

lightweight, high-opacity paper by fiber loading: filler comparison

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SUMMARY: To compare the effects of commercial calcium carbonate fillers on handsheet properties with that of fiber loading, we obtained rhombohedral and scalendrohedral precipitated calcium carbonates (PCCs) and wet and dry ground calcium carbonates (GCCs). These commercial calcium carbonate fillers were selected to cover a wide range of particle diameter and shape. The properties of fiber-loaded handsheets, at 25% filler level, were compared to that of five commercial calcium carbonate samples at the same filler level for two fully bleached market pulps, eucalyptus and pine kraft pulps. Fiber-loaded pulps were shown to be stronger than similar direct-loaded pulps at the same calcium carbonate levels as a result of (1) precipitation of calcium carbonate within the fiber wall and lumen and (2) the low energy input refining of the pulp at high pH and consistency. Our results show that fiber loading results in somewhat higher freeness values than does direct loading along with higher strength. Brightness values were slightly lower or the same for fiber-loaded handsheets compared to direct-loaded handsheets. Opacity and scattering coefficients were slightly lower for fiber-loaded handsheets, but the higher strengths of fiber-loaded pulps permit an increase in the filler level in the fiber-loaded pulps. Production of fiber-loaded PCC (FLPCC) pulp can be a key factor in developing lightweight, high-opacity printing papers.

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a Methods for incorporating fillers within pulp fibers have been the subject of extensive research (Green et al. 1982; Scallan and Middleton 1985; Allen and Ritzenthaler 1992). The initial goal of this research was to incorporate filler within fiber in order to produce high strength fiber at high ash content. A later goal was to substitute low-cost calcium carbonate for high-cost fiber using fiber loading (Klungness et al. 1993). We now propose that the production of fiber-loaded precipitated calcium carbonate (FLPCC) pulp can be a key factor in producing lightweight, high-opacity printing papers and in increasing the return of investment for the paper industry (Klungness et al. 1999).

Fiber loading, which was developed at the Forest Products Laboratory, consists of two steps (Klungness et al. 1993). First, calcium hydroxide slurry is mixed into moist pulp. Second, the pulp and calcium hydroxide mixture is reacted using a high-consistency pressurized reactor (refiner or disk disperser) under carbon dioxide pressure to precipitate calcium carbonate. The FLPCC is deposited within, on the surface of, and outside the pulp fibres in this heterogeneous reaction medium.

Fibre loaded pulps are stronger than similar direct-loaded pulps at the same calcium carbonate levels. This increase in strength is the result of two independent mechanisms: (a) deposit of PCC within the fiber wall and lumen (Klungness et al. 1994) and (b) low energy input refining

at high pH and consistency (Klungness et al. 1998; Sykes et al. 1998). The low energy (approximately 6.84×10^4 kJ/metric ton) needed for the reaction process during fiber loading causes a small increase in pulp Canadian standard freeness (CSF) (Heise et al. 1996; Klungness et al. 1998). Measurement of fiber length, kink, curl, and fines (<0.2 mm) content after fiber loading reveals no change in fiber characteristics (Sykes et al. 1997). By allowing fiber bonding without any significant change in freeness, fiber loading could be a key factor in producing lightweight, high-opacity paper.

One question that arises in comparing the paper properties of handsheets produced by fiber loading to the properties of commercial direct-loaded handsheets is the role of the size and morphology of the commercial calcium carbonate fillers (Fairchild 1992, Fairchild and Clark 1996, Brown 1998). That is, by changing the size and morphology of the fillers, can the properties of the commercial calcium carbonates develop better handsheet paper properties than does FLPCC?

To investigate the role of commercial calcium carbonate fillers in the development of handsheet properties, we obtained rhombohedral and scalendrohedral PCCs as well as both wet and dry ground calcium carbonates (GCCs). A wide range of particle sizes was selected for the fillers. Properties of FLPCC handsheets at 25% filler level were compared to those of five commercial calcium carbonate samples selected from two fully bleached and refined eucalyptus and northern pine kraft commercial pulps.

Materials and methods

Materials

Fully bleached eucalyptus and northern pine kraft commercial pulps were used for this study. Commercial calcium carbonate fillers were obtained from suppliers and tested for both particle size and shape. Particle size analysis was conducted with a Horiba LA-910 laser light-scattering particle size analyzer for the commercial calcium carbonates. Scanning electron photomicrographs were obtained for the commercial fillers and the fiber-loaded calcium carbonate. The results are shown in Figs. 1 to 3. The dual polymer retention aids used were cationic (polyacrylamide, BMB 2410) and anionic (colloidal silica, BMA-780) materials obtained from Eka Chemicals, Inc. (Marietta, GA). Mississippi Codex Hydrated Lime (mean particle diameter $\sim 2.9 \mu\text{m}$) industrial-grade calcium hydroxide was used for the fiber-loading process.

Equipment

Low consistency refining was performed with a 305-mm atmospheric double-disk Sprout Waldron refiner. The fi-

Ober-loading equipment was a 305-mm-diameter pressurized disk refiner manufactured by Sprout Bauer (Springfield, OH) using Pattern D 2B505 refiner plates.

Methods

Both the eucalyptus and pine kraft pulps were refined at a low (~3%) consistency level to about 350 mL CSF. After the pulp was dewatered, it was shredded in a laboratory shredder. For fiber loading, 500 g (oven-dry basis) pulp was used for each batch. A slurry of calcium hydroxide and water was prepared and added to the pulp in a Hobart-style dough mixer. The consistency of the pulp was adjusted to 21%. After the pulp slurry was mixed for about 30 min, it was placed in a polyethylene bag and sealed for later use in the fiber-loading process.

The refiner plate gap was adjusted to 0.7 mm. Pulp and calcium hydroxide were removed from the sealed bag and placed in the refiner holding tank. The holding tank was then pressurized with carbon dioxide to 207 kPa. The pulp was held in the holding tank for about 3 min before screw-conveyed to the refiner zone and passed through to the pressurized receiver tank. About 6.8×10^4 kJ/metric ton of energy was used. The refining and holding time of the pulp in the receiver tank was 12 min. The total time allowed for fiber loading was 15 min.

For handsheet preparation, the consistency of the fiber-loaded pulp was measured and diluted with the needed water in a doler tank. The concentration of fiber-loaded pulp or the pulp being evaluated was adjusted to result in a 1.5-g (75-g/m²) handsheet from 790 mL slurry. This grammage was selected because it is commonly used for copy paper. A 790-mL doler of slurry was taken from the doler tank and the cationic polymer (polyacrylamide) was added, followed by the anionic (colloidal silica) polymer. The dosage of the retention aids was adjusted to give final ash content of 25% ($\pm 1\%$) as calcium carbonate. The mixture was then placed in a handsheet former and mixed for uniformity before being drained and formed into handsheets. For direct loading, the refined pulp was added to the doler tank as described for fiber loading, and the calcium carbonate being evaluated was added as needed. The remainder of the direct-loaded handsheet preparation, in-

Table 1. Effect of 25% addition of calcium carbonate fillers to bleached eucalyptus and pine kraft pulps.

Pulp	CSF (mL)	
	Eucalyptus	Pine
Initial pulp	325	340
Fiber-loaded pulp	460	440
Direct-loaded pulp ^a		
Rhombohedral PCC 1.42 μ m diameter	410	670
Scalendohedral PCC 0.97 μ m diameter	400	660
Scalendohedral PCC 1.28 μ m diameter	400	650
Dry GCC 0.47 μ m diameter	420	670
Wet GCC 1.54 μ m diameter	430	700

^aDirect-loaded pine pulp was not refined (730 mL CSF).

cluding the addition of retention aids, was identical to that for the fiber-loaded handsheets.

The following test methods were used for handsheet testing: Scott bond, Tappi T833; tensile index, Tappi T494; tensile energy absorption (TEA), Tappi T494; taber stiffness, Tappi T489; tear index, Tappi T414; diffuse brightness, Tappi T525; color (yellowing) of paper and board, Tappi T527; diffuse opacity of paper, Tappi T519; and diffuse scattering coefficient, Tappi 519.

Results and discussion

Fiber loading of refined eucalyptus kraft pulp to 25% calcium carbonate increased CSF from 325 to 460 mL (Table 1). For each type of PCC and GCC, direct loading of refi-

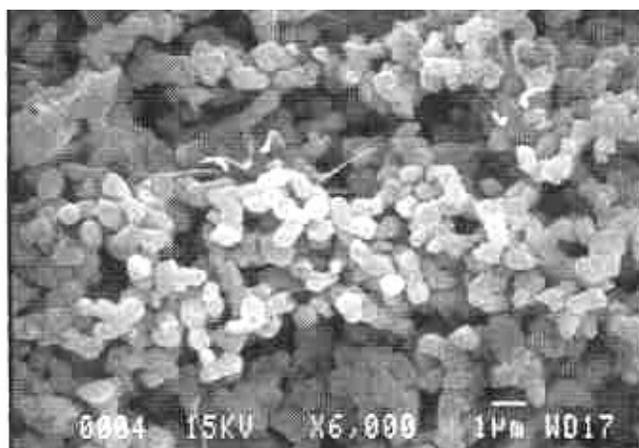
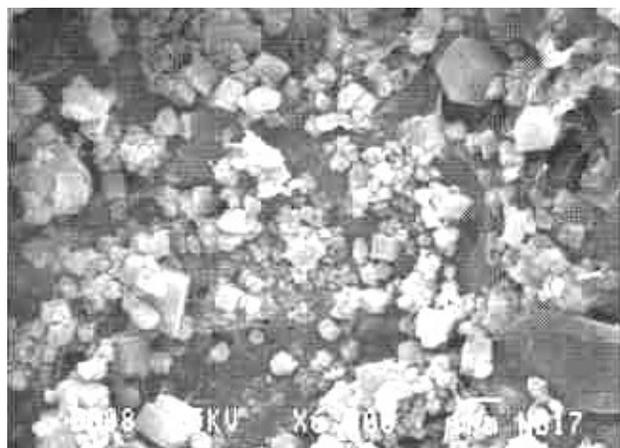
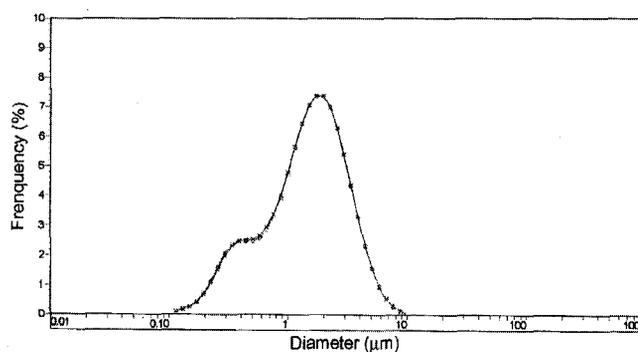


Fig. 1. Scanning electron microphotographs of fiber-loaded PCC (left) and rhombohedral PCC (right). Graph shows rhombohedral particle size analysis; mean particle diameter was 1.42 μ m.

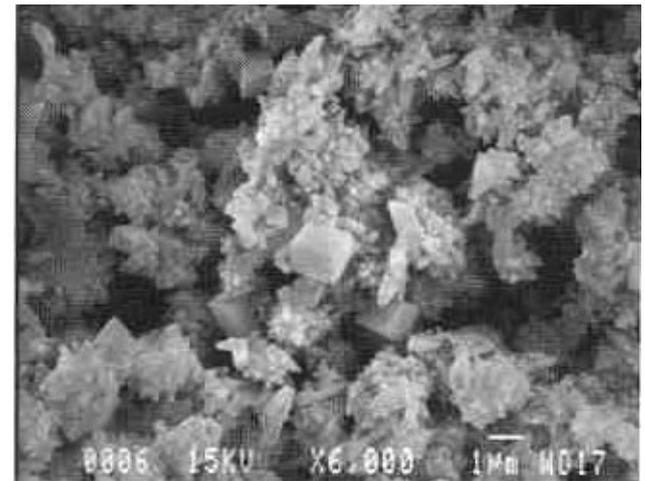
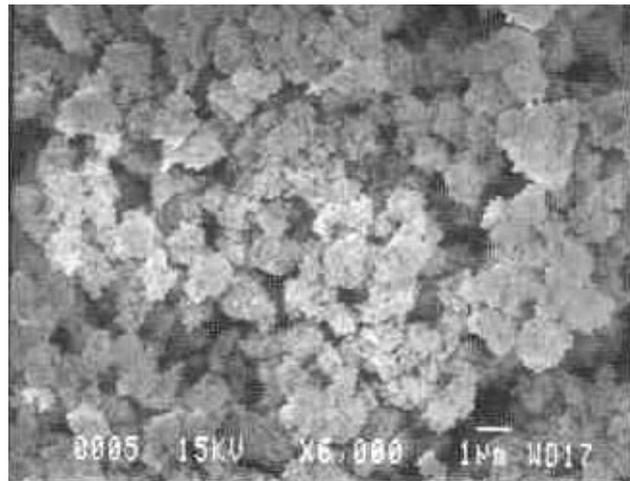
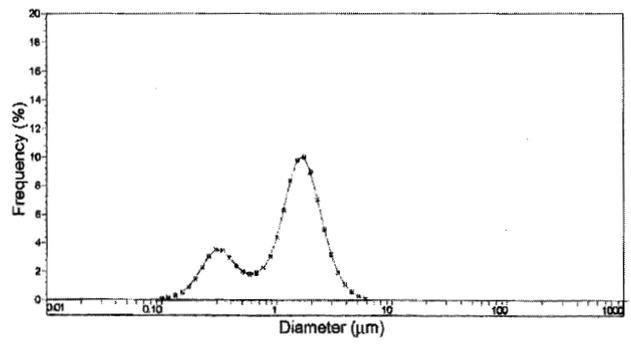
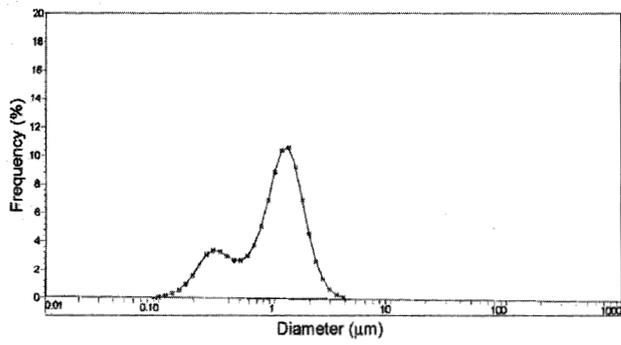


Fig. 2 Scanning electron microphotographs of scalenohedral PCC. Graphs show scalenohedral particle size analysis for the two particle sizes: (left) 0.97 μm , (right) 1.28 μm .

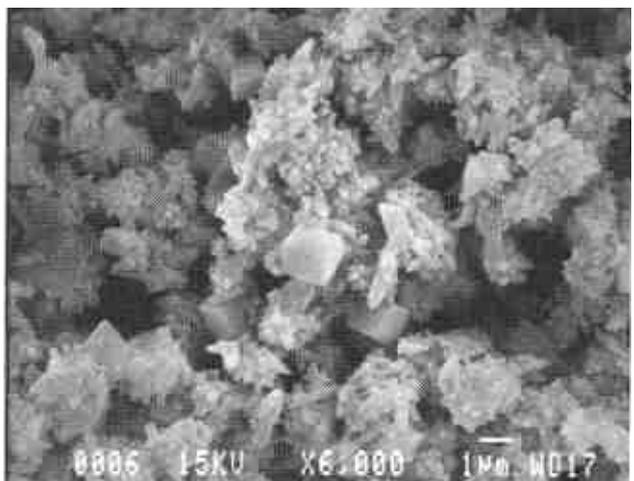
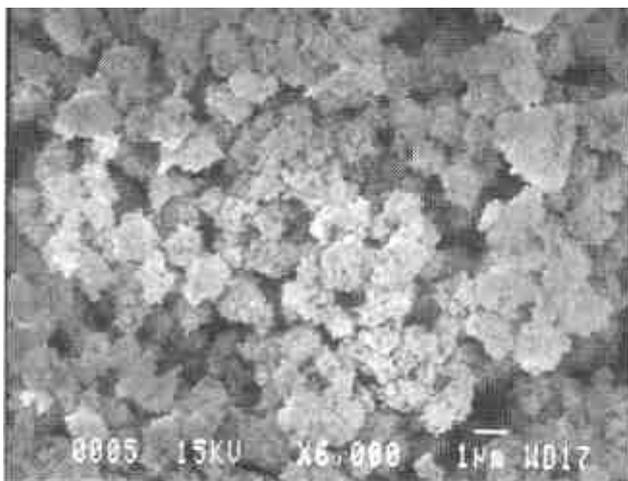
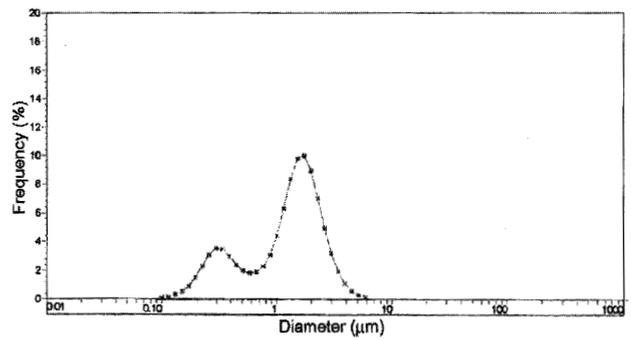
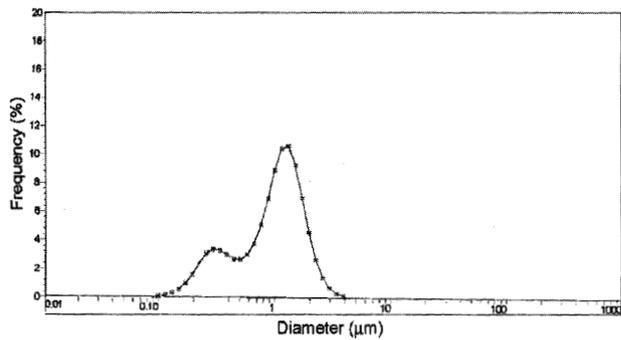


Fig. 3. Scanning electron microphotographs of wet (left) and dry (right) GCC. Graphs show GCC particle size analysis for two particle sizes: (left) 0.47 μm , (right) 1.54 μm .

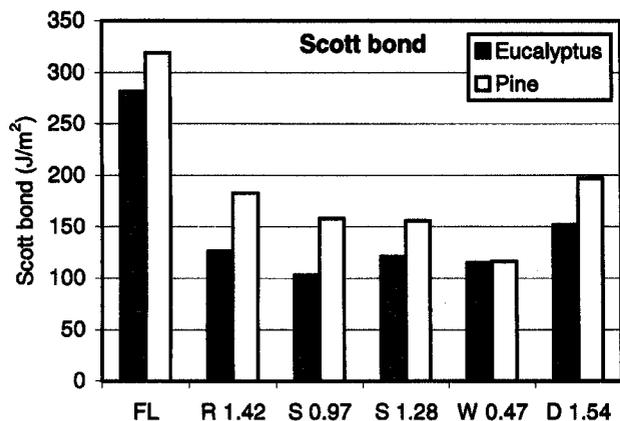


Fig. 4. Scott bond values for handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers: FL is fiber loaded; R, rhombohedral PCC, 1.42 μm ; S, scalenohedral PCC, 0.97 and 1.28 μm ; W, wet GCC, 0.47 μm ; and D, dry GCC, 1.54 μm .

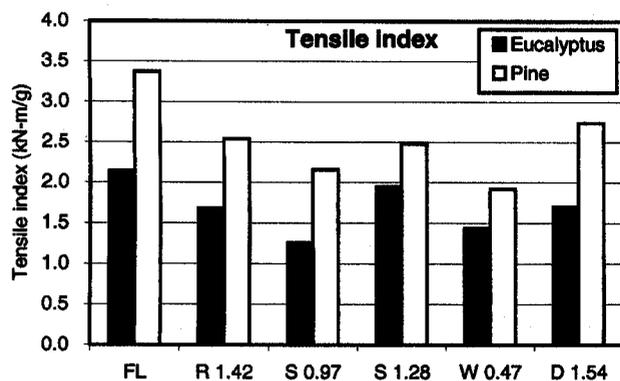


Fig. 5. Tensile index of handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

ned pulp resulted in somewhat lower CSF values, ranging from 400 to 430 mL. The higher CSF value for fiber loading with respect to direct loading suggests an advantage for fiber loading in regard to drainage and pressing in paper manufacture.

Fiber loading of pine kraft pulp increased CSF from 340 to 440 mL. The effect of direct loading of pine kraft pulp was compared to that of unrefined pulp. Direct loading of 25% calcium carbonate to the unrefined pulp reduced CSF from 730 to 650–700 mL.

Microphotographs with corresponding particle size analysis are shown in Figs. 1 to 3 for the calcium carbonates investigated. These figures show a wide range of morphology and particle sizes. Scott bond values of 285 and 315 J/m^2 were measured for fiber-loaded eucalyptus and pine kraft pulp handsheets, showing nearly a 100% increase with respect to the values measured for the direct-loaded handsheets (Fig. 4). These results clearly show that fiber loading develops greater internal bonding strength of paper than does direct loading of any of the fillers investigated. These results were expected because the filler for fiber loading is partially deposited within the fiber and thus does not interfere with internal bonding. All the direct-loaded handsheets exhibited nearly the same internal bonding strength, indicating that the morphology and particle size of the PCC or GCC fillers played a small role in developing internal bonding strength.

Tensile index values are given in Fig. 5. Fiber loading

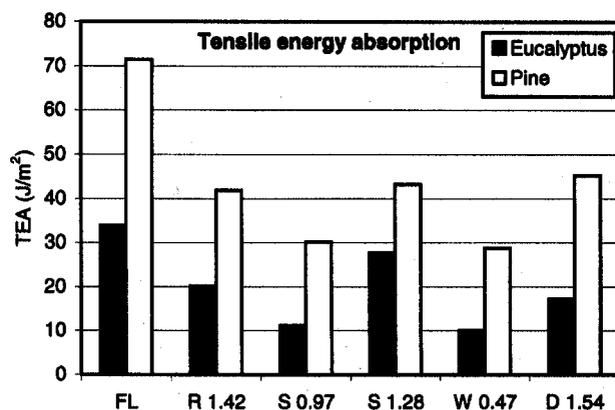


Fig. 6. Total energy absorption (TEA) of handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

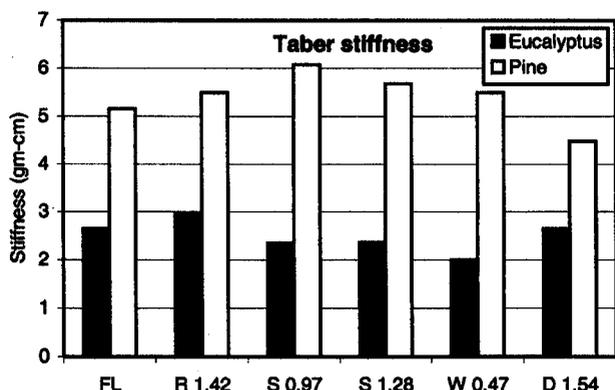


Fig. 7. Taber stiffness of handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

of handsheets from both eucalyptus and pine kraft pulps resulted in greater tensile index values compared to that of handsheets from direct-loaded pulps. This is an indication of stronger fiber-to-fiber bonding, most likely due to less interference to interfiber bonding from filler deposited within the fibers. The tensile index values for fiber-loaded pulp were about 10% to 40% higher than those of direct-loaded pulp depending on which type of pulp or filler was compared. No trend in the data for direct loading or pulp type was noted.

The ability of paper to absorb energy (toughness) is indicated by total energy absorbed (TEA). The TEA values we obtained are given in Fig. 6. The TEA of handsheets from fiber-loaded pulps was 15% to nearly 70% greater than that of handsheets with direct-loaded fillers, indicating a greater toughness and durability for paper products made from fiber-loaded pulps.

Taber stiffness values are compared in Fig. 7. Fiber loading resulted in higher values for eucalyptus pulp compared to those of direct-loaded pulp, except for rhombohedral PCC at a particle size of 1.42 μm diameter. This filler resulted in slightly higher Taber stiffness than that of the fiber-loaded handsheets. Taber stiffness values of handsheets made from direct-loaded pine kraft pulp were all slightly higher than those of fiber-loaded handsheets, except for the handsheet filled with dry GCC. For fiber-loaded handsheets, opposing factors of reduced bulk and increased fiber bonding apparently resulted in similar

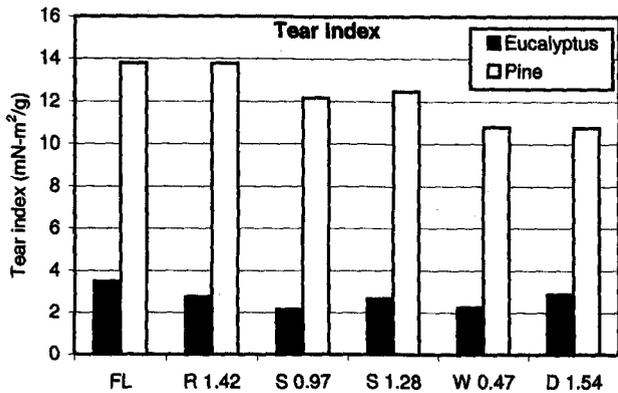


Fig. 8. Tear index values for handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

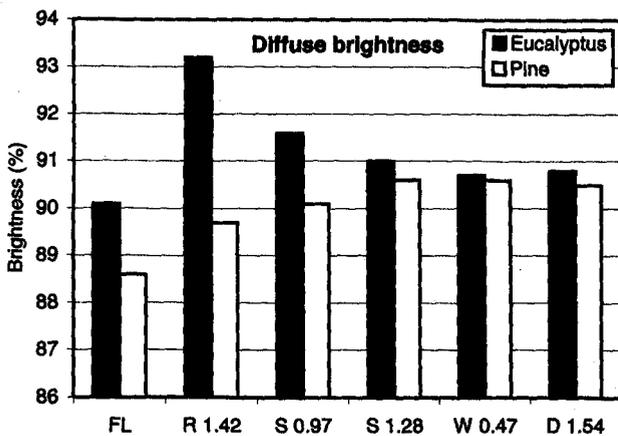


Fig. 9. Diffuse brightness of handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

values for Taber stiffness.

Fiber loading resulted in the greatest tear index values for handsheets made from either eucalyptus or pine kraft pulp (Fig. 8). Differences between fiber loading and direct loading were small. Tear index depends to a great extent on the fiber length of the pulps. Because the addition of filler does not change fiber length, no great differences were noted between the types of pulp loading.

Handsheet brightness as measured by diffuse brightness is shown in Fig. 9. Handsheets made from fiber-loaded eucalyptus and pine kraft pulps were slightly less bright than those made from direct-loaded pulps. This was apparently due to the alkalinity of the calcium hydroxide used for fiber loading. It is interesting to observe that the diffe-

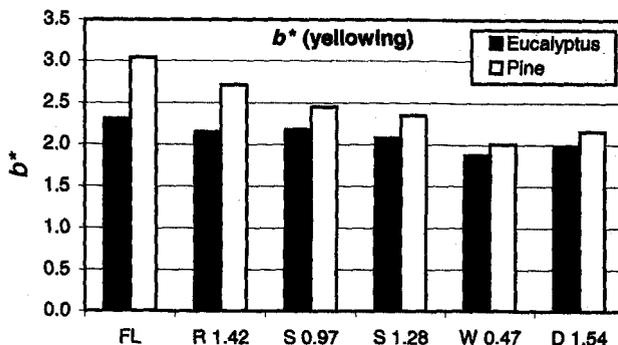


Fig. 10. Yellowing (b^*) values for handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

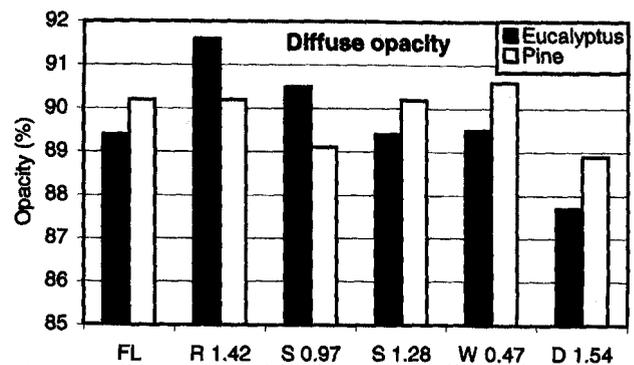


Fig. 11. Diffuse opacity of handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

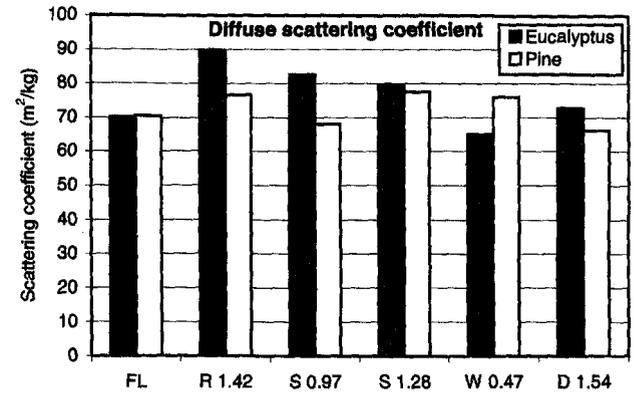


Fig. 12. Diffuse scattering coefficients of handsheets from bleached eucalyptus and pine kraft pulps with 25% PCC or GCC fillers. See Fig. 4 for definition of abbreviations.

rence in handsheet brightness was slightly less for eucalyptus pulp compared to pine kraft pulp. This may be an indication of less residual lignin in the eucalyptus pulp. A potential method for increasing brightness, if needed, is to add a small amount of hydrogen peroxide to the fiber-loading reactor.

The color of the handsheets was determined by b^* values, which are given in Fig. 10. Fiber-loaded handsheets had slightly higher b^* values (i.e., were more yellow) than direct-loaded handsheets. As with brightness, the differences in b^* values were small and were lower for the eucalyptus pulps, which probably contained less residual lignin. Alkaline darkening of the residual lignin in the pulps apparently causes a slight yellowing of the handsheets prepared from fiber-loaded pulp. The addition of a small amount of hydrogen peroxide to the fiber-loading process is suggested as a potential method for reducing yellowing.

Opacity values are given in Fig. 11 and scattering coefficient values in Fig. 12. For both properties, values for fiber-loaded handsheets were in the middle range of all values measured, for both eucalyptus and pine kraft pulps. Because of the strength advantage of fiber-loaded pulps, increased levels of filler can be used to offset any loss in opacity or scattering coefficient. The increased strength due to fiber loading could be used in paper manufacturing for reducing grammage, increasing filler content, or both.

Conclusions

The handsheets made from fiber-loaded pulps are stronger than those from similar direct-loaded pulps at the same cal-

cium carbonate levels as a result of the carbonate within the fiber wall and lumen and the low energy input refining of the pulp at high pH and consistency. The results show that fiber loading causes somewhat higher freeness values than does direct loading, along with an increase in strength. Brightness values were slightly lower or the same for handsheets made from fiber-loaded pulps compared to those from direct-loaded pulps. Opacity and scattering coefficients were slightly lower for fiber-loaded handsheets, but this can be easily offset by increasing the filler level in the fiber-loaded pulps. By enhancing fiber bonding with a slight increase in freeness, fiber loading could be a means for producing lightweight, high-opacity paper. W

Literature

- Allen, G.G., and Ritzenthaler, P.** (1992): The microporosity of pulp, *Tappi J.* 75(3), 239.
- Brown, R.** (1998): Particle size and structure of paper fillers and their effect on paper properties, *Paper Technol.* 29(2), 44.
- Falchild, G.H.** (1992): Increasing the filler content of PCC filled alkaline filled papers, *Tappi J.* 75(8), 85.
- Falchild, G.H. and Clark, E.B.** (1996): PCC morphology and particle size effects in alkaline paper, In: 1996 50th Appita Annual General Conference Proceedings, vol. 2, Carlton, Victoria, Australia. New Generation Print & Copy, Brunswick East, Victoria, Australia, pp. 427–433.
- Green, H.V., Fox, T.J., and Scallan, A.M.** (1982): Lumen-loaded pulp, *Pulp Paper Can.* 83(7): T203.
- Helse, O., Finneran, W., Klungness, J., Tan, F., Sykes, M., AbuBakr, S., and Eisenwasser, J.** (1996): Industrial scale-up of fiber loading on deinked wastepaper, In 1996 Tappi Pulping Conference Proceedings, Oct. 27–31, Nashville, TN. TAPPI Press, Atlanta, GA, pp. 895–900.
- Klungness, J., Caulfield, D., Sachs, I., Sykes, M., Tan, F., and Shilts, R.** (1993): U.S. Patent 5,223,090, (1997) RE 35,460.
- Klungness, J.H., Caulfield, D.F., Sachs, I., Tan, F., Sykes, M.S., and Shilts, R.A.** (1994): Fiber loading: a progress report, In 1994 Tappi Recycling Symposium Proceedings, May 15-18, Boston, MA. TAPPI Press, Atlanta, GA, pp. 283–290.
- Klungness, J.H., Planta, F., Strolka, M.L., Sykes, M.S., Tan, F., and AbuBakr, S.M.** (1998): Lightweight, high-opacity paper: process costs and energy use reduction, In Proceedings of 1998 AIChE Symposium Series, Oct. 25–29, Montreal, Canada.
- Klungness, J.H., Strolka, M.L., Sykes, M.S., AbuBakr, S.M., Witek, W., and Helse, O.U.** (1999): Engineering analysis of lightweight high-opacity newsprint production by fiber loading, In 1999 Tappi Joint Conference proceedings, March 1–4. TAPPI Press, Atlanta, GA, pp. 1047–1053.
- Scallan, A.M. and Middleton, S.R.** (1985): In "Papermaking Raw Materials," vol. 2. Mechanical Engineering Publications, London, pp. 613–630.
- Sykes, M., Klungness, J., Tan, F., AbuBakr, S., Rantanen, W., and Aziz, S.** (1997): Effects of processing and recycling on properties of fiber-loaded handsheets, *Prog. Paper Recycling* 6(4): 37–43.
- Sykes, M.S., Klungness, J.H., Tan, F., and AbuBakr, S.M.** (1998): Value added mechanical pulps for lightweight, high-opacity paper, In 1998 Tappi Pulping Conference proceedings, Oct. 25–29, Montreal, Canada. TAPPI Press, Atlanta, GA, pp. 539–544.

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