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Importance of Processing in Advanced Cement-Based Products

by K.G. Kuder and S.P. Shah

Synopsis: The design versatility of cement-based composites continues to make them attractive for a variety of specialized applications. Advanced processing techniques, including the Hatschek process, extrusion, self-consolidating concrete and slipform-cast concrete paving, offer great promise for improving innovation in the modern construction world. However, to advance the state-of-the-art of cement-based products, the fresh state characteristics of these materials need to be well understood. Processing has a significant impact on composite performance, affecting fresh and hardened state properties as well as overall cost. In spite of its importance, relatively little is known about the relationship between processing and composite performance. Recent work at the Center for Advanced Cement-Based Materials (ACBM), headquartered at Northwestern University, has focused on developing a better understanding of this critical relationship. The role of processing on composite performance has been examined for a variety of advanced processing techniques, including the Hatschek process, extrusion, self consolidating concrete and slipform-cast concrete paving. The results indicate that overall composite performance can be enhanced by controlling fresh state properties. This paper presents a review of these studies and discusses ongoing research to link composite performance to microstructural changes.

Keywords: extrusion; flocculation; fresh state properties; Hatschek process; processing; self-consolidating concrete; slipform casting

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INTRODUCTION

Cement-based composites offer great design versatility due to their formable nature in the fresh state. As a result, a variety of different processing techniques can be used depending on the intended application. Processing can substantially influence the fresh and hardened state properties of cement-based composites, as well as overall cost. In spite of its importance, relatively little is known about the relationship between processing and composite performance.

Recent work at the Center for Advanced Cement-Based Materials (ACBM), headquartered at Northwestern University, has focused on the effects of processing on composite performance. A number of different processing methods have been studied, including the Hatschek process, extrusion, self-consolidating concrete and slipform paving. The influence of these techniques on composite performance has been systematically investigated. This paper presents a review of the results from these studies as well as discusses ongoing work to link composite performance to microstructural changes.

PROCESSING OF HATSCHEK-PRODUCED COMPOSITES

Introduction

The Hatschek-process is currently used to manufacture fiber-reinforced cement board (FRCB) for non-structural elements in residential construction. The composites are an attractive alternative to wood because they are more fire resistant, can better withstand fading, are not susceptible to insect attack and are more durable. Despite these advantages, use of Hatschek-produced FRCB in colder climates has been limited due to its vulnerability to freeze-thaw attack.

Hatschek process

Figure 1 presents a schematic of the Hatschek formation process. A dilute slurry of cement, silica, water and cellulose fibers is contained in a series of bins. Screens pass through these bins, collecting a monolayer of the material, which is then vacuumed to remove excess water. The monolayers are added to each other as the conveyor belt continues on to subsequent bins. This series of monolayers comes together to form a single layer of the FRCB. Multiple layers are gathered on the accumulator role until the desired thickness is attained. At this point, the composite is then autoclave cured. The resulting FRCB is a laminated composite that is composed of a large volume of cellulose fibers, approximately 30%.

The freeze thaw-durability of FRCB produced by the Hatschek process is poor due to its high porosity, the organic cellulose fibers that reinforce it and its laminated structure. To overcome these weaknesses, some manufacturers have started pressing the FRCB after it is formed, expelling excess water and possibly improving the interlaminar bond (ILB). Research has shown that applying pressure to FRCB improves its mechanical performance[1, 2, 3]. The effect of pressure on the freeze-thaw durability of FRCB was investigated, with particular attention to the role of the ILB[4].

Experimental program and results

Commercial Hatschek-produced FRCB that were 8 mm (5/16 inch) thick, reinforced with cellulose fibers and autoclave-cured were investigated. Immediately after the boards were formed, external pressure was applied at varying levels: 0, 10, 20 and 30 bars. Mechanical testing indicated that pressure increases the flexural strength, decreases flexural toughness (quantified as area under flexural stress – displacement curve) and increases interlaminar tensile strength.

Figure 2 presents the ILB as a function of pressure. ILB increases by 200% from 0 to 30 bars of pressure. Microstructural analyses were conducted using scanning electron microscopy and mercury intrusion porosimetry. These results suggest that the application of pressure improves the fiber-matrix bond and the interlaminar tensile strength by reducing the interlaminar space and the area between the fibers and the matrix[4, 5].

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[6]." Flexural performance and the interlaminar properties of the FRCB were evaluated after 50, 100, 150, 200 and 300 cycles. 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. Figure 3 presents the ILB strength as a function of freeze thaw cycles and indicates that there is a significant decrease in strength (at least 80% for all materials) after only 50 cycles. However, 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.

Visual observations also suggest that pressure treatments do improve the ILB and, consequently, the freeze-thaw durability of the FRCB. Figure 4 shows 20 and 30 bar materials that have been subjected to 200 freeze-thaw cycles during the three-point bend test. A delamination failure is seen with the 20 bar material, whereas the layers remain in tact for the 30 bar material.

Summary

Overall, the results indicate that modifications to the processing of FRCB can improve the performance of FRCB. 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.

PROCESSING OF EXTRUDED COMPOSITES

Introduction

Extrusion is an advanced processing technique that is used to produce high-performance, fiber-reinforced cementitious composites (HPFRCC). Extruded HPFRCC are excellent candidates to replace currently-used building enclosure products. Compared with conventional materials, extruded composites can be stronger, more durable, more ductile, more environmentally friendly, more cost effective, offer more design flexibility and improve safety in the event of natural hazards. In addition to enhanced mechanical performance, composites demonstrate a significant improvement in durability due to the high density that results from the extrusion technique[7, 8]. Despite the great potential of extrusion technology, it has not been widely adopted by the North America concrete products 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.

Extrusion technique

In the extrusion process, 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. Mix formulations and processing parameters (extruder barrel and die size, extrusion velocity, etc.) must be carefully designed to optimize fresh state properties. In the fresh state, extruded composites should be sufficiently soft to flow through the die (minimizing extrusion pressure), yet rigid enough to maintain shape upon exiting the die (shape stable). These fresh state properties will in turn affect hardened state properties, including fiber dispersion, composite density and the fiber-matrix bond. Currently, the fresh state properties of extruded composites are controlled using cellulose ethers. However, these materials are expensive. Research at ACBM investigated the effects of processing aids on the extrusion process using extrusion rheology, with an aim at reducing the overall processing aid cost[9].

Extrudability

Extrudability was evaluated by extruding open cross-sections, using a cellular die, with two cells, that had a total length of 25.4 mm (1 in) , a width of 15 mm (0.59 in) and a wall thickness of 3.25 mm (0.13 in). Specimens were extruded by a piston extruder at a rate of 1 mm/s (0.04 in/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. However, it is important to note that extrudability is related to extrusion velocity as well as to the shape being extruded. 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. The properties of the cellulose ethers and clays are given in Table 1 and Table 2, respectively. 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. Mixes were prepared using a planetary (Hobart) mixer. A piston extruder, mounted in a closed-loop, MTS hydraulic testing machine with a 24 kN (5.4 kips) load cell, was used with a barrel diameter of 38.1 mm (1.5 in) and a length of 125 mm (4.92 in).

Table 3 summarizes the extrudable mixes. In the absence of clay binders, half the amount of W was required compared to D. Figure 5 shows the effect of clay on extrudability. 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 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.

Capillary rheology

Capillary rheology was used to describe the fundamental flow properties of the extruded mixes. The 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.

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

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

Where P is the extrusion pressure (kPa or psi), V is the mean extrudate velocity in the capillary (mm/s or in/s), L is the capillary length (mm or in) and D is the capillary diameter (mm or in). End effects are taken into consideration using Bagley's end correction [10], 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.

Capillary analysis was conducted by extruding mixes through three different die lengths at six different velocities. 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 (0.008, 0.020, 0.040, 0.079 and 0.197 in/s) , which correspond to extrudate velocities of 1.8, 4.5, 9, 18, 27 and 45 mm/s (0.071, 0.178, 0.354, 0.709, 1.063 and 1.772 in/s), respectively, were used.

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 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 that the zone of extrudability, with low yield stress values and high viscosities, is similar to the zone of rheological parameters required for self consolidating concrete [11], 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.

Summary

Clay binders were found to be suitable partial replacements for cellulose ethers, significantly reducing the cost of the extruded composite. 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.

FIBER-REINFORCED SELF CONSOLIDATING CONCRETE

Introduction

Self consolidating concrete (SCC) is concrete that is highly deformable in the fresh state, flowing under its own self weight without segregating. This technology is suitable for applications in which complicated shapes are being formed and in cases where congested reinforcement is used. SCC eliminates the need for external vibration, reducing labor and energy costs. Furthermore, when properly designed, the hardened state properties of SCC can be superior to conventional concrete, with a more homogeneous matrix and better bonding to reinforcement. The extension of this technology to fiber-reinforced self consolidating concrete (FRSCC) offers great promise, likely producing composites that exhibit superior mechanical performance and durability due to the enhanced fiber-matrix bond and greater compactness. Recent research efforts have focused on developing methodology to design fiber reinforced SCC to optimize fresh state properties, which should in turn enhance hardened state properties[12].

Rheological paste model – solid skeleton grading

The "rheology of paste" model developed by Saak and colleagues[13] has been modified to account for fiber reinforcement. The model determines the minimum volume of cement paste needed to allow for the required deformability and segregation resistance by analyzing the optimum grading for the solid skeleton system. The average aggregate spacing, d_{ss}, which is twice the thickness of the excess paste layer coating the aggregates, is determined according to:

$$d_{ss} = d_{av}\left[\sqrt[3]{1+\frac{V_{paste}-V_{void}}{V_{concrete}-V_{paste}}}-1\right] \tag{4}$$

Where d_{av} is the average diameter of the solid skeleton particles, V_{paste}, V_{void} and $V_{concrete}$ are the volumes of the paste, voids and concrete, respectively. Fibers are treated based on an "equivalent spherical particle" fraction based on the specific surface of the fibers such that an equivalent diameter, $d_{eq\text{-}fibers}$, can be calculated by:

$$d_{eq-fibers} = \frac{3L_f}{1+2\dfrac{L_f}{d_f}}\frac{\gamma_{fiber}}{\gamma_{aggregate}} \tag{5}$$

Where d_f and L_f are the diameter and length, respectively, of the fibers, γ_{fiber} is the specific weight of the fibers and $\gamma_{aggregate}$ is the weighted average of the specific weight of all the aggregates. Finally, the average equivalent diameter of the solid particles, d_{av}, is determined:

$$d_{av} = \frac{\sum_i d_i m_i + d_{eq-fibers} m_{fibers}}{\sum_i m_i + m_{fibers}} \tag{6}$$

Where d_i is the average diameter of the aggregate fraction (defined as the average size opening of two consecutive sieves), m_i is the mass of the aggregate fraction and m_{fibers} is the mass of the fibers.

Rheological characterization
The rheology of the cement paste is characterized using the mini slump-cone test and rheometer tests. Mini slump-cone tests were used to determine flow diameter (related to yield stress) and rheometer tests employing a concentric cylinder geometry, along with equilibrium shearing protocol, indicated the apparent viscosity. These two parameters were considered jointly using the ratio of the flow diameter/apparent viscosity.

Application of model
The optimum flow diameter/viscosity ratios for self compactability were determined as a function of the average aggregate spacing, d_{ss}, as is shown in Figure 8. Three zones are shown: segregation, allowable (self consolidating) and poor deformability. These zones were determined based on experimental data using regression analysis. Fiber dispersion was analyzed by evaluating the amount of fibers in the top, middle and bottom thirds of cylindrical specimens and the effects included in the model based on segregation tendencies. The model lines show that minimum and maximum flow diameter/apparent viscosity of the cement paste are needed to achieve sufficient deformability and prevent segregation, respectively. As the average aggregate spacing decreases, the required flow diameter/viscosity increases, suggesting a greater flow diameter and lower viscosity are needed for self consolidation. Furthermore, a larger range of possible flow diameter/viscosity exists for larger average aggregate spacing, indicating more design flexibility.

The concept of optimum limiting values of flow diameter/viscosity for varying average aggregate spacing can be used to design FRSCC. Rheology tests are used to analyze the cement paste, while the grading of the optimum solid skeleton is determined through the measurement of the average diameter of the particles and the void ratio. The correlation between cement paste rheology and solid skeleton grading is then determined. Based on this relationship, the allowable values for the average spacing of solid particles as well as the cement paste rheology and solid skeleton grading can be determined.

The proposed model was used to design three FRSCC containing the same cement paste and 50 kg/m^3 of steel fibers (Dramix 65/35), but which fell within the different mix design zones – poor deformability, self-compacting and poor segregation resistance for mixes A, B and C, respectively.
Figure 9 presents the results of this analysis. The cement paste had a flow diameter/viscosity of 650, V_p varied from 0.32, 0.36 and 0.40, and d_{ss} ranged from 0.272, 0.360, and 0.460, corresponding to mixes A, B and C, respectively. Slump tests, a visual segregation index and the T_{50} slump flow test (time required to reach 50 mm diameter spread) were used to assess and verify that the intended fresh state properties were achieved. These results indicate that a rational method can be used to design FRSCC.

Summary
A methodology has been developed to design FRSCC that considers the effect of the cement paste rheology and the solid particle skeleton (considering the contribution of the fibers via an equivalent spherical particle fraction) on fresh state properties and fiber dispersion. The results indicate that through the use of optimum limiting values of slump flow diameter/viscosity for varying average aggregate spacing, mixes that exhibit self consolidating behavior can be achieved.

SLIPFORM PAVING

Introduction
Currently, concrete pavement is placed using a slipform paving machine. Slipform paving is a continuous process in which the concrete is poured, consolidated and finished as the paving machine moves at a constant rate over the fresh concrete. The concrete is a low slump concrete (less than 5 cm [2in]) that maintains its shape without edge support once placed. To consolidate the concrete, equally spaced internal vibrators are used. However, if the frequency is not properly selected, or if the paving machine moves too slowly, the concrete can become over-vibrated. The result of this over vibration is aggregate segregation, a reduction in air-entrainment and vibrator trails that are a source of inherent weakness. To resolve this issue, a low compaction energy concrete has been designed that does not require vibration[14]. The concrete demonstrates both a high workability and a high shape stability.

Strategy

Figure 10 depicts the strategy adopted to produce low compaction energy concrete. A typical SCC mix design (i.e. one with high workability) was taken and modified using various mineral (fly ash, clays) and chemical (plasticizers, viscosity modifying admixture) admixtures to achieve shape stability. Flowability was evaluated using a flow test and a drop table test. Results from the drop table test were also used to assess shape stability. In addition, compression strength tests were conducted on freshly-mixed concrete samples to determine green strength. Finally, a model paver that was constructed to simulate the slipform paving process in the laboratory was used to evaluate the compaction ability of the concrete.

Experimental results

The effect of plasticizers and fine materials on the flowability and green strength of concrete was determined. The results are presented elsewhere, but summarized here[14]. The amount of additive needed to achieve a flow ratio of zero (i.e. shape stability) was obtained by beginning with a typical SCC mix design and then reducing or increasing the amount of plasticizer or fine material, respectively. Flowabilty and green strength of the shape stable mixes were then evaluated using the drop table tests. These results show that by properly tailoring the type and amount of plasticizer and fine material(s) in a mixture, both shape stability and flowability can be achieved.

The relationship between green strength and the flow ratio after 25 drops is presented in Figure 11. As expected, a general trend exists whereby green strength reduces dramatically with the flow ratio. However, it is interesting to note that some mixes do not follow this trend. These mixes contain the naphthalene-based plasticizer, or fine materials (clay or fly ash). Further research is needed to understand this trend.

Figure 12 shows different mixes that were cast using the laboratory-scale slipform paver for four different mixes: typical slipform, modified plasticizer (naphthalene-based), and two with clay additives. The last three of these are mixes that were shown to have desirable flowability and shape stability. These composites demonstrate excellent shape stability, edge stability and flowability when cast without vibration.

Summary

Concrete mixes were successfully designed for slipform paving applications that can be cast without applying vibration. Mixes were developed using the concept of SCC and modified to obtain shape stability. The effect of additives on the flowability and shape stability was evaluated. A good correlation was observed between high flowability, shape stability and the ability to be formed without vibration using a simulated slipform paver.

FUTURE DIRECTIONS: THE STUDY OF FLOCCULATION

Introduction

Advanced processing techniques rely on microstructural changes that occur during the first few hours of the life of concrete. Applications such as extrusion, self consolidating concrete and slipform casting require cementitious systems that are highly flowable yet shape stable. Ongoing work at ACBM is investigating how admixtures, including plasticizers, clays and fly ash, affect the flocculation behavior of cement paste. It is hypothesized that flocculation is related to shape stability (green strength).

Compressive rheology and green strength

The flocculation behavior and green strength of different cement pastes were investigated. Flocculation behavior was examined using the centrifugal approach in which the compressive yield stress is measured as a function of local particle volume fraction. In addition, green strength was measured. Six different mixes were investigated: control (CM), cement and Class C fly ash (FA), cement and napathalene-based plasticizer (HW), cement and purified magnesium alumino silicate clay (C1), cement and kaolinite, illite, quartz clay (C2) and cement and kaolinite clay (C3). The water/binder ratio was approximately 0.40.

Figure 13 presents the compressive yield stress of the six different mixes as a function of local volume fraction. These results show that the HW and FA mix are more compressible than the control (CM), whereas C1, C2 and C3 have a higher compressive yield stress. These findings correlate well with green strength testing, which shows that the HW and FA have green strengths that are lower than the CM, while the C1, C2 and C3 have a greater green strength than the CM. Thus, flocculation appears to be related to green strength.

Summary
Enhanced fresh state properties have been achieved by controlling workability and green strength. The flocculation of particles in the cement paste mixes was studied using compression rheology and shows good agreement with green strength. These results suggest that flocculation is an important mechanism to understand and control for the processing of advanced cement-based composites.

CONCLUSION

Recent work at the Center for Advanced Cement-Based Materials (ACBM), headquartered at Northwestern University, demonstrate the potential of cement-based composites to be used in a variety of specialized applications. The success of these new technologies relies on advanced processing techniques in which the fresh state properties must be controlled and optimized. The role of processing on composite performance has been examined for the Hatschek process, extrusion, self consolidating concrete and slipform-cast concrete paving. The results indicate that overall composite performance can be enhanced by controlling fresh state properties. Furthermore, ongoing research suggest that by studying the behavior of the microstructure at early ages, in particular with respect to flocculation, the mechanisms by which enhanced fresh state properties are achieved can be understood.

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Table 1 – Properties of cellulose ethers

Trade name	Producer	Chemical designation	Specific gravity g/cm^3 (lb/in^3)	Viscosity Pa*s (psf*s)
Methocel 4fm*	Dow Chemical Company	Hydroxypropyl methylcellulose	1.3 (0.05)	2.3-3.8 (0.05-0.08)
Walocel M-20678**	Wolff Celulosics	Methylhydroxyethy l cellulose	3.2-4.0 (0.12-0.14)	10.4-12.4 (0.22-0.26)

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

Table 2 – Properties of clay binders

Trade name	Producer	Mineral Composition	Mean particle size μm (μin)
Concresol	Stephan Schmidt Group	kaolinite (45%), mica (35%) and free silica (20%)	≈ 0.5 (20)
Metamax HRM	Engelhard	calcined kaolinite	1.2 (47.2)

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	

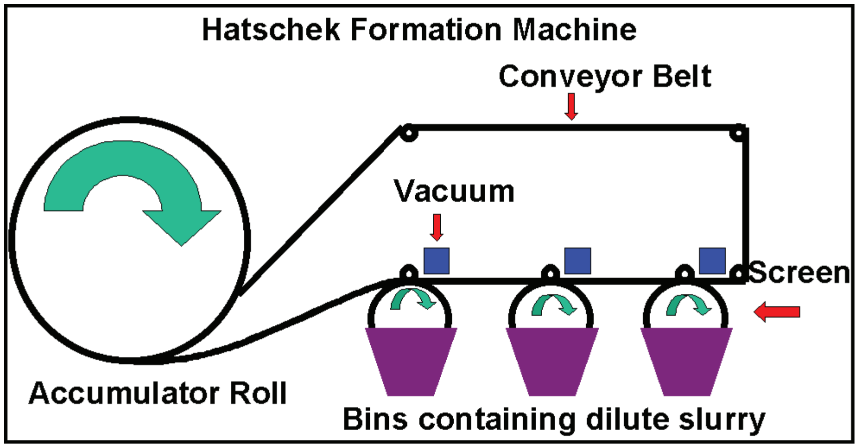

Figure 1 – Schematic of Hatschek slurry-dewatering process.[4]

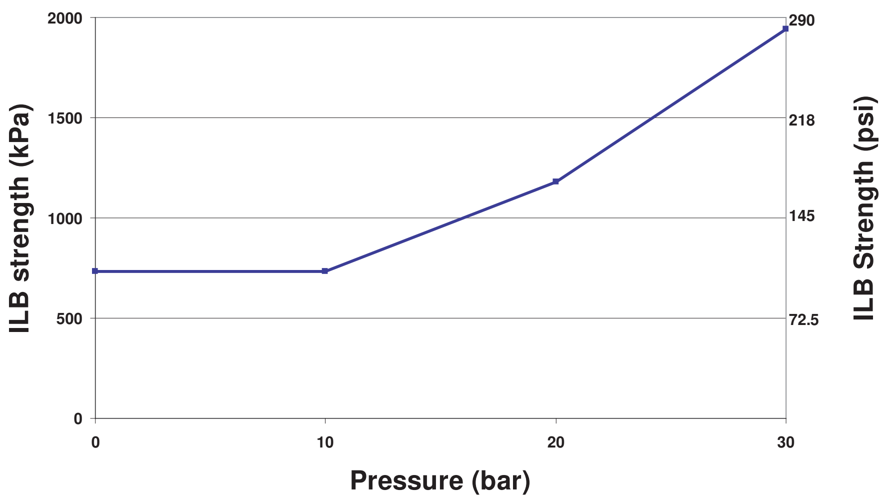

Figure 2 – Interlaminar tensile bond (ILB) strength as a function of pressure.[4]