure and back pressure were kept constant at 50 and 5 psig (340 to 34 kPa) respectively. The sludge was diluted to two consistencies, 0.1 percent and 0.5 percent. Both the accepts and the rejects from the cleaner were analyzed.

Foam Flotation--

The foam flotation work was done in a Fegergran Lab Flotation machine made by WEMCO (Western Machinery Company). The surfactant, its concentration and the sludge conditioning had been predetermined by work done by Prakasha Misra for his Senior Thesis in conjunction with the NCASI. The surfactant was a

partially soluble, cationic quaternary ammonium chloride, Kemamine Q-6502, which is produced by Humko Products. Enough sludge to yield 20 grams dry suspended solids was put into a Waring Blender and broken up for 5 minutes. Next the pH was adjusted to 6.5 using dilute NaOH or H₂SO₄. The concentration of the surfactant was 0.75 percent of the 20 grams suspended solids. The surfactant was added to the pH adjusted sludge and then mixed well in the Waring Blender for 5 minutes at low speed. Finally, the sludge was put in the flotation cell and diluted to approximately 0.70 percent solids. The air flow to the cell was regulated to maintain a small bubble size and a supporting layer of foam on the surface. The flotation cell was run for 3 to 4 minutes and then the volume of both the froth and the tailings were measured and analyzed.

Screening--

The separation of the filler from the fiber by simple screening was accomplished with 18-inch SWECO vibrating screen. The sludges were diluted to 0.1, 0.4, 1.0, 2.0 and 3.0 percent solids. The sludge was placed over the vibrating screen at a constant dry solids loading of .04 pounds per minute (18 gm/min). Two screen mesh sizes, 230 and 325, were used at all five of the percent solids levels. The weight setting on the bottom of the motor was kept at 20° so that the fiber would go out across the screen rather than spiraling around the screen which tends to trap the fillers. Samples from the overflow and the flowthrough were analyzed.

Wet Oxidation --

The final method used to recover the fillers was wet oxidation. The sludge was diluted to 2 to 3 percent solids. One liter was poured into the stirred reactor built by Autoclave Engineers and sealed. Next, enough oxygen was added to bring the pressure up to 500 psig (3400 kPa). The conventional sludge was brought up to 600°F (316°C) and held there for 10 minutes. The maxium pressure that developed during these cooks was 2900 psig (20,000 kPa). The deinked sludge had to be heated up to 620°F (327°C) for 30 minutes before complete oxidation took place. A maximum pressure of 3400 psig (23,000 kPa) was recorded during the deinked cooks. After the bomb had cooled off, it was vacuumed out and the contents analyzed.

Analysis of Recovered Fillers

The recovered fillers were analyzed using the following tests.

Percent Filler or Ash Content--

The ash content was determined following TAPPI Standard Method T-113. A 15 percent loss of filler from ignition in the muffle furnace was assumed in all of the calculations.

Percent TiO2 --

A modification of TAPPI Standard Method T-439 m-60 was used to find percent TiO₂ of the recovered filler. This modification, which originated at Du Pont, uses potassium pyrosulfate to fuse the sample before it is put into solution.

Brightness--

A brightness pad was formed in a Buchner funnel, dried and then measured for brightness using the elrepho brightness tester. The brightness was reported percent elrepho. For the wet oxidation filler and the standard water-washed clay filler, a special disc was made due to the fact the dried fillers on the filter paper cracked badly.

Abrasion--

The flat disc method proposed by Frank and Schmitz, modified by the NCASI (4,5) was used to find the relative abrasiveness of solutions. A felt wrapped around an abrading block which was mounted in a drill press was placed in contact with a bronze disc and rotated while the filler slurry was circulated over the disc. The relative loss of weight of the disc measures the abrasion tendency of the material.

Handsheet Procedures

These samples of recovered fillers from the screening and wet oxidation method were used to make up laboratory handsheets. These were compared to sheets made with ones using a standard water-washed Kaolin filler clay. The filled sheets contained 20 percent clay addition and no addition of TiO₂ to give a target filler content of about 10 percent filler in the final handsheet. All of the handsheets were made on the Noble and Wood handsheet machine with target basis weight of 60 gm/m². The furnish, 60 percent bleached softwood kraft and 40 percent bleached hardwood kraft, was refined in a Valley beater until Canadian Standard Freeness of 350 was reached. The sheets were sized with 1.0 percent rosin and 2 percent alum. The pH in the mixing tank and in the sheet mold kept close to 4.5 with dilute sulfuric acid. They were conditioned overnight in a constant temperature and humidity room, then the following tests were run on them:

TAPPI Standard Method	<u>Test</u>
410	Basis weight
411	Caliper
452	Brightness (elrepho)
425	Opacity
494	Tensile
494	<pre>% Stretch</pre>
494	Tensile energy absorption
403	Brust (Mullen)

TAPPI Standard Method	Test
414	Tear (internal)
511	Fold (MIT)
413	Ash Content
-	Sizing - Hercules
499	IGT Pick
TAPPI Useful Method	K and N Ink
553	Hiding power

From the ash content of the handsheet, one pass filler retention was calculated. The tensile factor was calculated by dividing the tensile by the basis weight.

DISCUSSION OF RESULTS

Centrifugal Separation

Although centricleaners are quite widely used in the paper industry as a method to separate solids from fibers, their application as a filler recovery method did not prove to be fruitful. From Tables 1-A and 2-A it can be seen that the maximum increase in percent filler was only 8 to 10 percent. Also, the inconsistency in which the two sludges behaved seemed to bring out the ineffectiveness of the centricleaners. The conventional sludge had a maximum purity in the first pass rejects at 0.5 percent solids while the deinked sludge had its maximum purity in the accepts after four passes through the cleaners at 0.1 percent solids. The fillers used in the handsheets were those recovered in the first pass rejects at 0.5 percent solids for both sludges.

Foam Flotation

From previous work done, it was found that 90 to 92 percent of the filler materials could be recovered with purity increase of 17 to 18 percent or a total purity of 75 to 80 percent. work done on flotation for this project was also aimed at these figures but with a different flotation cell. The Fergergran laboratory flotation cell was a larger unit and it did not have the exact control over the bubble size that the previously used cell had. However, we did match results very closely as the figures of Tables 3-A and 4-A point out. The conventional sludge had a purity of 75 percent and a recovery of 90 percent. The deinked sludge was somewhat lower with a purity of 61.5 and a recovery of 70.5. The main variable with the Fergergran cell was the length of time that each batch should be run, because the purity decreased with time. By taking samples at 1-minute intervals, the purity and recovery was found throughout the transition of a flotation run. Figure 1-A is a plot of purity

Table 1-A. RECOVERED FILLER PURITIES FROM CENTRIFUGAL SEPARATION DEINKING SLUDGE

Centricleaner Pass	Total solids %	Ash,	Filler,	Fiber,
Raw sludge	28.5	45.0	52.9	47.1
At 0.5% solids				
<pre>lst pass: accepts lst pass: rejects 2nd pass: accepts 2nd pass: rejects 3rd pass: accepts 3rd pass: rejects 4th pass: accepts 4th pass: rejects</pre>	0.38 1.59 0.38 0.88 0.36 0.69 0.35 0.58	53.5 34.4 51.8 33.9 53.8 39.3 54.2 41.5	62.9 40.5 61.0 39.9 63.3 46.2 63.7 48.8	37.1 59.5 39.0 60.1 36.7 53.8 36.3 51.2
At 0.1% solids				
lst pass: accepts lst pass: rejects 2nd pass: accepts 2nd pass: rejects 3rd pass: accepts 3rd pass: rejects 4th pass: accepts 4th pass: rejects	0.11 0.52 0.11 0.20 0.10 0.15 0.10 0.13	52.7 27.0 55.9 38.1 57.0 43.7 57.9 50.1	62.0 31.8 65.7 44.9 67.1 51.4 68.1 59.0	38.0 68.2 34.3 55.1 32.9 48.6 31.9 41.0

Table 2-A. RECOVERED FILLER PURITIES FROM CENTRIFUGAL SEPARATION CONVENTIONAL SLUDGE

	Total	Ash,	Filler,	Fiber,
<u>Centricleaner pass</u>	solids %	ૠ	ક	8
Raw sludge	4.91	55.0	64.7	35.3
At 0.5% solids				
<pre>1st pass: accepts 1st pass: rejects 2nd pass: accepts 2nd pass: rejects 3rd pass: accepts 3rd pass: rejects 4th pass: accepts 4th pass: rejects</pre>	0.40 2.52 0.40 0.94 0.39 0.64 0.40	49.4 61.8 49.3 46.2 49.5 45.4 49.0 48.6	58.2 72.7 58.0 54.3 58.3 53.4 57.6 57.2	41.8 27.3 42.0 45.7 41.7 46.6 42.4 42.8
At 0.1% solids				
lst pass: accepts lst pass: rejects 2nd pass: accepts 2nd pass: rejects 3rd pass: accepts 3rd pass: rejects 4th pass: accepts 4th pass: rejects	0.11 0.72 0.11 0.20 0.10 0.16 0.09 0.13	51.9 53.3 52.9 44.0 54.1 45.4 53.5 48.8	61.1 62.7 62.3 51.8 63.7 53.5 62.9 56.4	38.9 37.3 37.7 48.2 36.3 46.5 37.1 42.6

Table 3-A. SUMMARY OF HIGH ASH SLUDGE LABORATORY RECOVERY TECHNIQUE DATA - PURITY (% of product which is filler material)

Separation technique	Conventional sludge %	Deinking sludge %
Original sludge	48.0	52.9
Centricleaning	72.7	62.9
Screening	91.5	76.5
Foam flotation	75.5	61.5
Wet oxidation	100.0	100.0

Table 4-A. SUMMARY OF HIGH ASH SLUDGE LABORATORY RECOVERY TECHNIQUE DATA - RECOVERY (% of filler in original sludge which is recovered)

Separation technique	Conventional sludge %	Deinking sludge %
Centricleaning	1.5	98.3
Screening	97.7	97.3
Foam flotation	90.5	70.5
Wet oxidation	100.0	100.0

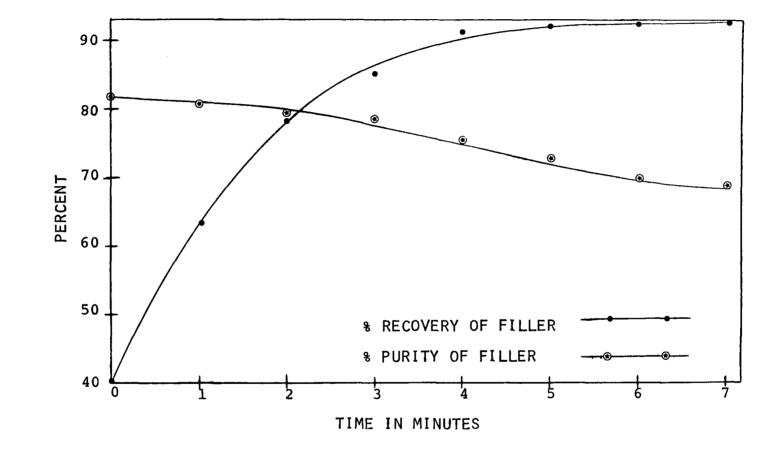


Figure 1-A. Foam flotation: time vs. % filler purity and recovery

and recovery versus time. From this figure, it was decided that a batch run would be 3 to 4 minutes long.

Screening

Figure 2-A summarizes all of the work done with the SWECO vibrator screen. It shows how the recovery for both sludges was very high with 96 percent minimum and that the 325-mesh and 230-mesh lines were almost the same. This means the 230-mesh screen was just as effective as the 325-mesh screen for the recovery of the filler. An analysis of variance was run to find out if the percent solids, mesh size or their interaction significantly affected the purity and the recovery.

At a 95 percent confidence level, neither the mesh size nor their interaction affected the purity or the recovery. However, as the percent solids increased, the purity also increased. Although recovery decreased slightly with increasing solids, it was not significant at a 95 percent confidence level.

Wet Oxidation

The conditions for wet oxidation were chosen to ensure a complete oxidation of all fibrous materials. At these extreme conditions, the purity and recovery were both 100 percent.

Summary of Separation Results

A final summary of the methods of recovering filler materials from high ash primary sludge is shown in Tables 3-A and 4-A. Wet oxidation and screening have high recovery for both types of sludges. The purity of the deinked sludge was lower than the conventional sludge in every method except wet oxidation. The centricleaners and vibrating screen were much easier unit operations to run. Because of its high recovery with either sludge, good purity and ease of operation, screening appeared to be the best method to recover the filler material from the sludge.

In Table 5-A the brightnesses of the recovered filler for both sludges is listed. When these were compared to a standard filler clay which has a brightness of 81 to 82 percent elrepho, it was decided that the recovered filler would probably cause some loss in sheet brightness when employed.

Abrasiveness

One of the main concerns in reusing the filler was that the filler would become more abrasive. From Table 6-A it can be seen that the fillers from wet oxidation were more abrasive than the filler from the other methods. However, the relative

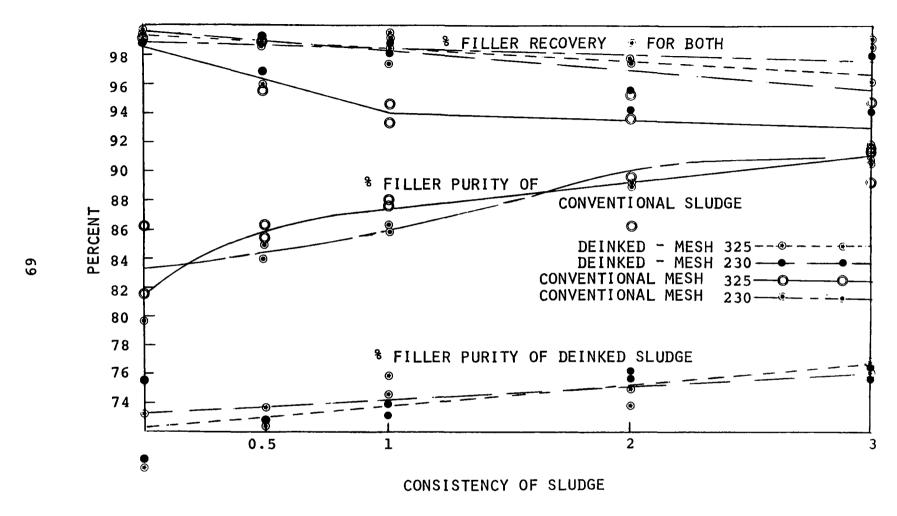


Figure 2-A. Screening. The effect of screen mesh and consistency on purity and recovery.

Table 5-A. BRIGHTNESS RESULTS (Elrepho)

Separation tech.	Deinking	Conventional
Raw sludge	50.8	63.0
Centricleaners	54.5	61.1
Screening	56.0	71.3
Flotation	50.6	71.7
Wet oxidation	73.5	71.7

Table 6-A. ABRASION RESULTS BY FLAT DISC METHOD

Sample	Slurry consistency	Deinking wt loss (mg/disc)	Conventional wt loss (mg/disc)
Filler grade clay	10	7.1	7.1
Filler grade clay	6	6.3	6.3
Centricleaned sludge	6	1.4	1.0
Screened sludge	10	4.1	4.2
Foam floated sludge	6	1.3	2.4
Wet oxidized sludge	10	6.4	7.0

abrasiveness of the filler from any of the methods was not greater than a normal filler grade clay.

Effect of Screened and Wet Oxidized Recovered Filler on Physical and Optical Properties of Hand Sheets

Contained in Tables 7-A and 8-A are the results of handsheet tests made employing filler recovered by the screening and wet oxidation techniques for the conventional and the deinking mill sludges. The blocked figures are numbers which were shown to be statistically lower than the control at a 99.5 percent confidence level using the t test. The only property of practical concern which changed was the brightness. The screened, recovered filler caused a drop of 3.6 points for the conventional sample and 10.9 points for the deinking sample. The 3.6 drop in brightness may be able to be tolerated but certainly not the 10.9 drop. Thus, it appears that from a laboratory handsheet viewpoint, screening for filler recovery may be a possibility for a mill not practicing deinking. Wet oxidation will be the route a deinking mill will probably have to pursue. None of the other physical or optical properties tested seem to be affected.

Table 7-A. HAND SHEET PROPERTIES MODIFICATION (conventional)

Property	Control	Screened	Wet-oxidized
Basis wt. (G/M ²)	64.6	64.4	63.4 a
Caliper (.001 in.)/(mm)	5.41/0.137	5.38/0.137	5.22/0.133
Brightness (elrepho)	72.5	68.9 a	73.4
Opacity (TAPPI)	83.6	87.6	85.4
Tensile (Kg)	7.98	7.99	7.59
Tensile factor (Kg M ² /gm)	0.125	0.126	0.119
Stretch (%)	2.64	2.62	2.54
Tear (gm)	52.8	48.0	56.8
Mullen (PSI)/(kPa)	22.6/156	24.3/167	25.8/178
Fold (number)	21	30	34
Hercules size (sec)	66.5	35.9	118.5
Filler (%)	9.4	10.9	11.4
Retention (%)	46.9	54.6	56.9
IGT Pick	255	245	245
Hiding power	0.77	0.87	0.85
K & N Ink (% red.)	46.3	50.8	53.3

a Significantly different from control at 99.5 confidence.

Table 8-A. HAND SHEET PROPERTIES MODIFICATION (deinking)

Property	Control	Screened	Wet-oxidized
Basis wt. (G/M ²)	64.6	63.7 a	61.3 a
Caliper (.001 in.)/(mm)	5.41/0.137	5.39/0.137	5.28/0.134
Brightness (elrepho)	72.5	61.6 a	75.2
Opacity (TAPPI)	83.6	92.1	89.1
Tensile (Kg)	7.98	7.52	7.50
Tensile factor (Kg M ² /gm)	0.125	0.117	0.122
Stretch (%)	2.64	2.42	2.16
Tear (gm)	52.8	48.8	55.2
Mullen (PSI)/(kPa)	22.6/156	23.7/163	20.0/138
Fold (number)	21	27	13
Hercules size (sec)	66.5	72.7	77.1
Filler (%)	9.4	12.1	10.9
Retention (%)	46.9	60.6	5.4
IGT Pick	255	255	230
Hiding power	0.79	0.93	0.88
K & N Ink (% red.)	46.3	47.5	54.8

Significantly different from control at 99.5 confidence.

APPENDIX B

DETAILS OF PRINTING TRIAL AND EVALUATION FOR COATED FINE PAPER SAMPLES

ASSESSMENT OF PRINTING QUALITY OF COATED OFFSET PRINTED SAMPLES CONDUCTED BY E.W. RAYFORD

The absence of standards upon which the characteristics of printability of the test sheets could be based, limits the comprehensiveness of this evaluation. In this case, the separate test sheets were evaluated one against the other in an attempt to arrive at comparative values.

The primary difficulty encountered in the evaluation was the poor quality of the printing. Sharpness and gloss were two of the characteristics to be evaluated. Close examination with 10X, 12X and 30X glasses revealed more than minimal differences in these two characteristcis between the samples. Color, gloss and sharpness are a function of the amount of ink laid on the Inspection revealed visible differences in ink coverage Not knowing the printing sequence of the samples, I and color. was unable to determine whether the differences were due to paper factors or press problems. After determining that there was little difference in the amount of showthrough and strikethrough between the samples, and that the printing quality was markedly different on the back sides of some of the samples, I decided to run a series of densitometer readings on the four process colors. These curves showed wide latitudes in color control on the press. Correlations can be drawn between the amount of ink on the sheet and the differences in penetration and gloss, and printing sharpness.

Examination of the slur gauges also reveals press problems. Good dot hardness and sharpness is most often a result of good press conditions. Evidence of the word "slur" on all of the samples indicates less than optimum printing quality. Examination of both highlight and shadow dots under a 30X glass indicates soft, mushy and slurred dot formation, particularly on the blue and black. This would result in a lack of printing sharpness and halftone quality.

A certain amount of scumming is apparent on many of the samples. I think this can also be attributed to press conditions rather than to paper factors.

The mis-register also lowers the quality of the printed job and the determination of fine detail.

After making close examinations of both sides of each sample, and close comparisons between the samples, keeping in mind the lack of uniformity of press control, I found no major differences in the printability of the samples. Samples 14, 17 and 26 were less opaque; and samples 21, 23 and 26 in particular, lacked sharpness in the halftones. There were little apparent differences in strikethrough, mottle, pick resistance, fiber puffing and blister, and ink spread. These were all considered to be satisfactory. Opacity was considered to be somewhat low in all samples where there was no backup, particularly samples 23 through 26, but showthrough was not evident when backed up with light colors.

One characteristic noticed was the softness of the samples and the lack of resistance to tear in handling.

In my opinion, the printing characteristics of all of these samples were satisfactory, and that most of the objectionable qualities found in the printing were due to press conditions. However, whether or not these conditions could be controlled to a closer degree on the press using these papers cannot be ascertained from these samples, but should be determined from the reports of the press conditions and paper runability.

APPENDIX C

DETAILS OF UNCOATED WEB OFFSET PRINTING TRIAL

AND PRINTING EVALUATION

ASSESSMENT OF PRINTING QUALITY OF UNCOATED FINE PAPER PRINTED SAMPLES CONDUCTED BY E.W. RAYFORD

An honest and genuine attempt was made to evaluate the printing qualities of the thirty-five samples submitted to me. Because of wide variation in the quality of the printing and ink coverage, I feel the evaluation of the printing quality of the paper is subject to some question.

The differences, in my opinion, were due more to printing quality and press condiitons than to paper characteristics. Of course, there is a big difference between most of the printing qualities of the lightweight stocks and the heavier weight stocks.

Because of the imposition of the backup forms, evaluation of some qualities were limited. The solid color bars were backed up by another bar of the same color; the black halftones were backed up by the GRETAG Control Bar; large color areas, screens and process were backed up by large color areas. Therefore, strikethrough and showthrough were confined to limited areas. The amount of strikethrough, showthrough and lack of ink holdout was in most cases, directly related to the amount of ink applied. Color density could be expected to be greater in the color bars because of being backed up by the same color, particularly in the lighter weight stocks.

Printing sharpness was evaluated in the half tones and the screened areas. Because of the large differences in ink coverage, evidence of plate wear and breakdown, and other printing factors, I think sharpness was more a condition of printing quality than paper quality. An additional twenty-two samples were added to the original thirty-five to give me a better example of plate image breakdown, but because these samples too, were randomized, they had no more meaning than the original thirty-five. Where ink coverage was light and the plate new, sharpness was good in all categories, highlights, mid-tones and shadows. As the run progressed, all areas got progressively

worse, particularly when ink coverage was heavier. It was also noted to deteriorate more in the shadow areas where dot structure disappeared entirely in some areas.

A special effort was made to evaluate the printing sharpness in the halftones on the additional samples which were used to assess the importance of plate wear on printing. Highlights, midtones and shadows were evaluated, and recorded individually as well as the average overall sharpnes. It should be noted that there was little, if any difference noted between these samples and the other thirty-five samples. The same faults, too little or too heavy ink coverage, dot breakdown and loss of shadow dot detail, slur and scum are all evident in various samples. Densitometer readings were recorded for the black on these samples in an attempt to relate the amount of ink coverage with printing sharpness. No densitometer readings were recorded for the other three colors on these samples.

As a whole, I would say printing sharpness was poor, except in a few cases. However, these conditions were noted to be similar on all paper samples, with the difference between weights again being noted.

Printing smoothness or mottle was evident in the blue on all samples, and in the black on some samples where the ink coverage was heavy. The other colors were good.

Ink spread was noted on the head of the red color bar in some cases, and on the tail of this same color bar on others. It was noted on the tail of the black bar on several samples. Also, the dot structure of the screens was noted to be soft on many samples. This could be a condition of ink spread or dot slur. The SLUR gauges were noted as being bad on many samples.

Ink coverage was very inconsistent, both between samples, and in many cases, within a sample. In some cases, coverage was very light, the paper showed through the ink so badly that no evidence of solid coverage was apparent. On these samples, sharpness was rated accordingly higher. Ink coverage can be noted on the densitometer charts.

There was a good deal of black ink transfer on the other colors caused by press rollers. There was also scumming evident. This made taking densitometer readings in some areas impractical.

Register is bad on many of the process samples, and is also evident in the back-up of the Control Strip.

In my opinion, the average quality of the printing is low, but I think this is due to press conditions and problems as similar faults are evident on all of the different types of paper, the exception being between the lightweight papers and

the heavier papers. Quality, overall, is lower on the lighter weight samples.

There was little, if any, evidence of fiber pick on any of the samples.

In all cases, the lightweight and the heavier weight samples were separated for evaluation, but samples within the separate weights were randomized. The charts are sequential for ease of recording. (See Table 11 in "Discussion of Results" Section.)

Densitometer Readings Macbeth RD-100

Densitometer readings were taken in several areas: on the solid patch of the Control Strip, in the center of the solid color square, and on the left end, center and right end of the solid color bar. As the yellow GRETAG color patch, the color square and the left end of the solid color bar are all in line of press travel, these densitometer readings were the same. However, by moving the aperture about one and one-half inches to the right, a much higher reading was obtained. This difference is noted between the columns marked GRETAG and Color Bar, left on the yellow.

Also, differences in color values could be obtained on all colors by moving the aperture fractions of an inch. Therefore, a special effort was made to read the same respective area on all colors on all samples (except as noted above on the yellow).

Areas marked NG could not be recorded, either because the area was contaminated with another color, mostly black, or showthrough of mis-register on the lighter samples made the reading meaningless.

DENSITOMETER READINGS

		Yell	.ow			Magen	ta			Cyan	1			Blac	:k			GRETAG					
•		Color har					Color bar Color bar Color bar								Color bar				Color control system				
			0101				101 1				101 1	1				1		1					
Sample number	Square color patch		Center	Right	Square color patch	Left	Center	Right	Square color patch	Left	Center	Right	Square color patch	Left	Center	Right	Yellow	Magenta	Cyan	Black			
1. 2.	0.82	0.91	1.00		0.93	0.88		0.92 0.96	1.20			1.02	1.04	0.94		1.06	0.82	0.89	1.17	1.00			
3.	0.82	0.92		0.86	0.99		1.04		1.22			0.98	1.12	0.91			0.82	0.90	1.13	1.00			
4.	0.80		1.00		1.04		1.14		1.18	1.06	1.10	1.06	1.16	1.01	1.18	1.10	0.80	0.92	1.14	1.00			
5.	0.82	0.93		0.86	1.04		1.12		1.15			1.06	1.10	1.02			0.82	0.97	1.15	1.04			
6.	0.87	1.00	1.03		1.14		1.18		1.19		1.17		1.30	1.18			0.87	1.08	1.15	1.32			
7.	0.90	NG NG	1.04	0.93	1.12	1.04		1.02	1.18			1.10	1.28	1.22	1.33	1.22	0.90	1.06	1.10	1.26			
8. 9.	0.86	NG			0.98	0.90		0.86	1.16			1.04	1.22	1.13			0.86	0.92	1.11	1.33			
10.	0.87	NG	1.00		1.04		1.06		1.14			1.12	1.26		1.30		0.86	0.98	1.06	1.22			
11.	0.86	0.97	NG	0.88	0.95	0.86		1.04	1.06	0.92	1.02		1.32		1.36		0.86	0.86	1.02	1.25			
12.	0.90	0.98		0.92	1.01		1.06		1.14	1.06		1.10	1.46	1.23			0.88	0.94	1.10	1.28			
13.	0.90	0.96	1.02		1.02		1.08		1.01	0.91		1.09	1.40	1.18			0.90	0.94	1.12	1.28			
14.	0.92		1.02		1.00	0.96		1.06	1.14			1.07 1.05	1.34	1.18			0.92	0.96	1.04	1.28			
15. 16.	0.90	0.98	0.98		0.98 1.12	0.90		1.08	1.16 1.22			1.05	1.33		1.38		0.90	0.94	1.04	1.26 1.34			
17.	NG		1.00		1.11		1.20		1.20			1.10	1.32		1.44		0.88	1.06	1.06	1.35			
18.	0.88	0.98	1.02		1.15	1.06		1.14	1.16			1.06	1.36	1.18			0.88	1.04	1.11	1.32			
19.	0.86		0.99		1.10	1.02	1.22	1.14	1.18			1.10	1.40		1.48		0.86	1.10	1.16	1.32			
20.	NG		0.98		1.04	1.02		1.16	1.20			1.06	1.40	1.10			0.88	1.06	1.14	1.36			
21.	0.92		1.06		0.99	0.92		0.88	1.20			1.12	1.21	1.24			0.92	0.96	1.12	1.20			
22.	0.93	1.02	1.07		1.00			0.89	1.28			1.10	1.23	1.10			0.93	0.94 NG	1.14	1.20			
23. 24.	0.92	1.03	NG 1.06	0.94	1.02		1.05	0.88	1.26			1.18	1.00	1.30			0.94	0.96	1.18	1.40 1.32			
25.	0.94	1.02			1.00		1.04		1.25			1.14	1.24	1.18			0.94	0.99	1.14	1.28			
26.	0.92	1.04			1.08			1.14	1.16			1.04	1.33	1.29		1.46	NG	0.98	1.07	1.39			
27.	0.92	1.08		0.98	1.06			1.12	1.23	1.18	1.22	1.12	1.46	1.29	1.47		NG	1.01	1.22	1.42			
28.	0.88	1.06	NG	0.96	1.06	1.02		1.15	1.24		1.24		1.42	1.32			NG	NG	1.18	1.41			
29.	0.87	NG	NG	0.94	1.10		1.20		1.30		1.18		1.44	1.33		1.42	NG	NG	1.20	1.36			
30.	0.85	1.06	NG	NG	1.07	1.01		1.15	1.25		1.23		1.44	1.28			NG NG	NG 0.96	1.17	1.32			
31. 32.	0.92	NG 1.04	NG 1.06	NG 0.94	1.04	0.95	1.04	1.02 i	1.25		1.18		1.48	1.34			0.90	1.00	1.18	1.40 1.50			

DENSITOMETER READINGS (continued)

		Yellow Magenta								Cyan				Blac	ck		GRETAG - RIT			
	}.		Color	bar		Co	olor l	oar		Cc	olor b	par	Color bar				Color control system			
Sample number	Square color patch	Left	Center	Right	Square color patch	Left	Center	Right	Square color patch	Left	Center	Right	Square color patch	Left	Center	Right	Yellow	Magenta	Cyan	Black
34.	0.90 0.93 0.94	1.04 1.06 1.04		0.96 0.99 0.98	1.00 1.02 1.05	1.01	1.08 1.20 1.18	1.10	1.25 1.14 1.12	1.05	1.24 1.14 1.12	1.10		1.33 1.24 1.40	1.55	1.50	NG NG NG	0.98 NG 1.00	1.24 1.06 1.04	1.3
Highest Lowest Range		0.91	1.10 0.98 -1.10	0.84	1.15	0.86	1.24 0.99 -1.24		1.30	0.91	1.36 1.02 1.30	0.98	1.48	0.91		1.06	0.94 0.80 0.14	1.10 0.86 0.24	1.24 1.07 0.17	1.0

DENSITOMETER READINGS

Sequen-	Yellow	Magenta	Cyan	Black			GRETAG - RIT				
tial Samples						Color	bar	C	olor c		1
Sample number				Square color patch	Left	Center	Rignt	Yellow	Mag- enta	Cyan	Black
A1 A2 B1 B2 C1 C2 D1 D2 E1 E2 F1 F2 G1 G2 H1 H2 I1 I2 J1 J2 K1 K2	NOT RECORDED	NOT RECORDED	NOT RECORDED	Ma 1.35 1.31 1.26 1.24 1.40 1.42 1.52 1.44 1.50 1.44 1.48 1.44 1.50 1.44 1.43 1.48 NG 1.45 1.40	1.46 1.44 1.34 1.28 1.36 1.36 1.30 1.34 1.16	1.32 M 1.16 1.34 1.36 1.54 1.54 1.54 1.50 1.60 1.56	1.23 1.30 1.18 1.19 1.32 1.21 1.41 1.41 1.38 1.40 1.52 1.34 1.46 1.38 1.42 1.56 1.47 1.22 1.28 1.39	NOT RECORDED	NOT RECORDED	NOT RECORDED	1.12 1.35 1.30 1.22 1.19 1.32 1.14 1.38 1.46 NG 1.52 1.38 1.40 1.20 1.40 1.32
Highest Lowest Range				1.52	0.86	1.60 1.14 -1.60					1.52 1.12 0.40

a_{Mottle}

APPENDIX D

DETAILS OF PRINTING EVALUATION OF UNCOATED

WEB OFFSET SAMPLES FROM WET OXIDIZED RECOVERED FILLER TRIAL

ASSESSMENT OF PRINTING QUALITY OF UNCOATED WEB OFFSET PRINTED SAMPLES CONDUCTED BY E.W. RAYFORD

A lack of standards limits the evaluation of these test sheets. The uncoated samples also appeared to have very similar properties of printability. All of the colors except black are screened, and most of them are arrived at through a combination of two or more screened colors; therefore, mottle, spread, and printing sharpness were more easily determined using the black printed areas.

Printing sharpness is satisfactory on all of the samples. Again, there is evidence of soft, mushy screen patterns but this was found to be similar on all of the samples. There was little evidence of ink spread into the unprinted areas.

Gloss was considered to be low on all colors except the black where it was judged to be satisfactory. There is evidence of scumming throughout the samples, more so on the black than on the other colors. This was attributed to press conditions such as ink-water balance (this could also be a factor in the low gloss), counter etching of the plate, or other plate or press factors. The higher gloss of the black and the larger amount of scumming could be a result of a heavier ink flow.

Printing smoothness was considered to be average for a base sheet such as this.

Opacity was considered to be poor. The amount of show-throught is objectionable, even when backed up. Samples 6 through 10, the control samples, were considered to be slightly worse than samples 1 through 5, the recovered filler samples.

There is evidence of strikethrough on all of the samples. The amount of strikethrough and the lack of opacity and gloss would, in my opinion, determine ink holdout to be only fair.

The printability of these sheets was considered to be satisfactory except in the areas of strikethrough and showthrough.

Table 1-D. PRINTING SAMPLE EVALUATION BY USE OF 1-5 GRADING SYSTEM

Sample	(Gloss) Ink Holdout	Strike Through	Printing (Smoothness) (Mottle)	Ink Spread (Sharpness)	Show Through
1	2	2	3	3	2
2	2	2	3	3	2
3	2	2	3	3	2
4	2	2	3	3	2
5	2	2	3	3	2
6	2	2	3	3	1
7	2	2	3	3	1
8	2	2	3	3	1
9	2	2	3	3	1
10	2	2	3	3	1

	TECHNICAL REPORT DATA (Please read Instructions on the reverse before comp	pleting)
1. REPORT NO.		3. RECIPIENT'S ACCESSION NO.
EPA-600/2-77-123		
4 TITLE AND SUBTITLE		5. REPORT DATE
INVESTIGATION OF REUS	July 1977 issuing date	
PAPERMILL SLUDGES	6. PERFORMING ORGAN: ZATION CODE	
7. AUTHORIS)		B. PERFORMING ORGANIZATION REPORT NO.
Allan M. Springer, Du	ane W. Marshall, Isaiah	
Gellman		
9. PERFORMING ORGANIZATION NAME		10. PROGRAM ELEMENT NO.
National Council of the	1BB610	
and Stream Improve	11. CONTRACT/GRANT NO.	
Central-Lake States R	egional Center	
Kalamazoo, Michigan	EPA-R-803348-01	
12. SPONSORING AGENCY NAME AND A		13. TYPE OF REPORT AND PERIOD COVERED
Industrial Environment		
Office of Research and	14. SPONSORING AGENCY CODE	
U.S. Environmental Pro		
Cincinnati, Ohio 4526	EPA/600/12	
15. SUPPLEMENTARY NOTES		· — — — — — — — — — — — — — — — — — — —

16. ABSTRACT

Two techniques for recovery of filler from fine papermill high ash sludges, screening and wet oxidation, were evaluated for their technical feasibility. The alternative of screening the sludge and using the material passing through the screen as recovered filler was investigated in cooperation with two mills. The screening for recovery of filler was conducted at the mill site. Then the material was shipped to Western Michigan University where selected grades incorporating the The paper manufactured was recovered filler were manufactured. shipped to Rochester Institute of Technology where it was printed on their web offset press. The grades simulated were found lacking only in that the brightness of the paper was from four to seven points lower than grades made with virgin filler. The wet oxidation alternative was evaluated in a similar manner with a cooperating mill. oxidized recovered fillers only slightly lowered the brightness of the sheet simulated and gave an increase in opacity in exchange. The wet oxidation scheme appears technically feasible. The screening alternative would probably have to incorporate a bleaching step before it would be deemed acceptable. The technical feasibility of oxidative bleaching was demonstrated.

17. KEY WORDS AND DOCUMENT ANALYSIS						
a. DESCRIPTORS	b.IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group				
Sludge, clay minerals, refractories, kaolin, opacifiers, fillers, papermaking, paper mills, book paper, oxidation, screening, separation, vibrating screens, recovery, reclamation	Fine paper, Wet oxidation	13/B,H 11/B 07/A				
18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 94				
Release To Public	20. SECURITY CLASS (This page) Unclassified	22. PRICE				