PERFORMANCE OF SLASH PINE FIBERS IN FIBER CEMENT PRODUCTS

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ABSTRACT

Cellulosic fibers produced from slash pine (*Pinus elliotii*), a softwood from the southeastern U.S., by the kraft pulping process have been evaluated in fiber cement products. These fibers are longer and coarser than most wood fibers and have been found to provide many benefits in fiber cement applications due to their high modulus and high fiber strength. Lignin levels can be finely controlled ranging from 0% to 4%. These fibers can also be coated to provide enhanced alkaline stability. Laboratory preparations of multiple formulations of fiber cement products have been carried out incorporating these fibers. High modulus of rupture, high fracture energy (toughness), excellent dimensional stability, and low water absorption were observed. Accelerated aging tests were also run and high performance mix designs showed the most strength retention after aging. Recently conducted plant trials with these fibers have confirmed many of the laboratory findings.

KEYWORDS:

Fiber cement, cellulose fibers, slash pine, *Pinus elliottii*, cementitious composites

INTRODUCTION

Identification of health hazards associated with exposure to asbestos and asbestos-containing products led to the search for fibers to replace asbestos in fiber cement products. Although a wide variety of synthetic and natural fibers have been investigated academically, industrial practice has settled primarily on PVA (polyvinyl alcohol) and cellulose fibers, with smaller amounts of polypropylene and glass fibers used. Common industrial production uses the Hatschek or Mazza processes.

Asbestos fibers have a range of sizes that allows it to act both as reinforcing (load-bearing) fibers as well as filter fibers. (Filter fibers help retain small particles of cement and silica that are part of the dilute slurry that is flooded onto the dewatering felt associated with the Hatschek and Mazza processes.) In non-asbestos fiber cement products, the reinforcing fibers are usually PVA or refined cellulose fibers and the filter fibers are generally highly refined cellulosic fibers.

BACKGROUND

Fiber cement articles manufactured by the Hatschek process can be cured by allowing the products to set up at ambient conditions for 28 days, or placing the materials in a steam autoclave at about 180°C to accelerate the curing reactions. In this paper, samples prepared at ambient conditions will be called air-cured, and samples cured in an autoclave will be called autoclave (AC) products. Air-cured and autoclave products are made using different compositions.

PREVIOUS WORK

The use of bleached softwood kraft (BSK) fibers in fiber cement composites was reported by Harper (1982), Soroushian (1990, 1994a, 1994b), and by Vinson (1990). Sarigaphuti (1993), Shah (1994), and Soroushian (1998) have reported use of these types of fibers for reducing shrinkage cracking of concrete. Soroushian (1999) and Morton (2006) reported proposed use in structural materials. Brown (2005) reported use of slash pine fibers for slab-on-grade ready-mix concrete applications, and the alkaline stability testing of CF-16.

EXPERIMENTAL

Unless otherwise noted, all samples described in this paper were produced using the following compositions.
Air-cured: cellulose fiber, PVA fiber, amorphous silica, filler, Portland cement.
Autoclaved: 8% cellulose fiber, crystalline silica, Portland cement, special additives.

The cellulose fibers are refined to various freeness levels depending on the type of product being prepared. Refining (also termed beating) fibrillates the surface of the fiber and causes dilute slurries of cellulose fibers to drain more slowly. Freeness levels in this paper are given as Canadian standard freeness (CSF) values determined according to TAPPI T 227 om-04: Freeness of pulp (Canadian standard method).

Conditioned flex indicates the samples have been placed in a humidity/temperature-controlled room for at least 48 hours prior to testing. Wet flex means that the samples have been submerged under water for at least 48 hours, and oven dry flex indicates the samples were placed in an oven at 60°C for at least 48 hours. Conditioned and wet densities are measured on the samples prior to testing. Oven dry densities are determined after 3 days in an oven at 105°C. Water absorption is determined by using the weights from wet density and dry density measurements, expressed as a per cent.

Akers et al. (1989) developed a test for the accelerated aging of fiber cement products. "Aged" samples have been through 30 cycles of accelerated aging in a special aging chamber (a modified Blue M forced draft oven). Each 24-hour cycle consists of the following steps:
1. Reservoir containing samples is filled with water saturated with calcium hydroxide from an external bath at 20°C (15 min.), 2. Samples remain submerged for 8 hours, 3. Alkaline water is pumped out of reservoir and back into external bath (15 min.), 4. Oven is heated to 60°C (1 hour), 5. Oven is turned off and CO_2 atmosphere established (5 hours), 6. Oven is heated to 60°C while fan removes CO_2 (9 hours), 7. Oven cools for 30 min. before step 1 starts another cycle. Experience has shown that 30 cycles in the aging chamber is roughly equivalent to 7 years of natural aging in Switzerland.

FIBER PROPERTIES

The properties of fiber cement composites are determined by the properties of the constituent parts. The effect of cellulose fiber properties on the performance of the fiber cement product is often under estimated. Long fibers perform better than short fibers (therefore softwood pulps are used rather than hardwoods) since they have more embedded length. Coarse fibers with thick cell walls are stronger than fine fibers with thin cell walls. The coarseness of cellulose fibers is usually given as a linear density, where the units are mg/100 meters (also called decigrex). Synthetic fibers generally use the linear density unit called denier (g/9000 meter).

Table 1 lists some commercially available cellulose pulps used to make fiber cement products.

Table 1 – Examples of pulps used in fiber cement products.

Product	*Company*	*Country*	*Fiber Type*	*Type*
CF-16	Buckeye Technologies	USA	Slash pine (*P. elliottii*)	BSSK
CF-12	Buckeye Technologies	USA	Slash pine (*P. elliottii*)	USSK
Canfor	Canfor Pulp Limited Partnership	Canada	White spruce (*P. glauca*) & lodgepole pine (*P. contorta*)	UNSK
Celco	Arauco	Chile	Radiata pine (*P. radiata*)	USSK
Solombala	Sol. Pulp/Paper Mill	Russia	Spruce/pine	UNSK
Springwood Structa	SAPPI	South Africa	Slash, loblolly & Mexican pine (*P. elliottii, taeda, & patula*)	USSK
Tasman	Carter Holt Harvey	N. Zealand	Radiata pine (*P. radiata*)	USSK

BSSK= bleached southern softwood kraft, USSK= unbleached SSK, UNSK= unbleached northern SK

Fiber properties, determined using a Kajaani Fiber Lab 3 instrument, are given in Table 2.

Table 2 - Fiber properties of illustrative pulps used for fiber cement products

	Fiber Length	***Coarseness***	***Wall***	***Fiber***
Product Name	*Mm*	*mg/100m*	***Thickness, µ***	***Width, µ***
CF-16	2.94	25.28	9.5	31.4
CF-12	3.05	24.09	10.2	32.4
Canfor	2.70	15.09	8.1	29.3
Celco	2.73	20.27	9.7	32.5
Sappi	2.97	23.38	10.0	34.1
Solombala	2.70	15.55	7.9	30.7
Tasman	2.93	22.76	10.9	32.6

The data given in Table 2 agrees very well with literature values that show that Southern pines have higher coarseness and a thicker cell wall than Northern species. (For more information about wood fibers, see Kocurek, 1983.) Note that CF-16 is bleached, and all other pulps are unbleached.

FIBER CEMENT APPLICATIONS

Table 1 has 2 slash pine pulps. CF-16 is a fully bleached pulp with a coating to enhance alkaline stability (Brown (2005)). It is intended primarily for uses in air-cured fiber cement products, although it can be blended with unbleached fibers for use in both air-cured and autoclaved applications. CF-12 is an unbleached pulp made from the same slash pine furnish which is pulped to about 3.2% lignin (TAPPI T 222 om-02: acid-insoluble lignin in wood and pulp), or about 25 kappa # (TAPPI T 236 om-99: Kappa number of pulp). It is intended primarily for use in autoclaved products, although it can also be used for air-cured applications.

Air-cured (naturally aged)-fiber cement products

This section describes the improvements in performance of air-cured products prepared with bleached slash pine fibers containing the alkali-resistance coating compared to standard unbleached fibers. The performance benefits presented below include:

1. Improved flexural toughness
2. Improved durability based on flexural toughness after accelerated aging
3. Higher product density at equal pressing
4. Improved wet/dry shrinkage
5. Reduced refining energy/reduced time to achieve equivalent freeness.

These samples were prepared using the composition described earlier (see Background section). The bleached slash pine sample (designated CF-16) was split into 2 equal portions. One was refined to 180 CSF to act as a filter fiber and the other sample was refined to 360 CSF to be the reinforcing fiber. The control used equal portions of unbleached radiata pine as reinforcing fiber (360 CSF) and unbleached northern spruce/pine as the filter fiber (180 CSF). The refining was carried out in water saturated with calcium hydroxide to simulate industrial practice.

Figure 1 gives the results of flexural toughness testing. These results are based on the 3-point loading test described in ASTM method C 1185 with the exception that our lab samples are formed in 4 inch (102 mm) width and 8.5 inch (210 mm) length. The test span is 200 mm and the flexural toughness is the area under the load/displacement curve up to the point of failure (i.e. the peak load where Modulus of Rupture (MOR) is determined). The units for flexural toughness (sometimes called fracture toughness) are KJ/m^2. MOR for the slash pine and control samples were found to be statistically equivalent, but significant differences were observed in toughness.

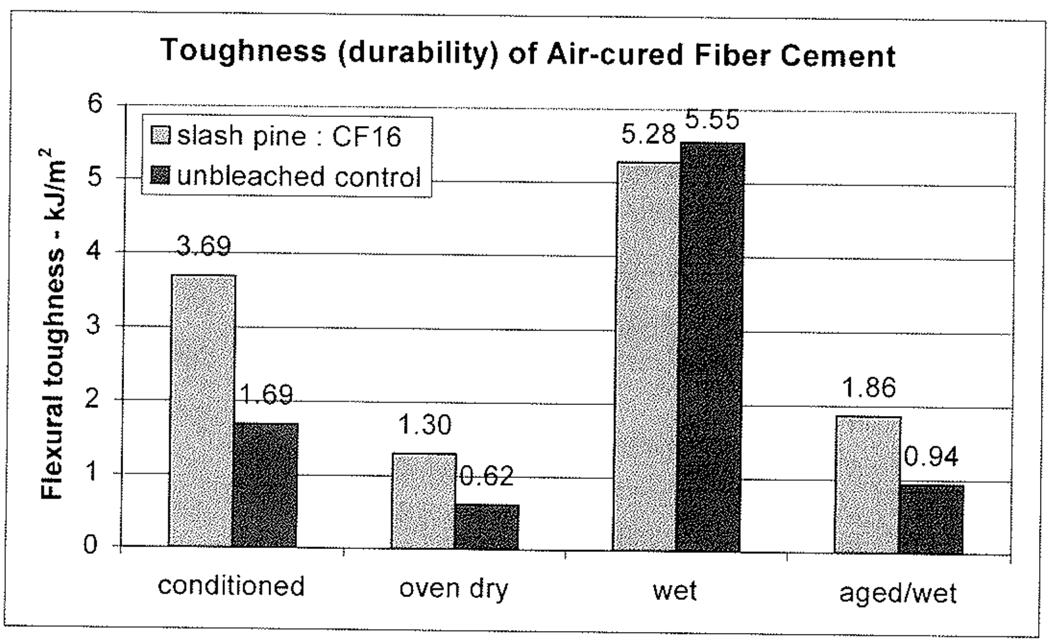

Figure 1: Flexural toughness of bleached slash pine fibers compared to unbleached control fibers

Note that the toughness is not only higher under ambient conditions, but the improvement in toughness is retained even when the fibers are heated to dryness in an oven. This suggests that the fiber coating provides protection in the alkaline environment, since it is generally observed that bleached fibers do not respond well to heating in an alkaline environment. The wet conditioned toughness is the same, presumably because the water makes the sheet more elastic, resulting in more fiber pullout and other complex mechanisms.

Although both samples have decreased in toughness, the reduction for the bleached slash pine fibers is less, indicating that the sample will become brittle more slowly than standard brownstock fibers.

Figure 2 shows the response of these fiber cement samples to pressing.

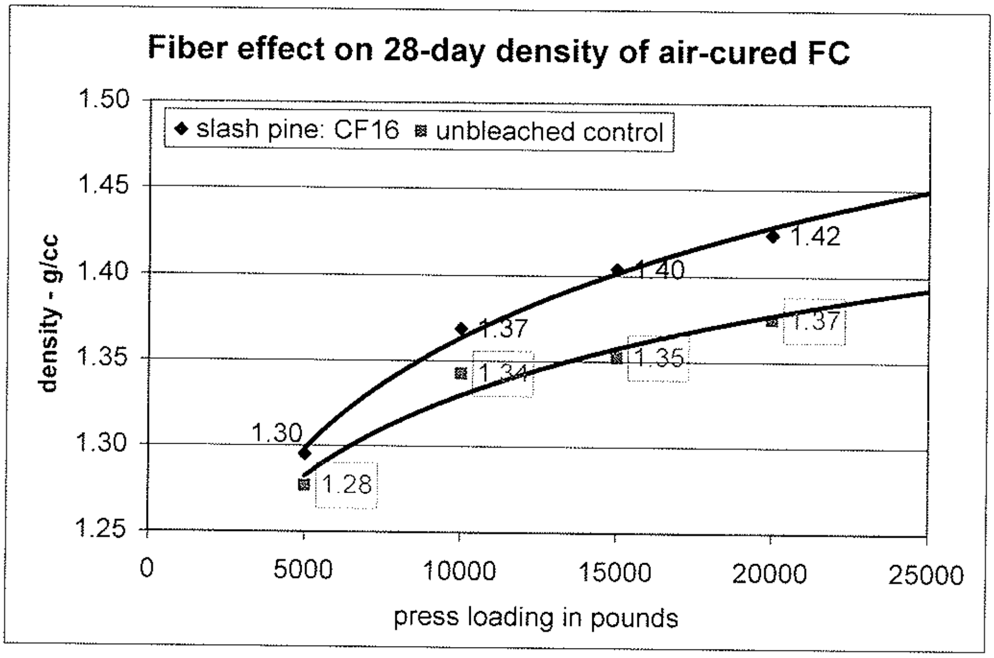

Figure 2: Response of fiber cement samples to Carver press loading

The mix design and pulp freeness combinations for these samples were the same as the samples described in Figure 1. These samples were prepared in a cylindrical mold of about 4 inch (110 mm) diameter and placed in a Carver press immediately after removing from the mold. Bleached fibers are known to be more flexible and more easily compacted than unbleached fibers. This would allow the manufacturer to either cut back on pressing while achieving the same density or maintain the press loading and get a denser product.

Improved beating response for bleached slash pine fibers (CF-16) is demonstrated in table 3 by a lower time requirement to obtain a desired freeness values by using a Valley beater.

Table 3: Beating response in Valley beater for bleached slash pine versus unbleached control

Fiber type	Freeness (CSF)	Time (minutes)
CF-16: reinforcing fiber	362	33
CF-16: filter fiber	189	45
Control: reinforcing fiber	355	63
Control: filter fiber	180	71

Co-refining (Valley beater) of a 1/1 blend of CF-16 & UNSK (different from Table 3) gave a freeness of 260 CSF after 52 minutes. Separate refining gave 170 CSF for CF-16 and 357 CSF for UNSK. The average of these values is 263, indicating co-refining gives the same result as refining separately, then blending. Co-refining of these pulps provides reinforcing and filter fibers in the same pass at lower refiner energy/time.

Wet/dry shrinkage and %absorption values are given in Figure 3. The % absorption is the same. Differences in wet/dry shrinkage seems small, but were found to be statistically significant. Improved dimensional stability is especially important for large façade panels.

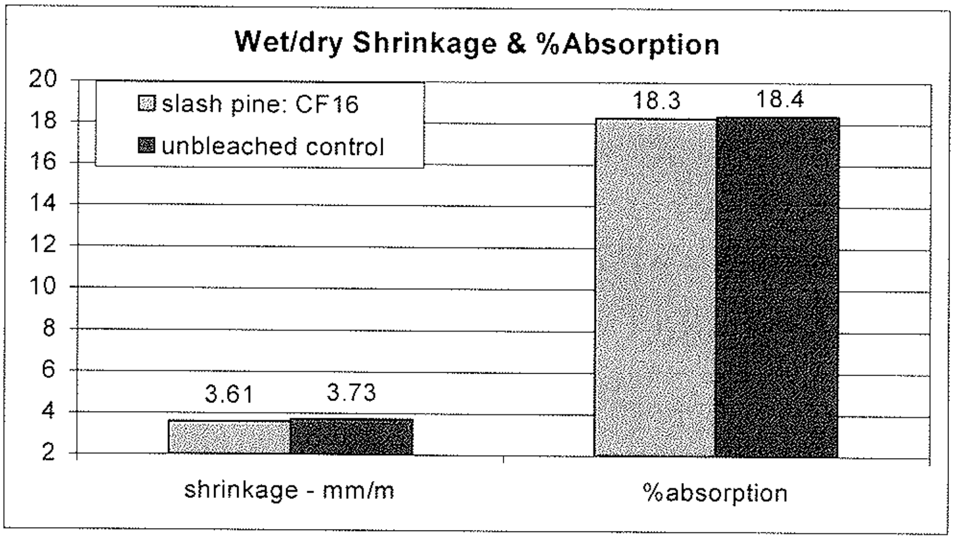

Figure 3: Wet/dry shrinkage and %absorption for slash pine and unbleached control samples

In order to confirm laboratory results on hand made samples, it was decided to carry out a full scale industrial trial using CF-16 fibers. This trial aimed at using CF-16 fibers as a partial replacement for PVA fibers in an air-cured mix. A special mix was designed where 40% of the PVA fibers were replaced with CF-16 fibers.

The filter fiber used for this mix design was a highly refined standard UNSK brownstock from an independent company (UNSK = unbleached northern softwood kraft). The standard mix was used as comparison, and the results are given in Table 4.

Table 4: Strength and shrinkage properties of samples prepared with commercial equipment

Flexural strength, N/mm^2	**Reference**	**Experimental**
Conditioned	10.0	12.7
Wet	8.5	10.0
Conditioned, aged	11.4	13.0
Wet, aged	9.6	9.7
Wet dry shrinkage, mm/m		
conditioned	3.6	3.1
aged	2.7	2.4

It can be seen from these results that improved mechanical properties were achieved with a trial mix, which confirmed laboratory results. This also confirms with industrial trials that there is a significant improvement in wet/dry shrinkage values when using Buckeye fibers. It has now been established that CF-16 fibers can be used on an industrial scale for partial replacement of PVA fibers

Autoclaved fiber cement products

The composition for the lab-made autoclave samples is given in the background section. The autoclave is steam vented to remove air, then held at 180°C for various lengths of time to give a mild cure, a moderate cure or a severe cure. The freeness levels are indicated for specific results.

Fiber cement products described in this section were prepared from unbleached slash pine fibers designated CF-12. Benefits observed include equivalent or higher strength and toughness versus other commercial species. Improved beating response was found versus radiata pine and similar beating was observed versus northern unbleached species. Improvements in wet/dry shrinkage were also observed.

Table 5 shows the flexural strength testing of unbleached slash pine versus a commercial unbleached radiata pine pulp from Chile.

Table 5: Comparison of flexural properties of unbleached slash and radiata pines

Sample:	toughness	MOR	density	CSF
	(kJ/m2)	(N/mm2)	(g/cc)	(ml)
unbleached slash - conditioned	0.879	17.1	1.31	175
unbleached radiata - conditioned	0.805	16.2	1.29	203
unbleached slash - wet	2.147	11.2	-----	-----
unbleached radiata - wet	1.857	10.3	-----	-----

Table 6 gives wet/dry shrinkage (WDS) and % absorption results for unbleached slash pine versus a commercial unbleached radiata pine pulp from New Zealand. WDS was determined for both moderate and severe curing conditions, where a moderate cure was about 7 hours in the autoclave, and a severe cure was about 10 hours in the autoclave.

The WDS results were found to be statistically different and the % absorption was not significantly different.

Table 6: Wet dry shrinkage and % absorption comparison of slash to radiata pines

Sample:	Wet dry shrinkage	Absorption
	(mm/m)	(%)
unbleached slash – severe cure	1.37	31%
unbleached radiata - severe cure	1.63	32%
unbleached slash – moderate cure	1.29	32%
unbleached radiata - moderate cure	1.65	30%

Figure 4 shows the relative beating responses of the unbleached slash and radiata pine fibers used in Table 7. Beating was carried out in a Valley beater in an alkaline solution (saturated calcium hydroxide). Although these fibers have similar coarseness values (Table 2), beating response is also influenced by the ultrastructure (microfibrillar winding angle, wall thicknesses, stiffness, etc) of the fibers. (See Smith, 1968 and Krassig, 1993.)

Co-refining of these fibers would also give a blend of reinforcing and filter fibers in a single pass with reduced refining but the difference in beating response is much less than for bleached fiber versus unbleached fibers.

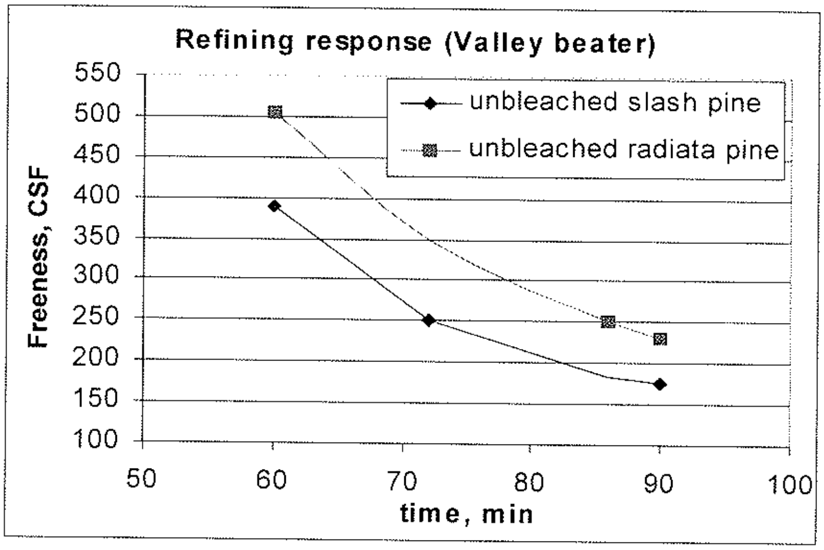

Figure 4: Beating comparison of unbleached slash pine (CF-12) to unbleached radiate pine

Laboratory fiber cement samples prepared from unbleached slash pine (CF-12) were compared to commercial fiber cement siding purchased from a local building supply store. Figure 5 shows the results of wet flexural testing. These lab samples are not oriented, so only 1 set of numbers is presented. Since the commercial sample was prepared on a Hatschek machine, both machine-and cross-direction (MD & CD) testing was carried out. Although we do not know the exact mix composition and processing details of this manufacturer, these results indicate that unbleached slash pine fibers are suitable for production of commercial fiber cement products.

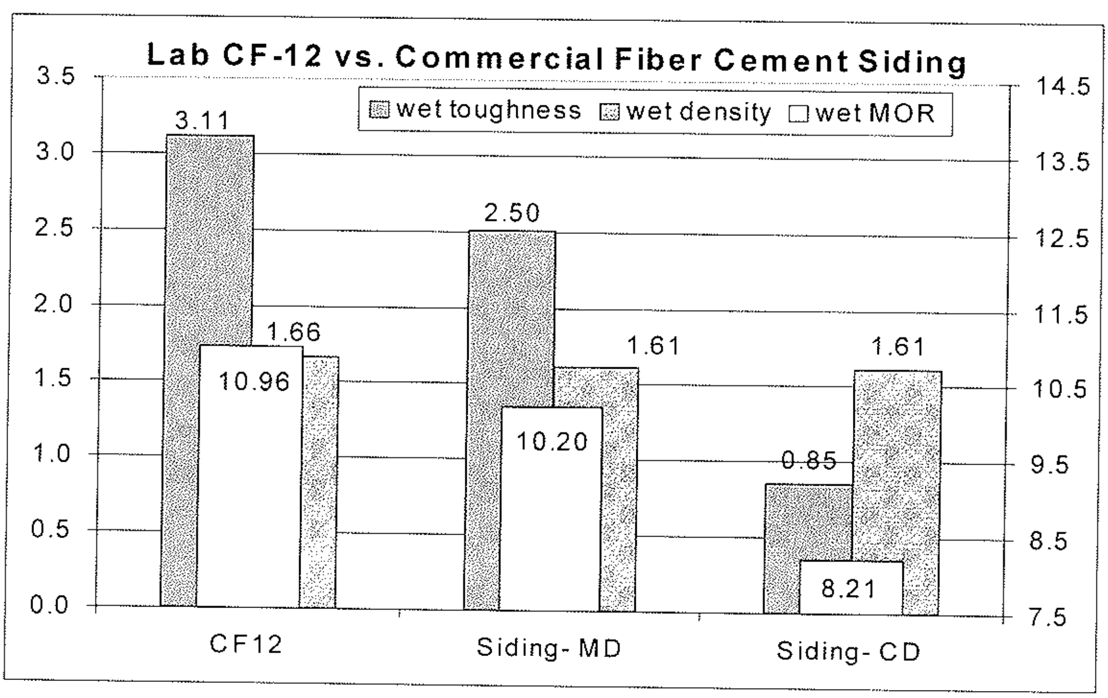

Figure 5: Comparison of unbleached slash pine (CF-12) to commercial FC siding

CONCLUSION

Fibers manufactured from slash pine trees (Pinus elliottii) by the kraft process have been shown to provide excellent performance in fiber cement composites produced by both air-cured (naturally aged) and autoclaved processes. These fibers are compared to other softwood fibers commonly used for fiber cement applications. CF-16 is a fully bleached pulp with a special coating to enhance alkaline stability that is intended primarily for uses in air-cured fiber cement products, although it can be blended with unbleached fibers for use in both air-cured and autoclaved applications. The improved alkaline stability increases the durability and strength in a cementitious matrix, as reported by Brown, 2005. CF-12 is an unbleached pulp made from the same slash pine furnish and is intended primarily for use in autoclaved products, although it can also be used for air-cured applications. CF-12 is different from CF-16 in that it is unbleached and uncoated.

Bleached slash pine fibers with a special coating to enhance alkaline stability provide strength, durability, processing, and dimensional stability benefits for air cured products. Unbleached slash pine fibers provide similar benefits for autoclaved products. Both fibers provide improved beating response versus other commonly used fibers and can be co refined with appropriate brownstock fibers to provide both filter and reinforcing fibers in a single pass. Lab results have been verified by full scale industrial production of fiber cement products.

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