PROCESSING OF HIGH-PERFORMANCE FIBER-REINFORCED CEMENT-BASED COMPOSITES

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ABSTRACT

High-performance fiber-reinforced cement-based composites (HPFRCC) are characterized by their high elastic limit and strain hardening and a progressive multiple cracking type of response to mechanical loading. The parameters that influence the performance of such composites include: fiber type(s), matrix properties and processing. Processing can substantially influence fiber dispersion, quality of performance and cost of production. In spite of its importance, relatively little is known about the relationship between processing and composite performance.

For cast concrete, the use of self-compacting concrete has been shown to result in superior performance of fiber reinforced concrete. Various options available for manufactured products include: glass fiber reinforced sprayed panels for industrial buildings (GFRC), Hatscheck process and extrusion for residential applications. In this work, the influence of processing on the performance of Hatschek- and extrusion- produced HPFRCC is investigated.

Hatschek-produced fiber-reinforced cement board (FRCB) is currently used in residential applications as siding and backerboard. Due to its laminated structure, FRCB is susceptible to freeze-thaw attack. The effect of applying external pressure to freshly formed FRCB was systematically evaluated. The results indicate that pressure can be used to enhance freeze-thaw durability.

Extrusion offers a number of benefits for producing HPFRCC for residential applications, including excellent mechanical performance and durability. To manufacture these composites, very specific fresh state properties are needed so that it is soft enough to flow through the die yet stiff enough to maintain its shape once it exits the die. These properties are currently achieved in part by the addition of cellulose ether, the cost of which can be substantial. A rheological study was successfully carried out to reduce cost by altering the matrix composition.

KEYWORDS:

Processing; Rheology; Hatschek Process; Extrusion; High-Performance Fiber-Reinforced Cementitious Composites

INTRODUCTION

Cementitious composites are typically characterized as brittle, with a low tensile strength and strain capacity. Fibers are incorporated into cementitious matrices to overcome this weakness, producing materials with increased tensile strength, ductility and toughness and improved durability (Balaguru 1992). The efficacy of

the fiber reinforcement is dependent upon many factors, including the properties of the matrix as well as the fiber geometry, size, type, volume and dispersion.

The general tensile behavior of cementitious composites is demonstrated in Figure 1. Plain unreinforced cementitious materials exhibit a strain-softening response with low tensile strength and ductility. Conventional fiber-reinforced composites (FRC) are reinforced with a low volume of fibers, typically 0.5 – 2%, and also have a strain-softening response, but demonstrate an increase in post-peak ductility. With HPFRCC, there is an increase in the elastic limit (defined as the point at which the first macrocrack is formed; prior to this, microcracking dictates performance), followed by a strain-hardening response as multiple cracks form but do not widen. Finally, strain softening is seen as cracks widen. Researchers have demonstrated that this high performance can be achieved in a variety of ways, including using micromechanical modelling (Li 2001), tailored fiber geometries (Naaman 2003) and advanced processing techniques (Li 1993; Shao 1993; Mobasher 1998; Peled 2000; Peled 2003).
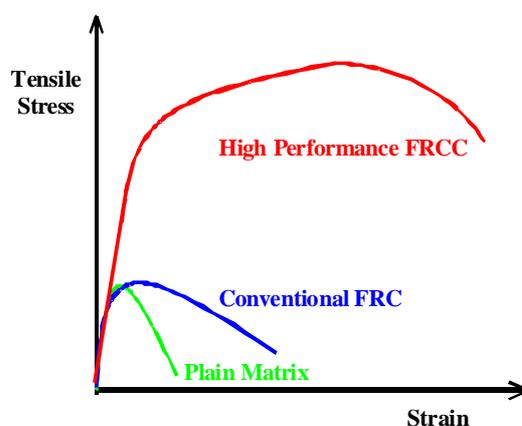
Figure 1. Effect of fibers on tensile performance of cementitious composites

Processing can substantially influence fiber dispersion and orientation, quality of the composite performance and cost of production. Incorporation of fibers influences the ease with which cement-based materials are handled when in the fresh state and can also affect hardened state properties (Ozyurt 2006). The fresh state properties needed depend on the processing method and parameters. If the fresh state properties of composites are not properly controlled, excessive voids and difficulties with fiber dispersion can result. Fiber dispersion has been shown to significantly impact mechanical performance (Akkaya 2001; Ozyurt 2006). Processing can also be quite expensive, limiting the extent to which new technologies are embraced by industry. In spite of its importance, relatively little is known about the relationship between processing and composite performance.

HATSCHEK-PRODUCED FIBER-REINFORCED CEMENT BOARD

FRCB is produced commercially for backerboard and siding as an alternative to wood products in domestic building applications because it is more fire-resistant, can better withstand fading, is not susceptible to insect attack and is more durable. Hatschek-produced FRCB is a laminated, autoclave-cured material that is reinforced with a significant amount of fibers, approximately 30-35%, by volume. Originally, these composites were reinforced with asbestos fibers; however, due to the hazardous nature of these fibers, they have been replaced with cellulose fibers.

Despite the benefits of using FRCB in residential applications, the freeze-thaw durability of the composites can be poor. FRCB is susceptible to freeze-thaw deterioration because of its high porosity, the organic cellulose fibers that reinforce it and the laminated structure of the material. As a porous material, FRCB

allows substantial amounts of water to ingress. Cellulose fibers have a tubular structure and absorb water. Consequently, FRCB is sensitive to ambient moisture, weakening significantly when wet. As water is absorbed, the fiber-matrix bond is reduced (Coutts 1984; Soroushian 1994). During freezing and thawing, the bond between the matrix and the fibers weaken because of the expansion and contraction of the fiber, significantly reducing the strength of the material.   The laminated structure of FRCB also makes it vulnerable to freeze-thaw action. These layers provide a path for the ingress of water (Nakamura 1992).

To overcome these material weaknesses, some manufacturers have started pressing the FRCB after it is formed, expelling excess water and possibly improving the ILB. Previous research has shown that applying pressure to FRCB improves its mechanical performance. Pressing asbestos-reinforced FRCB increased the density and flexural strength (Akers 1986). Applying pressure to wood-reinforced cement board during casting improved the pore structure and the interfacial bond (Gupta 1978; Coutts 1990). In this work, the effect of pressure on the freeze-thaw durability of FRCB was investigated, with particular attention to the role of the interlaminar bond.

Effect of Pressure on Mechanical Performance of FRCB

Commercial Hatschek-produced FRCB that were 5/16 inch thick, reinforced with cellulose fibers and autoclave-cured were investigated. After the boards were formed, external pressure was applied at varying levels: 0, 10, 20 and 30 bars.

Flexural Performance
Closed-loop three-point bend tests were used to evaluate the flexural performance of the FRCB. 175 mm x 50 mm specimens were loaded over a 152.4 mm span in displacement control at a rate of 0.00381 mm/sec. The load and displacement data obtained were converted to stress versus displacement curves. From these curves the flexural strength (maximum stress reached) and toughness (area under the curve) were determined.

Figure 2 presents the flexural strength of the FRCB as a function of pressure and shows that flexural strength increases with increasing pressure, improving by 65% from the 0 bar to 30 bar product. This increase is expected since applying a greater pressure reduces voids in the matrix and improves the bond between the cellulose fibers and the matrix. All other things equal, a more dense material has a higher flexural strength. An improved bond between the fibers and the matrix leads to a stronger, more brittle material. Flexural toughness was also evaluated. The results indicate that flexural toughness decreases with increasing pressure, again indicating an improvement in the fiber-matrix bond with increased pressure (Kuder 2003). These results were confirmed by examining the fracture surfaces of the composites using scanning electron microscopy after three-point bend testing (Kuder 2003).
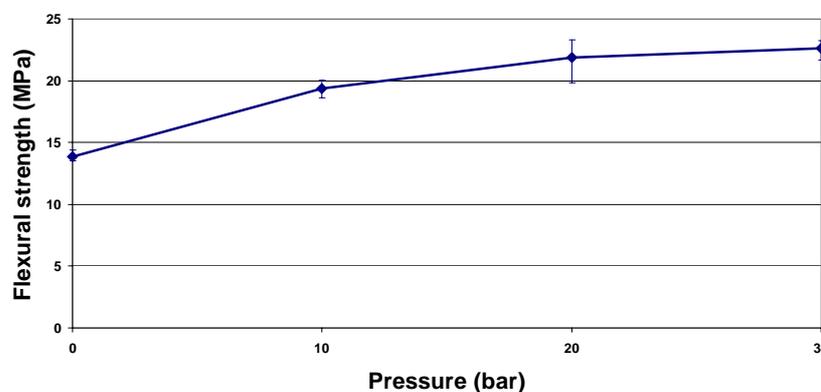
Figure 2. Flexural strength versus pressure with high and low values

Interlaminar Tensile Strength

To evaluate the interlaminar properties of the FRCB, a new interlaminar tensile strength test was developed. Square specimens, 25.4 mm x 25.4 mm, were glued in the testing machine with epoxy, such that the load was perpendicular to the laminates. A uniform compressive load of 44.5 N was applied until the epoxy cured. Closed-loop testing was then used to apply uniform tension at a rate of 0.004 kN/sec until interlaminar (tensile) failure occurred. The maximum force sustained divided by the cross-sectional area gave the ILB strength of the material.

The ILB strength of the FRCB is shown in Figure 6. A 200% increase in ILB strength is observed, from 0 to 30 bars, demonstrating that pressure improves ILB.
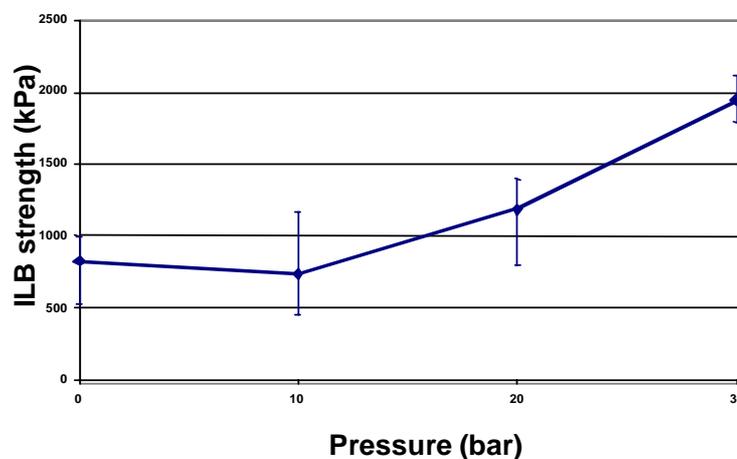
Figure 6: ILB Strength vs. pressure with high and low values

Mercury intrusion porosimetry (MIP) was used to characterize the porosity of the composites. Complete details from this investigation are presented elsewhere (Kuder 2003), but are summarized here. The results indicate that the improvement in flexural and ILB strength with increasing pressure may be a result of the reduction in interlaminar space and the area between the fibers and the matrix. The total porosity (cumulative volume) is reduced with increasing pressure, indicating a reduction of voids. The macroporosity (pore radius > 1 micron) decreases with increasing pressure, but the microporosity stays the same. The critical pore diameter for all of the materials is the same, suggesting that the matrix is not affected by pressure.

Effect of Pressure on Freeze-Thaw Durability of FRCB

To assess the freeze-thaw durability of the FRCB, specimens were subjected to as many as 300 accelerated freeze-thaw cycles according to a modified version of ASTM Standard C1185 "Standard Test Methods for Sampling and Testing Non-Asbestos Fiber-Cement Flat Sheet, Roofing and Siding Shingles, and Clapboards (3)." Saturated specimens were cycled from -20°C to 20°C in a freeze-thaw chamber that was manufactured by Humbolt Manufacturing Company. Each cycle took approximately 12 hours.

Flexural performance and the interlaminar properties of the FRCB were evaluated after 50, 100, 150, 200 and 300 cycles. Complete details are given elsewhere, but are summarized here (Kuder 2003). The results show that flexural strength decreases after freeze-thaw conditioning. After 50 cycles, flexural strength improves with increasing pressure treatments. However, after more than 50 cycles, pressure treatments do not affect the flexural performance.

The effect of pressure on the interlaminar bond strength is shown in Figure 3. A significant decrease in strength, at least 80% for all materials, is seen after only 50 cycles. Despite this breakdown of the material structure, there is still a difference in the ILB strength for the different pressures. Even after 200 cycles, the

ILB strength of the 30 bar material is twice that of the 0 bar, indicating that pressure may improve the ILB strength.
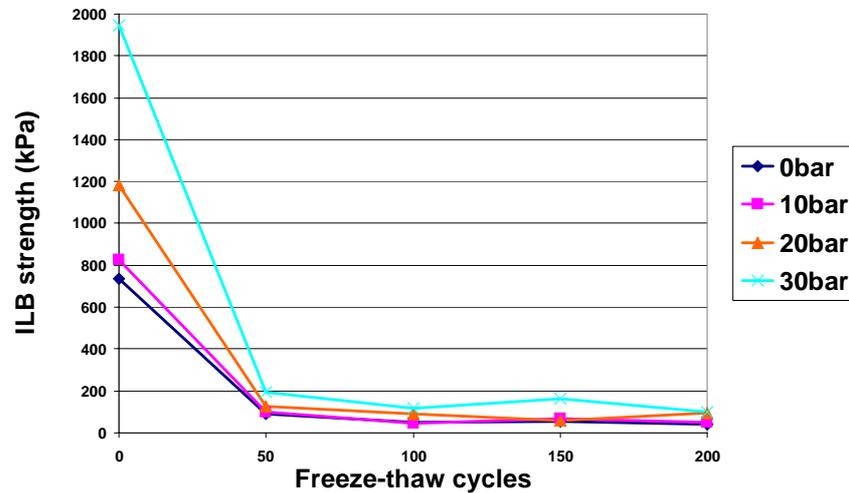
Figure 3. ILB versus freeze-thaw cycles

Visual observations during the three-point bend test confirm that the ILB strength is improved by pressure. Even after 300 cycles, the 30 bar material does not delaminate under the shearing action imposed by the test. However, after only 50 cycles, the 0, 10 and 20 bar material exhibit delamination. Figure 4 shows 20 and 30 bar products that were subjected to 200 freeze-thaw cycles during the three-point bend test. Interlaminar failure occurs with the 20 bar specimen, but the laminates of the 30 bar product remain intact.
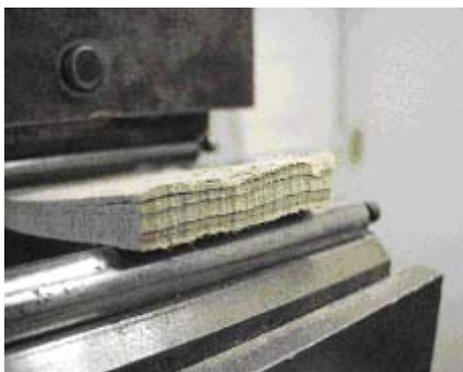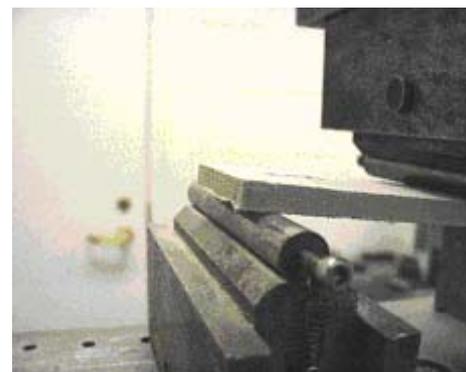
(a)   (b)

Figure 4. Three-point bend testing after 200 cycles (a) 20 bar (b) 30 bar

Summary – Hatschek-produced FRCB

The laminated structure of FRCB makes it vulnerable to freeze-thaw attack. Pressure can be applied to freshly formed FRCB to increase the density, improve the fiber-matrix bond, reduce porosity and improve interlaminar tensile strength. FRCB undergoes severe degradation due to exposure to freeze-thaw conditioning; however, the research suggests that some improvement in performance is seen with composites that were pressed.

EXTRUDED HPFRCC

Extrusion is a special processing technique that is used to produce HPFRCC. Composites are formed by taking a stiff cementitious dough and forcing it through a die of desired cross section with either an auger or a ram. In addition to enhanced mechanical performance, composites demonstrate a significant improvement in durability due to the high density that results from the extrusion technique (Burke 1999; Peled 2000).

Despite the great potential of extrusion technology, it has not been widely adopted by industry. One reason for this limited use is that expensive cellulose ether processing aids are needed to control the fresh state properties of extruded materials. In this research, the use of less-expensive processing aids is examined. The extrudability of mixes containing various amounts of processing aids is evaluated and then capillary rheology is used to describe the rheological parameters of both the extrudable and not extrudable mixes.

Extrudability

For a material to be extrudable, 1) it must be soft enough to flow through the die, yet rigid enough to maintain it shape upon exit of the die, 2) the pressure required for extrusion must be reasonably low (to control manufacturing costs), 3) phase migration must be avoided and 4) the material should be shape stable. No standardized test method exists to determine whether or not a composition is extrudable. In this work, extrudability was evaluated by extruding open cross-sections, using a cellular die, with two cells, that had a total length of 25.4 mm, a width of 15 mm and a wall thickness of 3.25 mm. Specimens were extruded by a piston extruder at a rate of 1 mm/s. If poor shape stability, phase migration, an excessively high extrusion pressure, or surface defects (usually edge tearing) was observed, the material was considered not extrudable.

It is important to note that extrudability is related to extrusion velocity as well as to the shape being extruded. Therefore, the minimum amounts of binders defined here are dependent on the velocity and die used.

The matrix composition used, by volume, consisted of 33% Class F fly ash (produced by Dynegy Midwest Generation, Inc., mean particle size = 10 μm), 12% silica fume (W.R. Grace Force 10,000), 14% cement (Lafarge Type I), 39% water and 1% high-range-water-reducing admixture (Daracem 19). Two different cellulose ethers, Methocel (D) and Walocel (W), and two different clay types, Concresol (C) and Metamax (M), were studied. Mixes were prepared using a planetary (Hobart) mixer, with the dry and wet ingredients first mixed separately, then combined and mixed by hand, followed by mixing on the slow mixer speed for approximately 5 minutes, and then mixing on the medium speed for 10-15 minutes until a cohesive dough was formed.

The properties of the processing aids are given in Table 1 and Table 2. The clay binders are approximately one hundredth the cost of the cellulose ethers and previous research has shown that clay has the potential to enhance the fresh state properties of stiff cementitious materials (Malonn 2005; Voigt accepted). The amount of cellulose ethers and clays incorporated is given as a percentage of the weight of the total binder (cement + fly ash + silica fume). Mix designs are described with the binders of the mix described by weight percentages. For example, W0.25C0.25, contains 0.25% Walocel and 0.25% Concresol.

Table 1. Cellulose ether properties (Dow Chemical Company 2002; Wolff Cellulosics Company 2004)

Trade name	Producer	Chemical designation	Specific gravity g/cm^3	Viscosity Pa*s
Methocel 4fm[*]	Dow Chemical Company	Hydroxypropyl methylcellulose	1.3	2.3 - 3.8
Walocel M-20678[**]	Wolff Celulosics	Methylhydroxyethyl cellulose	3.2 - 4.0	10.4 - 12.4

[*] Viscosity determined for 1% solution, 20° C, Rotovisko rheometer
[**] Viscosity determined for 1% solution, 20° C, "by rotation"

Table 2. Clay properties (Engelhard Company 2002; Stephan Schmidte Gruppe 2004)

Trade name	Producer	Mineral Composition	Mean particle size (μm)
Concresol	Stephan Schmidt Group	kaolinite (45%), mica (35%)and free silica (20%)	˜ 0.5
Metamax HRM	Engelhard	calcined kaolinite	1.2

Table 3 summarizes the extrudable mixes for D and W. By incrementally adding cellulose ethers to the base mix, the minimum amount needed for extrusion was determined. As Table 3 indicates, half that amount of W was required compared to D. Once these minimal amounts were determined, the amount of cellulose ether was reduced by half and the clays were added. As Figure 5 demonstrates, once 0.3% of the clay was added, an extrudable mix was achieved. Similar results were found when either C or M was added. However, if all the cellulose ether was removed, the material was no longer extrudable. These results, which are explained in more detail in (Kuder 2005), indicate that it is important to find the most effective type of cellulose ether (here twice as much D is needed, compared to W, while the costs are comparable) and that cellulose ethers can be partially replaced with clay binders.

Table 3. Extrudable mixes

(a) D

Material	Extrudable
D0.5	
D1	x
D2	x
D0.5/C0.15	
D0.5/C0.3	x
D0.5/C3	
D0.5/M0.15	
D0.5/M0.3	x
D0.5/M3	

(b) W

Material	Extrudable
W0.25	
W0.5	x
W1	
W0.25/C0.15	
W0.25/C0.3	x
W0.25/C3	
W0.25/M0.15	
W0.25/M0.3	x
W0.25/M3	

0.25W   0.25W0.15C   0.25W0.3C   0.25W3C
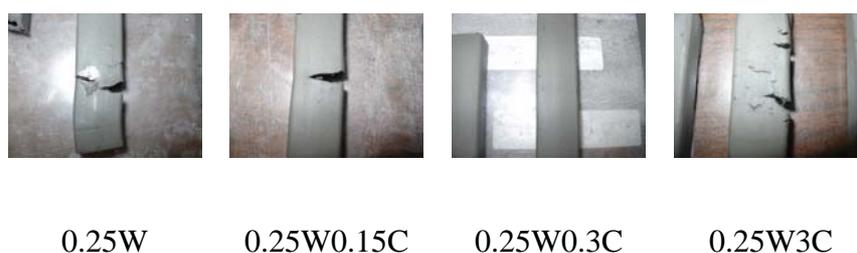
Figure 5. Effect of clay addition on extrudability

Capillary Rheology

The rheological properties of the mixes presented in Table 3 were characterized using the Benbow-Bridgewater model and capillary rheology. The results obtained using the Benbow-Bridgewater model can be found elsewhere (Kuder 2005). Results from the capillary rheology analysis are presented here.

Capillary rheology can be used to determine fundamental flow properties. Capillary analysis assumes that flow is laminar (Reynolds number < 2000), is fully developed and that there is no slip at the wall. The apparent shear stress (τ_{app}) and shear rate ($\dot{\gamma}_{app}$) are given in Equation (1) and (2), respectively.

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$$\tau_{rx} = \tau_{app} = \frac{PD}{4L} \tag{1}$$

$$\dot{\gamma}_{app} = \frac{8V}{D} \tag{2}$$

Where P is the extrusion pressure (kPa), V is the mean extrudate velocity in the capillary (mm/s), L is the capillary length (mm) and D is the capillary diameter (mm). In addition, the end effects that occur when the flow regime is complicated, as is the case for paste systems, can be taken into consideration using Bagley's end correction (Bagley 1957), which determines the true wall shear stress in the capillary, τ_w, by:

$$\tau_w = \frac{PD}{4(L + ND)} \tag{3}$$

Where N is the end correction factor for the imaginary extension of the capillary length.

By using Equations (3) and (2), shear stress versus apparent shear rate curves can be obtained.

Capillary analysis was conducted by extruding mixes through three different die lengths and at six different velocities. The extruder barrel had a diameter of 38.1 mm and a length of 125 mm, allowing approximately 120 ml of material to be extruded at a time. For each mix that was tested, eighteen experimental runs were made. Three die lengths (giving L/D = 1, 2 and 4) and six piston velocities, 0.2, 0.5, 1, 2, 3 and 5 mm/s, which correspond to extrudate velocities of 1.8, 4.5, 9, 18, 27 and 45 mm/s, respectively, were used. The rheometer was mounted in a closed-loop, MTS testing machine with a 24 kN load cell. Preliminary work showed that repeatable results were obtained from extruding the same mix in two different runs. Therefore, subsequent testing only involved one extrusion run per variable tested. The stiff cementitious dough was forced through the die at a constant displacement rate and the load and piston displacement were recorded.

Figure 6 presents an example of a rheometric curve obtained using capillary analysis. Yield stress (τ_0) was approximated using the lowest two data points and extrapolating to the y-axis. Using the differential viscosity versus apparent shear rate curve, an equilibrium viscosity ($\eta_{equilibrium}$) was defined as the differential viscosity at which the system equilibrated.
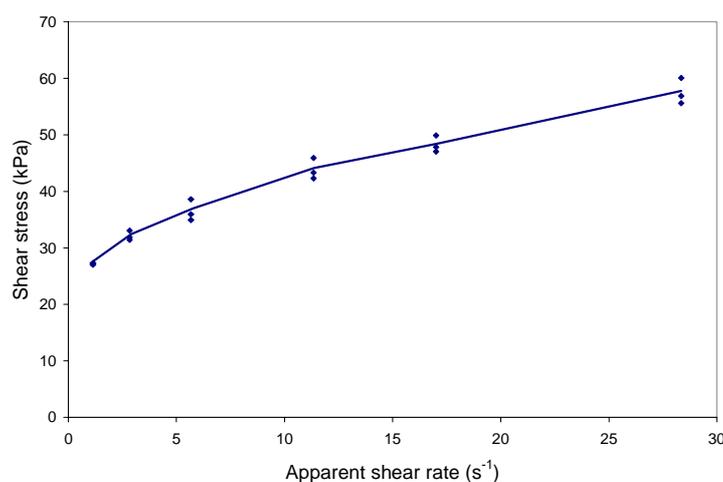
Figure 6. Example of rheometric curve obtained using capillary analysis (shown for W0.5)

The two rheological parameters obtained, τ_0 and $\eta_{equilibrium}$, were examined independently to see if either gave an indication of extrudability. However, no trends were observed. Figure 7 presents the two parameters plotted together, for both the extrudable and not extrudable mixes, and demonstrates that, when considered together, τ_0 and $\eta_{equilibrium}$, can be used to evaluate extrudability. Figure 7 suggests that an extrudable mix is one in which the yield stress is reasonably low (facilitating extrusion) and the equilibrium viscosity (probably related to thixotropy) is high.

It is interesting to note the relationship between these results and previous research. Benbow and Bridgewater also suggested that a low yields stress was needed for extrudability (Benbow 1993). In addition, the zone of extrudability (Figure 7), with low yield stress values and high viscosities, is similar to the zone of rheological parameters required for self consolidating concrete (Saak 2000), which requires a low yield stress and a high viscosity. The similarities with these rheological parameters suggest that both the yield stress and viscosity (related to cohesion) are important factors to describe flow behavior.
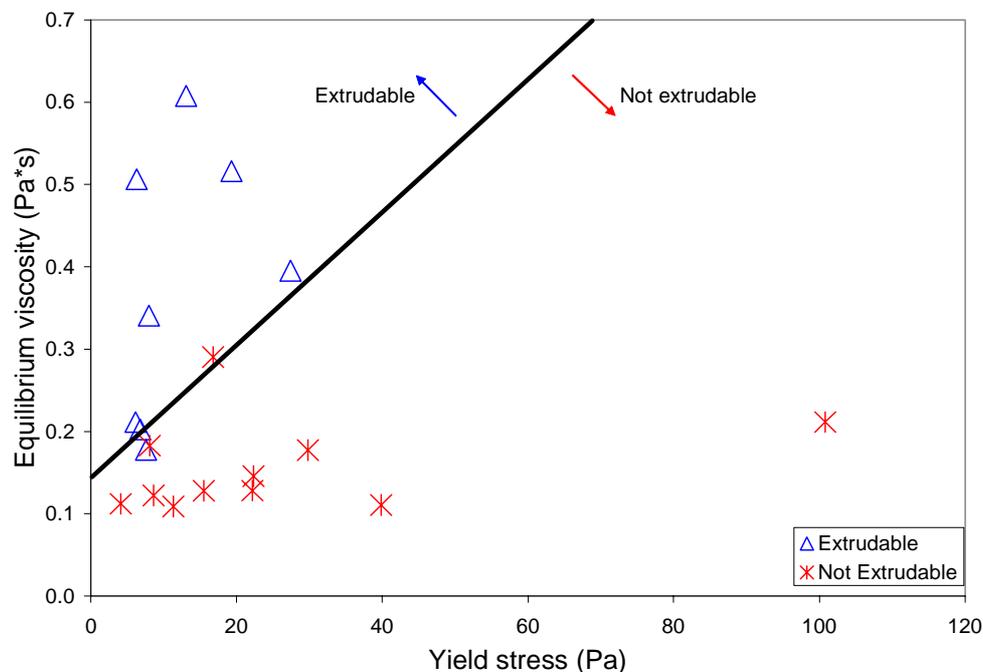
Figure 7. Equilibrium viscosity versus yield stress for extrudable and not extrudable mixes

Summary – Extruded HPFRCC

Extrusion can be used to produce HPFRCC for residential applications and offers a number of advantages over currently-used materials, including enhanced mechanical performance and durability. However, the cost of typical extrusion composites is high due to the expensive cellulose ethers that are used for processing. Clay binders were found to be suitable replacements for part of the cellulose ethers, significantly reducing the cost of the material. Capillary rheology was used to describe the fresh state properties for extrusion and showed that extruded HPFRCC have high equilibrium viscosities and low yield stresses.

CONCLUSION

Processing methods and parameters play a significant role in composite performance and the cost. In this work, the effect of processing on two different fiber-reinforced cement-based composites was examined: Hatschek-produced FRCB and extruded HPFRCC. The results indicate that processing does have a significant effect on composite performance. External pressure was found to improve the mechanical performance and freeze-thaw durability of Hatscheck-produce FRCB. The cost of extruded HPFRCC was significantly reduced by using alternate clay binders and capillary rheology was successfully used to link rheological parameters to extrudability.

ACKNOWLEDGEMENTS

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