Effect of fiber loading on paper properties

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ABSTRACT

The authors examined the effect on paper properties of fiber-loaded calcium carbonate compared to conventional, direct-loaded calcium carbonate. Research in this area has developed the basic technology for a process of precipitating and loading calcium carbonate within and on papermaking fibers. Scaleup from handsheet to semicommercial scale revealed some technical obstacles. This study focuses on changes in color and brightness, dimensional stability, retention aid demand, and apparent density. Fiber loading offers a practical processing method to decrease the cost of calcium carbonate production and substitute more filler for fiber in the production of printing and writing papers.

PREVIOUS PUBLICATIONS REPORTED the benefits of processing wood fiber pulp so that inorganic paper-making fillers are forced into the interior of wood pulps *(1–3)*. Recent work reported on fiber loading—a method for manufacturing calcium carbonate during the refining process *(4–6)*. The benefits reported include using carbon dioxide from stack gases as a chemical reactant, extending fiber resource by substituting low-cost filler for more expensive fiber at a higher level than possible with conventional methods, retaining filler during recycling to minimize sludge, and improving brightness and color of the finished paper.

Initially reported experiments were on handsheet scale. Our paper also reports results of handsheet experiments. The results of semicommercial scale trials using fiber-loaded pulp prompted our trials, however. Semicommercial trials often reveal aspects of proposed industrial technology not shown in handsheet experiments.

We conducted fiber-loading experiments on industrial-scale equipment simultaneously using an atmospheric, high-consistency refiner as a mixer and refiner followed by high-consistency pressurized refining under carbon dioxide pressure. The pulp was processed at the Sunds Defibrator stock preparation pilot plant in Sundsvall, Sweden. After the fiber loading, the pulp was processed on a pilot paper machine at the University of Manchester Institute of Science and Technology (UMIST) with a conventional direct-loaded calcium carbonate control.

Results of these experiments showed that fiber loading could be accomplished without process problems using conventional stock preparation equipment. The resulting fiber-loaded pulp could also be processed into paper using conventional papermaking techniques. We did observe differences in the finished paper, however, with respect to the control. This prompted our investigation reported here.

We observed a slightly more yellow and less bright paper than the control. We also noted that the sheet lost about 50 mm on a 635-mm width compared to the control. The fiber-loaded sheet lost a few percentage points in filler retention compared with the control. We also observed an approximate 10% loss in apparent density with the fiber-loaded paper as we had also noted in the handsheet studies.

Conditions of the semicommercial trials were duplicated in the laboratory on handsheet scale to explain the problems and develop methods for overcoming the differences between paper made with fiber-loaded pulp and that made with conventional, direct-loaded pulp.

We report results on the following:

- Color and brightness
- Dimensional stability
- Filler retention
- Apparent density.

EXPERIMENTAL PROCEDURE

Materials

Bleached, hardwood kraft, wet-lap pulps came from Potlatch Corp. and MoDo Cellkraft AB. The Potlatch pulp was mainly aspen and the MoDo pulp mainly birch. The Potlatch pulp was a fully bleached kraft pulp. Bleach sequence was essentially chlorine free with the bleached pulp essentially lignin free. The MoDo pulp was bleached by a totally chlorine free (TCF) method that resulted in some residual lignin. The pulps were refined when needed in a disc refiner under atmospheric conditions at the USDA Forest Service, Forest Products Laboratory, Madison, WI, for subsequent fiber-loading experiments. Reagent grade calcium hydroxide used for most experimental work was from Aldrich Chemical Co. Papermaker-quality precipitated calcium carbonate obtained from Speciality Minerals was used for the direct-loading experiments.

A commercial, dual polymer retention aid treatment evaluated

Sample	TCF PULP Brightness, %	TCF PULP b* value	ECF PULP Brightness, %	ECF PULP b* value
Initial wet lap	86.9	3.84	87.1	3.44
Pulp with calcium hydroxide	80.6	5.95	82.7	4.63
Carbon dioxide reacted pulp/ calcium hydroxide after 1 day	85.9	4.03	87.1	3.5
Carbon dioxide reacted pulp/ calcium hydroxide after 3 days	86.9	4.06	88.4	3.31
Pulp with calcium hydroxide plus hydrogen peroxide	86.2	4.17		
Carbon dioxide reacted pulp/ calcium hydroxide and hydrogen peroxide	88.3	3.31		

(b* value is blue-yellow.)

I. Optical properties of totally chlorine free and elemental chlorine free pulps

the effect of filler type on retention. A high molecular weight anionic flocculating agent—Nalco 625—with a low molecular weight cationic retention aid—Nalco 7607—were combined for filler retention. A commercial cationic potato starch was used as an additive for restoring strength to handsheets as indicated.

Consistency, %	Calcium hydroxide, %	Freeness (CSF), mL	Dimensional stability (% change at 50–90% RH)
15	30	290	0.35
15	30	330	0.42
25	30	360	0.70

II. Dimensional stability of fiber-loaded handsheets prepared from prerefined pulp

Equipment

This study used a bench model three-speed Hobart food mixer with a 76-L stainless steel bowl and flat beater. A Sprout Bauer 305-mm pressurized disc refiner was both the reaction chamber and refiner for precipitating calcium carbonate and incorporating it into pulp fibers. Sprout Bauer refiner plates with patterns C-2975-C and 2975-1C (Devil's teeth) were used in this study.

Test methods

Fiber-loading procedure. To incorporate calcium carbonate, pulp was blended in a Hobart mixer with varying amounts of calcium reactant and water required for the desired chemical load and consistency. The pulp mixed for 15 min at low speed to incorporate uniformly the calcium compound. Alternately, the pulp, water, and calcium blend passed through the Sprout Bauer refiner at atmospheric conditions using the refining plates to mix and refine simultaneously.

Precipitation of carbonate. High-consistency pulp containing calcium reactant was loaded into the refiner feed tank. Carbon dioxide injected into the feed tank reacted with the calcium hydroxide in the pulp. Carbon dioxide was held in the tank for 15 min at 138 kPa. During this interval, calcium carbonate was precipitated in and on the pulp fibers. The pulp was then dispersed in a carbon dioxide atmosphere at the desired plate gap and feed rate to provide intimate contact of the carbonate and fibers.

Direct-loading procedure.. An appropriately excessive amount of reagent grade calcium carbonate was added directly into the low-consistency pulp slurry in the doler tank during handsheet preparation to match the paper ash level of fiber-loaded handsheets. Adequate mixing in the doler tank ensured uniform distribution of the carbonate before making handsheets.

Pulp and paper tests. Handsheets (60 g/m^2) were prepared according to TAPPI T 205 om-88. Ash content of handsheets was assessed by TAPPI T 211 om-93. Retention levels of filler and total fines were determined using a Britt Drainage Jar. Burst was measured according to TAPPI T 403 om-91. Sheet density was determined according to TAPPI

III. Dimensional stability of fiber-loaded handsheets pulp mixed and refined together

Sample	Freeness (CSF), mL	Dimensional stability (to change)
15% Consistency	200	0.43
30% Calcium hydroxide	320	0.38
25% Consistency	360	0.44
30% Calcium hydroxide	120	0.64

T 220 om-88. Tear and tensile indices were measured according to TAPPI T 414 om-88 and T 494 om-88, respectively. Optical properties were evaluated on a Technidyne photometer using TAPPI T 525 om-92 for brightness and scattering coefficient. Dimensional stability was measured using the apparatus described by Gunderson *(7)*. Relative humidity varied from 50–90%, and the percentage change in length was reported after 1500 s.

RESULTS AND DISCUSSION

Color and brightness

An unexpected yellowing resulted on the fiber-loaded paper made at UMIST. The fiber-loaded paper was two brightness points less than the conventionally loaded control paper—85% compared to 87% brightness. Additionally, the fiber-loaded paper had a higher b* (blue-yellow) value. This contributed an ivory color to this paper compared with the control. This was the first time we observed a loss of brightness with the fiber-loading process. Therefore, we conducted a series of handsheet experiments to duplicate these results and isolate the factor responsible for the yellowing.

All our previous experiments used fully bleached hardwood kraft pulps. The bleach sequence was elemental chlorine free (ECF), and the bleached pulp was essentially lignin free. The pulp used for the paper-machine trials at UMIST was bleached by a TCF method that left some residual lignin. We hypothesized that the small amount of residual lignin (1.3% by Klason lignin) caused alkaline darkening of the pulp when exposed to the high alkalinity of the calcium hydroxide addition for fiber loading.

For our experiments, we used two bleached pulps: the TCF pulp from the UMIST trials and an ECF pulp that we had used previously for our fiber-loading experiments. **Table I** shows that the initial brightness and b* values for both pulps were almost identical. When calcium hydroxide was incorporated into both pulps, however, the TCF pulp displayed a larger change in both brightness and b* value than did the ECF pulp.

Because there could be a delay in reacting the calcium hydroxide loaded pulp with carbon dioxide, we examined the effect on brightness of various retention times before conversion to calcium carbonate in the fiber-loading process. Table I illustrates that holding the pulp for three days after adding the calcium hydroxide did not substantially change the brightness or b* values of the resulting carbonate-loaded pulps. We did find, however, that the fiber-loaded TCF pulp produced a slightly lower brightness and slightly greater b* value than did the fiber-loaded ECF control pulp.

To eliminate this brightness loss, we included 1% hydrogen peroxide (based on the dry weight of the pulp) with the calcium hydroxide added on the TCF pulp. The peroxide addition successfully prevented the brightness loss experienced previously. This confirmed our speculation that the brightness loss and yellowing of the pulp were probably due to residual lignin reacting with the alkaline calcium hydroxide. Future yellowing problems, if encountered, could be prevented by adding hydrogen peroxide when using TCF bleached pulps for fiber loading.

Dimensional stability

We observed about a 25-mm width shrinkage for the 635-mm wide fiber-loaded paper web compared with the conventional paper web made on the pilot paper machine. We investigated this phenomenon with a handsheet study of the dimensional stability variables of temperature and relative humidity. We compared handsheets from two different fiber-loading process techniques: fiber-loading pulp that had been conventionally refined at low consistency and combining the mixing and refining steps into one.

Table II lists the results of processing conditions on pulps refined at conventional low consistencies. Each sample had two passes in a refiner. The first pass was without carbon dioxide, and the second pass was with carbon dioxide as reactant. We first investigated the effect of the level of calcium hydroxide loading. At 15% fiber consistency, comparing 10% calcium hydroxide loading to 30% did not show much effect on dimensional stability. The values were 0.35% for the 10% loading and 0.42% for the 30% loading. A comparison of 15% consistency to 25% consistency both at 30% calcium hydroxide loading resulted in an increase in dimensional change from 0.42% to 0.70%. We consider this change significant. It indicates a potential dimensional stability problem in the papermaking process using fiber-loaded pulp.

Similar experiments were conducted for fiber loading when mixing and refining were combined in the same step. **Table III** summarizes the results of these experiments. In this table, each sample had two

Sample	FIBER-LOADED, % Total retention	Ash	Ash retention	DIRECT-LOADED, % Total retention	Ash	Ash retention
0 kg/ton* retention aid	70.4	25.3	91.1	61.2	22.2	83.3
0.5 kg/ton* retention aid	75.0	25.6	92.0	79.6	23.7	88.8
1.0 kg/ton* retention aid	84.2	24.6	88.4	92.3	23.1	86.4
2.0 kg/ton* retention aid	87.2	25.4	91.3	94.9	23.6	88.4
Direct-loaded pulp					26.7	
Fiber-loaded pulp		27.8				
Washed direct-loaded pulp					0.2	
Washed fiber-loaded pulp		6.1				

*1.0 kg/ton of flocculating agent

IV. Dynamic drainage jar retention

KEYWORDS

Brightness, calcium carbonate, calcium compounds, carbonates, color reversion, density, dimensional stability, fibers, filler addition, filler retention, mechanical properties, optical properties, paper properties, physical properties, problem solving, properties, retention, reversion, stability, yellowing.

passes in the refiner. The first pass combined mixing and refining and had no carbon dioxide. The second pass used carbon dioxide for reaction. When comparing dimensional stability for two experiments done at the same process conditions of 15% consistency and 30% calcium hydroxide load, we obtained comparable dimensional stability results of 0.43% and 0.38%, respectively.

Changing the consistency to 25% from 15% and holding the other conditions approximately the same resulted in a dimensional stability almost the same at 0.44%. Increasing the refining at the same conditions of 25% consistency and 30% calcium hydroxide load, however, resulted in an increase in dimensional change to 0.64%. This value is unacceptable for papermaking.

To summarize, a very low freeness or high consistency and high calcium hydroxide loading can result in unacceptable levels of dimensional change in the resulting handsheets when using either separate or combined mixing and refining. For industrial practice, specific pulp and processing equipment must be evaluated to ensure satisfactory operating conditions.

Filler retention

During the pilot paper-machine trials, we observed a slight reduction in filler retention for the fiber-loaded trial compared with the conventionally loaded trial. We reproduced these results on a laboratory scale using a Britt Jar to measure both ash and total retention.

Table IV lists the results of comparing fiber-loaded pulp with conventional direct-loaded pulps for retention. We used a commercially accepted two-step retention system. For both the fiber-loaded pulp and the control, we kept the high molecular weight anionic flocculating agent at a constant 1.0 kg/ton. The low molecular weight cationic retention aid varied from 0–2.0 kg/ton. Results demonstrated that the direct-loaded furnish gave the highest results for total retention of inorganic filler (ash) and pulp fines. For example, at 2.0 kg/ton, fiber-loaded pulp gave 94.9% retention compared with 87.2% for the fiber-loaded furnish.

For filler only retention, the results were reversed. For example, at 2.0 kg/ton retention aid dosage, the filler retention for fiber loading was 91.3% compared with 88.4% for the direct-loaded pulp. Both the fiber-loaded and direct-loaded pulps responded favorably to a commercial two-step retention aid treatment, however. The differences are well within the control of the papermaker.

Apparent density

Table V lists the results for wet pressure compared with density for fiber-loaded handsheets. Reducing the wet-press pressure to 138 kPa from the conventional 345 kPa resulted in an apparent density of 673.8 kg/m^3. This is almost the same as the 662.3 kg/m^3 obtained for a similar direct-loaded handsheet at 345 kPa.

Adding starch to the furnish improved the paper strength. At 4 kg/ton of starch, the burst strength increased to 1.56 kPa/m^3. This is approximately the same as the direct-loaded control.

These experiments illustrate that reducing wet-press pressure at a

	Wet-press pressure, kPa	Starch addition, kg/ton	Paper ash, %	Density, kg/m3	Burst index, kPa•m2/g
Direct-loaded control	230		17.7	662	1.12
Fiber-loaded handsheets	230		17.4	774	1.61
	184		17.2	729	1.62
	138		16.8	704	1.56
	92		16.5	674	1.56
	92	1.0	20.1	677	1.41
	92	2.0	18.9	672	1.47
	92	3.0	20.5	674	1.49
	92	4.0	20.9	686	1.56

V. Effects of wet pressing and wet-end starch addition to fiber-loaded handsheets

common ash content can decrease apparent density of the resulting handsheet. Adding a wet-end starch can increase handsheet strength at a common ash content. Unfortunately, the ash content for the wet-press pressures was somewhat less than the ash content for the wet-end starch experiments. The principle of how apparent density can be compensated by reducing wet pressing and adding wet-end starch is shown, however.

When recycling wastepaper, the density of the resulting paper is usually less than the original paper made from never-dried pulp *(8)*. Fiber loading offsets this by increasing apparent density. This indicates that fiber loading may be useful for controlling the apparent density for recycling wastepaper.

CONCLUSIONS

Using the results of our work, we offer the following conclusions:

- Brightness loss and yellowing that occurred in the pilot paper machine trials were traced to residual lignin in TCF bleached pulps. We demonstrated that including 1% hydrogen peroxide in the fiber-loading process prevented brightness loss and yellowing of the fiber-loaded pulp.
- Dimensional stability depends on the fiber-loading process conditions. Good dimensional stability results from either loading prerefined pulp or simultaneously refining and loading. Refining to very low freeness, adding high levels of calcium hydroxide, or processing at very high consistencies can cause unacceptable dimensional sheet shrinkage, however.
- Direct-loaded and fiber-loaded pulps showed small differences in retention when subjected to identical dual polymer retention aid treatments. Direct-loaded pulp gave slightly greater total retention. Fiber-loaded pulps gave slightly greater ash retention. The differences are well within the control of the papermaker.
- Apparent density decreased by approximately 10% by fiber loading. Decreasing the wet-web pressing pressure during handsheet preparation can offset this loss. Adding wet-end starch can regain the strength loss associated with decreased wet pressing. **TJ**

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