RETENTION OF CALCIUM CARBONATE DURING RECYCLING: DIRECT LOADING VERSUS FIBER LOADING

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ABSTRACT

Recycling mills produce two to four times as much sludge as virgin fiber mills, in excess of 200 kg/ton of paper processed. Recycling mills in the United States alone generates an estimated 4.1 million tons (dry basis) of sludge annually. This study compared the retention of calcium carbonate fillers by a pulp filled by fiber loading with the retention of calcium carbonate by conventional recycling process. Pulp obtained by both processes were recycled several times using a recirculating handsheet mold to evaluate relative impact on process water and, ultimately, sludge production. About 50% less calcium carbonate was needed to maintain the ash level of fiber-loaded handsheets than was needed for direct-loaded handsheets. Process water from fiber loading contained about 50% less calcium carbonate than the direct-loading control process water. Results indicate that fiber loading can reduce calcium carbonate loss by up to 50% compared with conventional direct loading of calcium carbonate, This study also indicates significant total sludge reduction in a deinking mill compared with current direct loading. This handsheet study should be verified by an industrial-scale evaluation.

INTRODUCTION

Worldwide, approximately 86 million tons of recycled fiber is used in paper and board manufacturing annually (1), and the use of recycled fiber is projected to increase to 130 million tons by the year 2001. Implicit in increased recycling is increased sludge production. Recycling mills produce two to four times as much sludge as virgin fiber mills, in excess of 200 kg/ton of paper processed. Recycling mill sludge typically has a high ash content, 50% to 65% (2). Currently the U.S. paper industry uses 3.9 million tons of kaolin clay, the largest category of filler consumed (3). The next largest filler type used is precipitated calcium carbonate (PCC), which is used as a paper filler at about half the rate of kaolin in the U.S. paper industry. The use of PCC has grown fourfold in the last decade and is expected to continue to increase, partly by the expected general increase in filler content in papers made in the United States. The U.S. wastepaper deinking mills alone generate an estimated 4.1 million tons (dry basis) of sludge annually (4), about a third of that is PCC and that amount will escalate with increased recycling. Disposal of recycling mill sludge is an immediate environmental and economic problem.

This study explored the impact a new fiber process could have on the calcium carbonate portion of sludge production during paper recycling. Fiber loading (5) precipitates a portion of calcium carbonate manufactured in situ during this process inside the fiber cell wall and lumen. Once precipitated on fiber interior and exterior surfaces, much of the carbonate remains with the fiber when recycled. This study compared the retention of calcium carbonate (pulp filled by fiber loading) with the conventional papermaking process that adds calcium carbonate to recycled pulp. Hot dispersion, frequently one of the last steps in recycling pulp, breaks down and distributes residual contaminants. Hot dispersion followed by direct loading of PCC (we designated these two steps as direct loading) is a common final step in deinking wastepaper. Both fiber loading and direct loading process high consistency pulp using a high consistency refiner. The fiber-loading process operates at ambient temperatures, and the direct-loading process dispersion step operates at elevated temperatures. However, our preliminary experiments suggest that the fiber-loading process results in at least as good dispersion of contaminants as the direct-loading process (6). So, fiber loading could possibly replace direct loading using the same high consistency refiners already in place. In this study, a common never-dried pulp filled by both processes was recvcled several times to evaluate relative impact on whitewater and ultimately, sludge production. The direct-loading and fiber-loading processes were compared with a control pulp to which calcium carbonate was added, directly followed by initial drying and recycled by fiberizing without additional mechanical treatment.

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EXPERIMENTAL

Materials

Commercial bleached virgin hardwood (90% birch, 10% aspen) kraft pulp as wet-lap was selected for this study to minimize variables in fiber type and papermaking history. Use of this pulp ensured that results would reflect differences caused exclusively by comparative fiber processing and level of calcium carbonate added. For direct loading of pulp, papermakers grade calcium carbonate was obtained from Specialty Minerals, Inc.; calcium hydroxide used for the fiber loading process was an industrial grade, obtained from Mississippi Codex Hydrated Lime.

Equipment

Hot dispersion processing for producing direct-loaded and fiber-loaded pulp was carried out in a 305-mm-diameter pessurized disk refiner manufactured by Sprout Bauer using refiner pattern plates. A semi-automatic recirculating handsheet mold, Model 255/SA manufactured by Messner Instruments, Limited, was used to prepare handsheets and recirculate process water (Fig. 1).

Methods

The control pulp was made from never-dried pulp. The pulp was fiberized in a high consistency pulper and diluted in a doler tank. Calcium carbonate was added to the pulp in the doler tank. Pulp pads were dewatered in a canvas bag on a vacuum crock and dried on a hot plate. Pulp pads were fiberized for recycling in a high consistency pulper.

For fiber loading, pulp, water, and calcium hydroxide were blended through the Sprout Bauer refiner at atmospheric conditions using the refiner plates to mix the materials. The gap was held at 1.9 mm for all pulp processing. After mixing, the materials for fiber-loaded pulp at about 25% consistency were passed through the refiner again, this time at 207 kPa (30 lb/in²) carbon dioxide pressure to precipitate calcium carbonate (PCC) (7,8).

For direct loading, pulp at 25% consistency was passed through the Sprout Bauer refiner at atmospheric pressure and 90°C at the same conditions as for fiber loading. For the direct-loaded pulp, calcium carbonate was added in the doler tank in the following step.

Pulp was added to the doler tank and handsheets formed until the system equilibrated, after three sets of 18 standard 1.2 g handsheets. That is, after two sets of handsheets, the process water had begun to approach steady state with respect to solids content. Handsheets from the third set of handsheets were selected for testing.

Pulp and Paper Tests

The handsheets were then pressed and air dried according to Tappi method T205. Physical testing of handsheets was determined by Tappi T220, Physical Testing of Handsheets (density brightness, opacity, scattering coefficient, burst index, tensile index and tear index). Pulp freeness was measured by Tappi Method T227. Pulp ash was determined by Tappi Method T211. Fiber length, fines measurement, and fiber coarseness were determined by a Kajaani FS 100 Analyzer. The process water analysis was performed by Tappi Method T620 em-83 for solids. Chemical oxygen demand (COD) was determined by the EPA-410.1 (titratamic) method used for examining water and waste water. Calcium ion demand was measured by atomic absorption.

RESULTS AND DISCUSSION

Handsheet Properties

For the control in Table I, the pulp was recycled by drying and re-slushing with no mechanical treatment. The optical and physical properties of the handsheets before and after recycling were similar in spite of a difference in ash content. Ash content of the handsheets made from the never-dried pulp was 13.19%, and the ash content for the handsheets from the once-dried pulp, even with additional calcium carbonate added, was 10.34%. Similar to the optical and physical properties, the 560 mL Canadian Standard Freeness (CSF) for the never-dried pulp was nearly the same as the 590 mL CSF for the once-dried pulp.

A more drastic treatment involving a simulation of hot dispersion of pulp is listed in Table I. The initial direct-loaded pulp gave a 620 mL CSF compared with 560 mL CSF for the never-dried control pulp. The ash content of this direct-loaded pulp was 10.71% compared with the 13.19% for the control. The lower ash content of the initial direct-loaded pulp should tend to decrease the freeness of the direct-loaded pulp, but we observed an increase for an unobvious reason. The increased strength properties of the initial direct-loaded pulp compared with the control may be explained in part by the lower ash content of the direct-loaded pulp.

Recycling the initial direct-loaded pulp for two more cycles with calcium carbonate added to hold the ash content of the resulting handsheets at between 10% and 11% showed somewhat reduced strength properties and steady optical properties. The handsheet apparent density increased about 5% between the initial and second cycles, and the freeness values decreased explaining the maintenance of strength properties.





Fig. 1. Semi-automatic handsheet mold

Table I. Handsheet and Freeness Properties

	Free- ness CSF (mL)	Density (kg/m ³)	Bright- ness ISO (%)	Printing Opacity (%)	Scattering Coef- ficient (m ² /kg)	Burst Index (kPa- m ² /g)	Tensile Index (N•m/g)	Tear Index (mN• m²/g)	Paper Ash at 525°C (%)
Control [*] with PCC	560	615.8/	88.4	84.9	61.7	0.84	22.95	2.40	13.19
Control recycled, additional PCC	590	613.2	87.7	84.7	58.5	0.92	21.93	2.16	10.34
	DL [•] /FL°	DL/FL	DL/FL	DL/FL	DL/FL	DL/FL	DL/FL	DL/FL	DL/FL
Initial	620/ 675	647.0/ 733.1	86.6/ 86.9	84.7/ 81.2	55.9/ 47.2	1.12/ 1.30	25.82/ 30.18	2.84/ 3.31	10.71/ 11.58
1st Cycle PCC added to DL	570/ 550	639.2/ 721.3	85.4/ 87.2	85.7/ 80.4	57 . 9/ · 45 . 5	0.85/ 1.62	19.97/ 30.01	2.68/ 4.41	10.21/ 9.13
2nd Cycle PCC added to DL	525/ 420	620.7/ 730.7	85.3/ 87.3	85.6/ 82.3	56.6/ 48.7	0.95/ 1.83	20.22/ 31.84	2.84/ 4.69	10.40/ 9.91
2nd Cycle PCC added to DL Ca $(OH)_2$ and CO ₂ added to FL	510/ 475	614.6/ 633.6	86.1/ 87.2	88.2/ 83.7	66.0/ 57.0	0.76/ 1.01	17.10/ 20.24	2.54/ 3.07	15.37/ 15.52

*Control is never-dried pulp with PCC added, followed by recycling without mechanical treatment.

^bDL is hot dispersed pulp followed by direct loading of PCC with additional PCC added where indicated.

°FL is fiber-loaded pulp with additional chemicals added where indicated.

The never-dried, fiber-loaded pulp under similar refiner conditions, but with calcium hydroxide and without heat and under carbon dioxide pressure, resulted in about the same freeness values when the individual differences in ash content were considered. However, at least a 10% greater apparent density was measured for the handsheets made from fiber-loaded pulps compared with the direct-loaded pulps. This higher apparent density for the fiber-loaded pulp is due to the loading of the denser calcium carbonate within the pulp fibers. The optical properties of the fiber-loaded pulp were somewhat less than the comparable optical properties of handsheets made from pulp recycled using the direct-loaded process. The physical strength properties were from 25% to 50% greater for the handsheets made from the fiber-loaded pulp compared with the handsheets made from direct-loaded pulps.

Additional chemicals were not added to the fiber-loaded pulps for the first two cycles of treatment. The ash content was remarkably constant. This shows that there is a great affinity for the initial PCC to the pulp compared with the control, which dropped in ash content about three percentage points after recycling even with additional calcium carbonate. The fiber-load pulp also retained ash much better than the directloaded pulp, which required additional calcium carbonate addition to mainain ash at 10% to 11%.

Data, which show that physical and optical properties of fiber loaded pulp are maintained without need for additional relining or calcium carbonate addition, suggest that the recirculated process water should be much lower in ash content than that produced by conventional hot dispersion of wastepaper pulps.

For an additional second cycle of fiber loading, an additional amount of calcium hydroxide was added to a portion of the pulp and the fiber was processed under carbon dioxide pressure. The additional PCC resulted in an ash content of 15.52%. Calcium carbonate was added to the direct-loaded pulp after the second cycle, and handsheets were compared with those made from the higher level of fiber-loaded pulp. Again, the same trends were observed as with the fiber-loaded pulp recycled without additional chemicals added. That is, the higher handsheet strength and greater apparent density of the handsheets made from fiber-loaded pulps compared with the direct-loaded pulps suggest that cleaner process water in paper manufacture should be realized.

	Kajaani fiber length, weighted average (mm)	Kajaani fiber length, arithmetic average (mm)	Coarseness (mg/m)
Control ^a , with PCC	0.82	0.62	0.130
Control recycled, additional PCC added	0.75 DL [°] /FL [°]	0.56 DL/FL	0.097 DL/FL
Initial	0.78/0.70	0.59/0.52	0.123/0.166
1st Cycle, PCC added to DL	0.60/0.61	0.42/0.45	0.161/0.218
2nd Cycle, PCC added to DL	0.76/0.67	0.56/0.49	0.111/0.154
2nd Cycle, PCC added to DL Ca(OH) ₂ and CO ₂ added to FL	0.76/0.64	0.57/0.47	0.128/0.127

^aControl is never-dried pulp with PCC added, followed by recycling without mechanical treatment.

^bDL is hot dispersed pulp followed by direct loading of PCC with additional PCC added where indicated.

^c FL is fiber-loaded pulp with additional chemicals added where indicated.

Fiber Properties

Fiber properties corresponding to the control, direct-loaded and fiber-loaded processes (Table II) indicate that fiber loading produced pulp with somewhat shorter fibers than the did the direct loading or control pulp. This can be interpreted as a result of the fines fraction being washed into the process water more readily from the control and direct-loaded pulps than from the fiber-loaded pulp. This would then result in a shorter fiber measurement for the fiber-loaded pulps than for the direct-loaded or control pulps. Coarseness measurements show that the fiber-loaded pulps had a greater weight per length than did the direct-loaded or control pulps. This is interpreted as a result of the high density calcium carbonate being deposited within the lower density pulp fibers. The increased coarseness measurement for fiber-loaded pulps could also indicate flocked fines on the fiber-laded fibers. Precipitated calcium carbonate (PCC) has a natural positive charge, compared with purchased bulk calcium carbonate that has a negative charge as a result of the typically added dispersing agents added for ease in dispersing in water (9). The positively charged calcium carbonate precipitated in the fiberloaded process probably had a greater affinity for the negatively charged fibers than did the direct-loaded purchased calcium carbonate.

Table II. Fiber Properties

Process Water Properties

Corresponding process water for the three pulps (Table III) show that fiber loading resulted in the lowest total suspended solids, total solids, and calcium ion concentration. These results for fiber loading were dramatically reduced, about 50%

reduced with respect to hot dispersion or the control. The fraction of fines less than 0.02 mm and COD values for all three pulp processes showed less variations between pulps.

Suspended solids measured were composed exclusively of calcium carbonate or pulp fines from fiber processing. The process water from the direct-loaded pulp contained more than twice as much suspended solids as did the whitewater from fiber loading, 266 mg/L for direct loading compared with 113 mg/L for fiber loading in initial pulp. As expected, the total suspended solids increased in both direct loading and fiber loading pulp process water after the initial cycle. The increase in suspended solids for the direct-loaded pulps was significantly greater than that observed for the fiberloaded pulps: 440 mg/L for cycle one of direct loading compared with 147 mg/L for fiber loading. Suspended solids was 440 mg/L in cycle two for direct loading and 240 mg/L for fiber loading, which demonstrates that introducing filler by fiber loading can minimize solids in whitewater by 50%. This improved retention accomplished by fiber loading was confirmed by calculating the amount of filler lost between cycle one and two for both processes by measuring handsheet ash content before and after recycling. Fiber loading lost 21% ash compared with 45% ash loss for direct loading on recycling, essentially half the ash loss of direct loading. This was calculated by comparing the amount of calcium carbonate added to the doler tank after revcling with respect to the total amount calcium originally added. Similar results are given for total solids.

Table III. Process Water Analysis

	Total suspended solids (mg/L)	Total solids (mg/L)	Kajaani arithmetic fines (% < 0.200)	Calcium ion concentration (mg/L)	COD (mg/L)
Control ^a , with PCC	205	330	47.2	128	<20
Control recycled, additional PCC	200	340	77.8	153	23
	DL ^b /FL ^c	DL/FL	DL/FL	DL/FL	DL/FL
Initial	266/113	240/190	79.5/74.3	252/89	23/20
1st Cycle,PCC added to DL	440/147	580/300	85.8/80.3	186/102	34/30
2nd Cycle, PCC added to DL	440/240	480/366	62.3/50.9	143/104	38/38
3rd Cycle, PCC added to DL, Ca(OH) ₂ and CO ₂ added to FL	500/246	606/260	76.9/56.4	162/94	50/62

*Control is never-dried pulp with PCC added, followed by recycling without mechanical treatment.

^bDL is hot dispersed pulp followed by direct loading of PCC with additional PCC added where indicated.

"FL is fiber-loaded pulp with additional chemicals added where indicated.

The total solids content in process water includes both suspended and dissolved solids. In this study, the dissolved solids can be attributed primarily to fiber fines as a result of the low water volubility of calcium carbonate, 18 mg/L at 20°C. Ashing the total solids concentrated from the process waters confirmed this. The ash obtained from fiber loading was 91%, 2% less than from direct loading. When converted to ash from fiber in the total solids, fiber loading lost approximately 29 mg/L after two recycles compared with 48 mg/L for direct loading. This study focused on comparative filler retention and effect of fiber reprocessing using both loading methods. Typical process water from a deinking mill increases in filler during flotation deinking, which is reflected in the substantially greater percentage of ash typically contained in deinking sludge than for virgin mills, ranging between 10% to 60%, depending on the filler content of the paper furnish and end product targeted. A report by NCASI placed the average deinking sludge content at 50% to 65% ash (2). High ash content in sludge diminishes its heating value as well as reduces the dewatering rate. Build up of inorganic solids in sludge reduces the efficiency of water clarification in activated sludge plants.

Fiber fines contained in the process waters were measured by Kajaani fiber distribution. The percentage of fines less than 0.2 mm increased for both processes with the first cycle. For fiber loading, fines increased from 74.3% for initially to 80.3% for cycle one. This compares favorably with direct loading in which fines content increased from 79.5% to 85.8% during the first recycling. Both methods displayed decreased fines in cycle two, possibly as a result of the

removal of frees during subsequent repulping and fiber reprocessing or the entrapment of some fines in the fiber web on drying

Whitewater solids are removed from the system as mill sludge. Because deinking mill sludge can be composed of as much as 60% fillers, the fiber loading process has the potential to reduce total mill sludge by 10%. In addition to the problem of quantity of sludge produced, the quality of whitewater is compromised as mills reduce the volume of water used for processing, which concentrates solids. Increased calcium carbonate in recirculating whitewater decreases flotation cell efficiency and causes excessive fiber loss (10,11). Higher carbonate levels require additional polymer for water clarification.

Measurements of COD values indicate the amount of oxygen required to oxidize the organic material in the whitewater which, in this study, is primarily fiber fines from processing. The initial pulp showed 20 mg/L COD for fiber loading, slightly less than the 23 mg/L value obtained for direct loading. The control, which had no fiber processing other than high consistency repulping, shows little increase in COD with recycling, confirming the contribution of fines produced by fiber processing to COD load.

In actual industrial wastepaper deinking mills, sludge is produced at various unit operations. However, our experiments do compare fiber loading with direct loading. In industrial practice, retention aids are used to keep first pass retention high on the papermachine wet end. These retention aids would be used for both fiber loading or conventionally produced wastepaper pulps. Assuming that retention aids would affect both pulps equally, the differential as a result of the processing would remain. By this reasoning, fiber loading would reduce the calcium carbonate in the industrial process water by 50%, as we have observed on our laboratory-scale experiments.

Calcium ion content of process waters measured by atomic absorption again indicates that substantially more calcium was released into whitewater by the direct-loading method than by fiber loading. Whitewater from fiber loading contained 89 mg/L Ca++ compared with 252 mg/L for direct loading for initial pulp. For fiber loading, the calcium concentration increased to 102 and 104 mg for cycles one and two, respectively. Although the calcium level for direct loading decreased in subsequent cycles, the levels of 186 and 143 mg were still greater than for fiber loading.

CONCLUSIONS AND RECOMMENDATION

- Handsheet physical and optical properties of recycled fiber loaded pulps are maintained without need for additional refining or calcium carbonate addition.
- Maintaining recycled handsheet ash levels for fiber-loaded pulps requires only about 50% of the calcium carbonate as that for direct-loaded pulps followed by direct loading.
- Recycled fiber loaded pulps tended to have shorter fiber length than the direct loaded pulps, indicating perhaps greater natural fines retention for fiber loading.
- Process water analysis showed about 50% less total suspended solids, total solids, calcium ion concentration than did the direct-loaded pulps, indicating about 50% less calcium carbonate in the fiber-loaded process.
- Cellulosic fines and COD analysis showed somewhat less cellulose fines in the fiber loading process water than for the direct-loaded pulps.
- Fiber loading is indicated to reduce calcium carbonate filler content of deinking sludge by up to 50%.
- Fiber loading is indicated to reduce deinking sludge by up to 10% overall.
- An industrial-scale fiber loading evaluation is recommended for determining the reduction of sludge generated compared with conventional direct load of calcium carbonate.

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