Specialty Cellulose Fibers for Cement Reinforcement

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Synopsis: This paper describes the investigation of a new range of cellulose fibers suited to the reinforcement of a Portland Cement matrix.

This investigation indicated that fibers selectively derived from high density summerwood are better suited for reinforcement than is the unmodified pulp which contains a large measure of fibers derived from springwood as well as summerwood.

Another cellulose fiber material, termed expanded fiber because of its finely fibrillated microstructure, was indicated to have potential as a processing aid. Expanded fiber displayed excellent suspending and retention properties and imparted relatively high uncracked strength to finished composites.

Overall, substantial performance differences were observed comparing tests on wet versus dry specimens and the long term durability was not evaluated. Despite these limitations, flexural stress/strain performance of the cellulose reinforced composites compared quite well to asbestos and glass fiber reinforced composites. The cellulose composites had substantially more ductility than asbestos cement; in this regard the load-deflection curve was similar to glass reinforced cement.

Keywords: asbestos; cellulose fibers; ductility; fiberboard; fibers; flexural strength; load-deflection curve; performance tests; portland cements; reinforcing materials

INTRODUCTION

Background

Asbestos for cement reinforcement consumed 1.5 million metric tons of fiber by early in this decade. Usage was principally for factory-made cement cladding panels and pipes produced in some 800-900 manufacturing units operating in virtually every country of the world. Consequently, the asbestos replacement activity during recent years has resulted in vast world-wide research into alternative cement reinforcement fibers.

Cellulose is widely regarded as offering one of the best cost/performance positions among the potential replacement fibers which include glass, carbon, aramid, acrylic, poly (vinyl alcohol), polypropylene, and others(1). Cellulose has been used for many years to some extent as an additive in the conventional asbestos cement industry; some of the asbestos cement replacement products utilize cellulose fibers as well(1). Cellulose fiber in these cases is sometimes used in small amounts, 1% or less by weight, in combination with the other fibers; in this role the cellulose contributes mainly processing benefits rather than reinforcement(1).

It is the purpose of this paper to report on an investigation designed to quantify the potential for cellulose fibers in general and, in particular, a new range of modified cellulose fibers to act as the sole reinforcement of a Portland Cement matrix.

Cellulose Fiber Preparation

It was a purpose of this investigation to focus on the effect of novel forms of cellulose fibers achievable by making modifications to the physical form of the fibers.

One of the modified fibers used in this study was made by a separation process by which a fraction composed predominantly of fibers of the summerwood type is created. Annual growth rings of coniferious (softwood) trees are comprised of low density, springwood, rings and high density, summerwood, rings. For illustration, the SEM micrographs in Figure 1 are two magnifications of a typical cross section of spruce, a Northern softwood. The thinner cell wall material of the springwood fibers compared to the summerwood fibers is clearly evident.

After the kraft pulping process, the fibers collapse into flat ribbons, with the springwood fibers being wider, but thinner than the summerwood fibers. Figure 2 is a photomicrograph of slash pine fibers after the pulping process. Slash pine is a Southern U.S. species and is the sole starting material for the cellulose fibers used in this investigation. Quantitatively, slash pine springwood fibers average 61 microns width at 12 microns cell wall thickness, while summerwood fibers average 37 microns width at 4 microns cell wall thickness.

Since there is little difference in the fiber length or in the density of the cell wall substance, a given mass of the summerwood fibers will contain about one-half as many fibers as a given mass of springwood fibers. However, the summerwood fibers have much higher fiber strength due directly to the greater mass of cell wall substance and indirectly to differences in the S_2 cell wall structure(2), in particular the average fibril angle(5).

The naturally occurring unweighted percentage of summerwood fibers in the slash pine pulp, designated SSK in the present investigation, is 55%. For the purposes of this study, a fractionated laboratory sample which contained 86% summerwood fibers was prepared (SSK-SUWD).

A second modified fiber type was prepared by reducing SSK fibers to a finely divided fibrillar material to examine the effect of extreme fine particle diameter on the reinforcement potential. Fibrillation is readily accomplished with naturally occurring cellulose fibers which are built-up of fibrils which can be separated and further sub-divided into smaller and smaller diameters owing to the high levels of molecular orientation. Indeed, a level of fibrillation is introduced by the action of beaters and refiners in common use by the paper industry to alter the drainage and bonding characteristics of paper pulps. A much higher level of fibrillation was used to prepare the modified fiber referred to as Expanded Fiber (EF) in this paper. In EF the fibrillation process was carried to extreme to render the constituent fibrils virtually completely separated.

The EF used in this investigation was prepared using SSK in a 1.5 liter horizontal media mill(3). A comparison of EF to SSK fiber prior to the fiber expansion process is illustrated by the micrographs in Figure 3, which display the fiber before and after the fibrillation process. The degree of fibrillation is best quantified by the tendency of an aqueous dispersion of EF to resist separating and settling from the aqueous medium. Specifically, the EF specimen in this investigation was fibrillated until a 0.1% dispersion of EF solids in water would settle to only 50% of its original volume upon quiescent standing for one hour.

Composites Preparation

Since fiber cement production technology based on the Hatschek process is widely available, it is highly desirable for a fiber to be compatible with this slurry dewatering method of composites formation(1). Consequently, we selected a laboratory, slurry dewatering process to investigate the potential of these fibers for reinforcement, essentially described as follows:

1. The cellulose fiber materials were dispersed with high speed mixing in an appropriate quantity of water for good dispersion. The solids content at this point varied from 1.75% to 4.6%.

2. Type 1 Portland Cement was added, with high speed agitation, to the fiber slurry in an amount necessary to give the desired final fiber content based on the weight of the dry ingredients.

3. The slurry was flocculated by adding an anionic polyacrylamide with gentle agitation, then poured into a mold fitted with an assembly of permeable screens, the finest being 100 mesh, to form the 15 inch (0.37 meter) square composite panels.

4. Excess water was removed through the screen using a vacuum dewatering device at 5 in (127 mm) mercury. Cement fines in varying amounts were visible in the drain water; this water was retrieved and the fines loss determined.

5. The moist cakes, which varied from 0.5 to 0.75 inch (13 to 19 mm) thick after dewatering, were pressed at 1200 psi (8300 kPa) for 3 minutes. Pressing was carried out in a hydraulic press designed to permit the excess water squeezed from the wet cake to be removed.

6. The procedures of steps 4 and 5 were adequate for all mixes except those containing EF only. In this case, the slurry would not dewater by vacuum and it was necessary to use the hydraulic press to remove all of the water. While dewatering in this way was a slow process, the maximum pressure of 1200 psi (8300 kPa) was maintained for only the 3 minute standard.

7. The composite boards after removing from the press were allowed to harden for four hours, then stored on edge and moist cured at 23°C and 100% relative humidity for seven days.

8. After moist cure, the boards were wet sawed into flexural test specimens of 2 X 12 X 0.5 inch (51 X 305 X 12.7 mm). These were stored at 23°C and 50% relative humidity until tested.

9. For each composite type, six specimens were tested dry and six specimens were tested wet. The dry specimens were tested as conditioned. The wet specimens were further prepared by soaking in water at 23^{o}C for 24 hours prior to testing.

10. All specimens were tested in third-point bending using a 0.09 in/min (2.3 mm/min) cross-head speed and a 10 inch (254 mm) free span. The resultant load versus deflection curve was used to compute the proportional elastic limit (PEL), the modulus of rupture (MOR), and the total flexural toughness. The PEL is calculated using the load value at the end of linearity of the load-deflection diagram and the MOR is calculated using the maximum load; these points on a typical load-deflection diagram are illustrated in Figure 7. The total toughness is the total area underneath the load-deflection diagram.

RESULTS

Fiber evaluations were normally completed in a series of three levels of addition, 4%, 8%, and 12% based on the weight of dry ingredients. A series of this type was prepared from each of the following types of fiber:

1. SSK fiber refined in a laboratory beater to 500 ml Canadian Standard Freeness (termed SSK-R)(4).

2. SSK-SUWD, unrefined.

3. SSK-SUWD, refined in a laboratory beater to a Canadian Standard Freeness of 500 ml (termed SSK-SUWD-R).

4. Expanded Fiber (termed EF).

During the course of the study, a dispersion benefit for composites prepared with the Expanded Fiber was recognized. To further investigate this finding a single composite was prepared at the level of 1.5% by weight EF, combined with 8% by weight of SSK-SUWD (termed SSK-SUWD/EF).

Hence, a total of thirteen sample types were prepared.

Figure 4 is a graphical representation of the flexural test results for PEL, Figure 5 for MOR, and Figure 6 for total toughness.

It should be pointed out that the specimens containing fiber types SSK-SUWD-R and SSK-SUWD/EF were tested at 49 days after preparation rather than the 28 day period common for the other specimens. Although all samples had the same 7 day moist cure period, the additional conditioning time could have affected the extent of cure of the matrix to a degree, although the effect should be negligible.

In addition to the flexural test results, some quantitative and qualitative observations were made relative to the processability of the fibers. For example, the fines retention varied considerably among the fiber types. Mixes containing SSK-R fibers had a 1.0%-1.5% loss of cement fines. With the SSK-SUWD fibers the loss increased markedly to 17%-18.5%. This system also had visibly poor distribution of fiber within the composites, especially when comparing one surface of the composite with the other surface. The loss of cement fines for the summerwood decreased markedly with refining to 1.8%-2.6%. The EF fiber mixes had a negligible fines loss. Finally, the mixed fiber system where the EF was blended with the unrefined summerwood (SSK-SUWD/EF) had a 3.8% fines loss.

Table 1 lists the water-cement ratios, the densities of the wet specimens, and the densities of the dry specimens.

CONCLUSIONS

1. The overall best performing fiber was the summerwood fiber type.

   Although the unrefined version of the summerwood fiber is unacceptable from a processing standpoint, as illustrated from the poor fines retention and visibly poor dispersion in the composites, these processing difficulties with the unrefined summerwood were completely addressed by either the moderate refining to 500 ml CSF, or by blending the fiber with a small amount of the Expanded Fiber.

   In both PEL and MOR, the refined summerwood version, and the summerwood/EF blend were the best performing fibers. In toughness, the refined summerwood fiber and the summerwood/EF blend were unsurpassed, and the unrefined version was clearly superior in this case.

   In view of the lower surface area to volume ratio present in the summerwood fiber, this perhaps indicates that the specific bonding strength of cellulose to the cement matrix might be sufficient to approach the intrinsic fiber strength.

2. Expanded Fiber has potential use as a processing aid.

   Composites containing Expanded Fiber possessed outstanding fines retention; as previously noted, the composites containing EF only had negligible fines loss. Although composites reinforced with EF lacked significant toughness, they did have excellent PEL. EF can thus be viewed as a matrix modifier which has a beneficial effect in suspending and retaining the solids materials in the slurry process.

3. While the relative performance among the various fiber types tested was essentially the same whether judged from wet testing or from dry testing, the absolute test values were quite different depending on the test condition.

   Specifically, the PEL and MOR strengths were generally reduced by one-half in wet testing versus dry. Despite this reduction in load bearing ability, the total toughness values were actually observed to be about 40% greater when taken from the wet specimens compared to the dry specimens. Figure 7 is particularly illustrative of this point; it is a typical load deflection curve for the SSK-SUWD-R at 8% for both the dry and the wet condition. There are two possible explanations for this difference in behavior, wet compared to dry. First, the elastic modulus of cellulose is known to be reduced under wet condition compared to dry(5). Second, it is possible that hydrogen bonding between fiber and matrix is disrupted by the water directly resulting in more fiber pull-out.

4. The optimum fiber addition level depends upon the desired balance of properties; higher fiber levels yielding lower densities and higher toughness levels, but reduced PEL.

   Generally, toughness goes up with addition of fiber. The only exception to this was the unrefined summerwood fiber which likely suffered from dispersion problems at the high, 12%, fiber level.

   The PEL and MOR values were less predictable by fiber content. Wet PEL generally declined with higher fiber levels; dry PEL generally remained constant from 4-8% and declined at 12%. No uniform trends were evident with regard to MOR.

   Water cement ratios increased and densities decreased with higher levels of fiber. These parameters depended more upon the fiber level of addition than upon the type of fiber.

5. Refining was shown to be highly beneficial, at least for the best performing fiber type, the summerwood fiber.

   Refining improves the processability as illustrated by the summerwood fiber fines retention before and after refining; the 17%-18.5% fines loss decreased to 1.8%-2.6%. Further, refining greatly improved both PEL and MOR at some expense to toughness. This is probably a result of some fiber shortening and increasing in the fiber-to-matrix bonding with the mechanical surface modification caused by refining.

6. The best cellulose containing composite, compared to representative composites using the asbestos and glass fibers, were shown to have higher toughness than the asbestos cement at somewhat reduced load bearing ability; the load deflection diagram was similar to GFRC rather than asbestos cement.

Figure 8 is an assortment of load deflection curves intended to compare the best of the fibers tested in this investigation (SSK-SUWD-R) with asbestos cement(6) and with alkali resistant glass reinforced cement(7). Compared to asbestos cement, the cellulose cement composite panel possesses much greater energy absorbing quality at sharply reduced load bearing ability. In this regard, the characteristic curve for cellulose cement is more similar to that of GFRC.

RECOMMENDATIONS

The desirable properties of cellulose cement in view of the low cost of cellulose and its proven compatibility with the Hatschek process would indicate that there might be many applications for panel products based on cellulose fibers in general, with specific forms of cellulose capable of substantially improving composite properties.

No attempt was made in this investigation to evaluate the durability of the cellulose cement composites. A durability investigation should be completed.

The vast differences in properties of dry versus wet test specimens need to be taken into account by designers using the composites. A further modified fiber or system which would overcome these differences would be desirable.

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TABLE 1--Summary of Specimen Properties After Pressing

FIBER TYPE	FIBER CONTENT (% BY WEIGHT)	WATER-CEMENT RATIO	WET DENSITY (lb/ft^3)	DRY DENSITY (lb/ft^3)
	4	0.33	126	118
SSK-R	8	0.34	120	108
	12	0.43	113	98
	4	0.34	124	119
SSK-SUWD	8	0.39	117	109
	12	0.57	109	96
	4	0.33	129	123
SSK-SUWD-R	8	0.36	122	112
	12	0.40	112	104
	4	0.30	132	123
EF	8	0.35	123	108
	12	0.42	117	101
SSK-SUWD/EF	8	0.36	117	109

SI equivalent: $1\ lb/ft^3 = 16.05\ kg/m^3$