EFFECTS OF DIFFERENT FIBERS ON THE PROPERTIES OF COMPOSITE SILICATE BOARDS

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ABSTRACT

Fibers play an important role in the preparation and the properties of the composite silicate boards. In this paper, fibers from two different varieties of trees were used in the boards. The cellulose, hemi-cellulose, and lignin contents in different fibers were investigated, as well as the morphology. The effects of different fibers on manufacture process and properties of the boards were also considered. The results show that different fibers have a significant effect on the resistance of water filtration and absorbability to cementitious material in the boards. Because fibers can reduce the mass loss of cementitious materials through absorption in the process of water filtration, the boards will be comprised by fiber layers and cement layers, and fine fiber will lead to longer water filtration time. Through these researches, the utilization of fibers can be controlled to improve the properties of composite silicate boards.

KEYWORDS:

Fiber, Cementitious material, Absorbability, Resistance of water filtration, Composite silicate boards

INTRODUCTION

With the growth of global population, the density of human beings is increasing rapidly as the constant area of land, which makes longitudinal development of the cities combined with continued urbanization. Therefore, the trend toward higher buildings just like Taipei 101 and over 800 m high Burj Khalifa in Dubai has been already identified in 1982 as one of the so-called "megatrends" (Picker, 2014). However, higher buildings lead to the higher requirement of the properties of modern building materials.

Composite silicate board is a kind of building plate, which is mainly comprised of fiber, silicate cementitious materials, and other functional fillers. The boards were produced with the mixed materials through slurry, moulding, autoclave maintenance, and drying. Fiber cement boards have superior performance to concrete, as they contain more fibers and are thin. Therefore, composite silicate boards have been widely used in various fields of the construction industry such as internal wall panels, external wall panels and fire protection boards.

In composite silicate boards, fibers play an important role for the strength of the boards. The fibers need enough tensile strength and toughness, as long as large specific surface area to improve the absorbent properties of cementitious materials, which can increase the gripping force between fiber and cementitious materials. Fibers can be divided into plant fibers and artificial fibers. Plant fibers have rougher surface and is more easily separated by frictional forces, such as grinding operation or milling operation,. Soplant fibers have much larger specific surface area comparing with artificial fibers of the same length and sectional area, which improves the absorbent properties of cementitious materials. Fibers from different kind of plants are also quite different. For example, pine is a kind of wood that grows all over the world. However, the fibers form the radiata pine which mainly grows in Canada, North Europe, and Northeast of China, have quite different properties compared with that form the spruce growing in Chile and South of China. The differences of growth periods between the two kinds of pines lead to different degrees of length and thickness, which affect much of the application performance in composite silicate boards.

Therefore, we mainly choose southern and radiata pines as the fibers in composite silicate boards. The contents and morphology in different fibers were investigated through field emission scanning electron microscopy (FESEM). Meanwhile, the effects of different fibers on manufacture process and properties of the boards were also considered. Morphology change and the interfacial bond between fiber and cementitious materials were investigated with FESEM. Through these researches, the utilization of fibers can be controlled to improve the properties of composite silicate boards.

EXPERIMENTAL

In this paper, spruce (WestRock, USA) and radiata pine (Arauco, Chile) were used to provide the fibers in composite silicate boards. According to different handling methods, they can be divided into straw boards and white boards (bleached), individually. Cementitious materials contain cement, silica, and portlandite.

The handling process of the wood pulp includes soaking, shredding, and refining. Fresh water and circulation water of the process (pH value of about 14) are respectively used in refining process to investigate the effect of pH value on beating degree of the fibers. The circulation water is from the production line. It is the supersaturated solution of calcium salt and calcium hydroxide, which is from the dissolution of lime and calcium silicate hydrate. Trace magnesium and other cationic also exist in the water. Fibers with different beating degrees and wet weight were obtained to prepare the boards through slurry, moulding, autoclave maintenance, and drying. The morphologies of fibers and cementitious materials in the boards were investigated through FESEM (JEOL JSM 6700F).

ANALYSIS OF DIFFERENT FIBERS

Typical fibers from radiata pine and spruce were chosen to investigate the components and morphologies. Circulation water and fresh water were respectively used to handle the wood pulp.

Components analysis

To investigate the differences of fibers from different kinds of wood, the components of the fibers have been analysed with Goering and Van Soest method (Goering, 1970). According to dissolution conditions of different contents in fibers, acidic detergent, neutral detergent, and sulphuric acid were used to selectively dissolve different fraction. The contents of cellulose, hemi-cellulose, and lignin were obtained through the dissolution amount of fibers, and the results are shown in Table 1:

Effects of refining conditions on the content of fibers are quite different. For radiata pine, cellulose content is lower after refining with fresh water than circulation water. It means the alkaline solution is beneficial for the protection of cellulose in solid phase, and lignin is easily dissolved into the solution in an alkaline environment. However, the properties of lignin in spruce are reversed. After refining in circulation water, content of lignin remains 17.23%, much higher than 5.47% in fresh water. The alkaline solution ia benefit for the protection of lignin in spruce. Cellulose contents in spruce are nearly the same under two refining conditions.

Table 1 – Contents of different kinds of fibers

Contents (wt %)		Cellulose	Hemi-cellulose	Lignin	Ash
Radiata pine	Circulation water	71.40	5.83	7.98	0.10
	Fresh water	62.23	7.11	12.76	0.37
Spruce	Circulation water	73.71	6.09	17.23	0.94
	Fresh water	70.12	5.26	5.47	0.58

Surface morphology analysis

Fibers are very important in composite silicate boards as its tensile strength and flexibility to improve mechanical properties of boards. Most of composite silicate boards producers have no ability to manufacture wood pulp. Therefore, the pulp was purchased and used after a series of treatments of soaking, shredding, and refining to meet the requirement for product manufacture. In the treatment process of pulp, the volume of fiber will expand after soaking, which can enlarge the effective friction area between the fibers and increase shredding and refining efficiency of fibers. Shredding is pre-treatment process to dissolve the pulp into fiber with certain concentrations. In the process of shredding, higher concentration is usually needed to improve the efficiency, provide more friction and collision between the fibers, and reduce the damage to the fiber from the machine. Refining is the process of more intense treatment of the pulp. It decides the fiber quality, which will directly influence the manufacturing and property of boards. There are three key points in pulp refining process including the flow rate (match to the relevant concentration), the pressure between the refiner plates, and full use of effective grinding area (the actual area). Through the control of listed points, fibers with reasonable fineness and length can be obtained by the interaction between the fibers, and it will meet the requirement of manufacturing technology and property of final products.

In composite silicate boards, large specific surface area of fibers is needed to increase the gripping force between fiber and cementitious materials. However, plant fibers are easily fibrillated, specific surface area is hard to be accurately measured. Beating degree is used to indirectly present the specific surface area. Higher beating degree leads to higher fibrillation rate of the fiber, which means better absorbance for the cementitious material. The beating degree has a certain association with the morphologies of fibers. Therefore, the morphologies of four kinds of fibers were studied, and the images are shown in Figure 1:

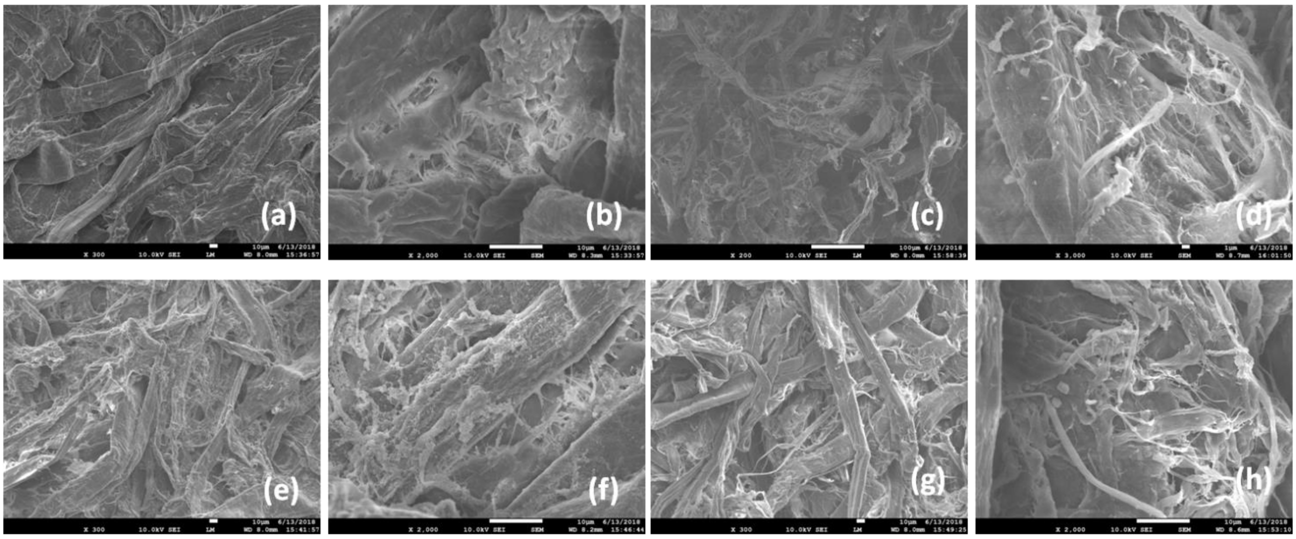

Figure 1 – Morphologies of different pulps handled with two refining methods.
(a), (b): Radiata pine, refining with circulation water. (c), (d): Radiata pine, refining with fresh water.
(e), (f): Spruce, refining with circulation water. (g), (h): Spruce, refining with fresh water.

It can be found that the pulp does not obviously change the state of the fiber. Meanwhile, the separation and dissolve fibrillation are also apparent after refining. Beating degrees of radiata pine refining with circulation water and fresh water are 46.50% and 34.67%, respectively. It means the increase of beating degree is useful for the removal of lignin. The same result can be obtained from spruce. The beating degrees of fibers refining with circulation water and fresh water are 41.67% and 47.00%, respectively. It can also be seen from the morphologies of fibers. Thinner fibers in Figure 1(b) and (h) are more than the others. It means the increasing of beating degree can improve the removal of lignin, and it makes fibers thinner.

RELATION BETWEEN BEATING DEGREE AND WET WEIGHT OF FIBERS

Improvement of beating degree is realized through the interaction between mechanical grinding and fiber itself to generate fibrillation. But the mechanical grinding can also easily result in short cut of the fiber, which will decrease the strength and flexibility of boards seriously. Therefore, the length of fibers is also an important parameter for the utilization of fibers, which can be reflected by wet weight. Beating degree was measured according to GB/T 3332-2004 (Schopper-Riegler method). Wet weight of fibers was obtained with mechanical grinding machine before beating degree measurement. The amount of long fibers stay on the copper frame of the machine is the wet weight, and beating effect is related to the wet weight. Therefor in order to interpret the effect of beating on the fibers it is important to consider wet weight and beating together. The beating degrees and wet weights of different fibers from radiata pine were investigated under different refining time, which can be seen in Figure 2.

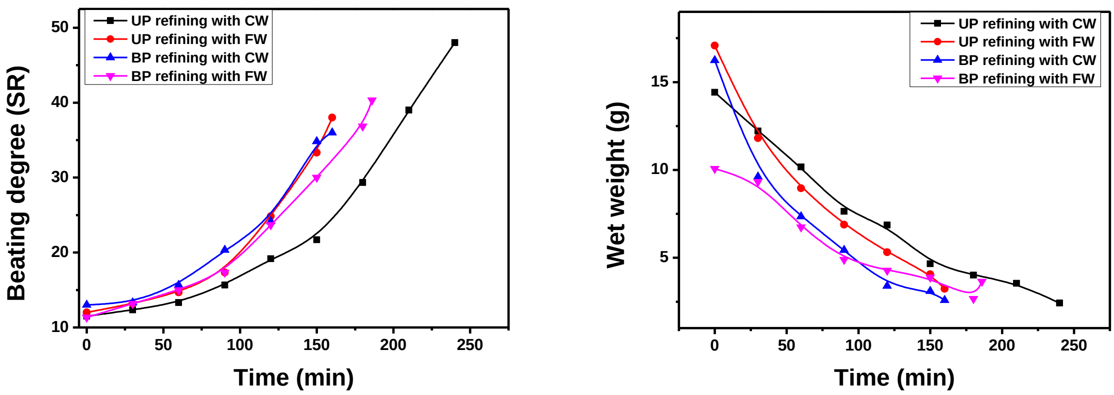

Figure 2 –Beating degree and wet weight versus refining time of different radiata pine fibers.
UP: Unbleached pulp. BP: Bleached pulp. CW: Circulation water. FW: Fresh water.

It can be concluded that the efficiency of refining is low before the beating degree reaches 22±2SR, for both circulation water (PH is close to 14) and fresh water (PH is close to 7) used to refine the radiata pine pulp. The beating degree increases about 2-3SR every half hour, and it grows rapidly with the increasing of 7-9 SR every half hour when the beating degree reaches 22±2 SR. The reason lies on that the fibers are long and thick in the initial stage of refining (which can be interpreted from the wet weights). The fibers are easily twined and rolled, unevenly dispersed, which leads to low fluidity and uneven pressure of the fibers because of constant starting pressure. Meanwhile, it also decreases the refining efficiency as the inconformity of effective contact area between fibers and refiner plates. With the continuous refining, the fiber gradually becomes finer and shorter. They are more easily fibrillated and show better fibrillation. The tangled fibers disintegrate to single small ones, which makes the fiber flow increase. The pressure becomes even and the efficiency of refining increases greatly.

Through the comparation of beating degree at the same time, it also can be found that the refining efficiency of unbleached pulp is higher with fresh water than circulation water. It means the neutral pH value is beneficial for refining efficiency comparing with the alkaline solution. However, the efficiency of refining is nearly the same with circulation water and fresh water for bleached pulp. When the circulation water is used for refining, the efficiency of unbleached pulp is higher than bleached one at the same time. With fresh water, the efficiencies of unbleached pulp and bleached one are almost equal.

Summing up, the refining efficiency will be higher by using neutral water for unbleached pulp. Bleached pulp can be refined by the alkaline water (circulation water), which will decrease utilization amount of water.

The relation between beating degree and wet weight was studied, which is shown in Figure 3:

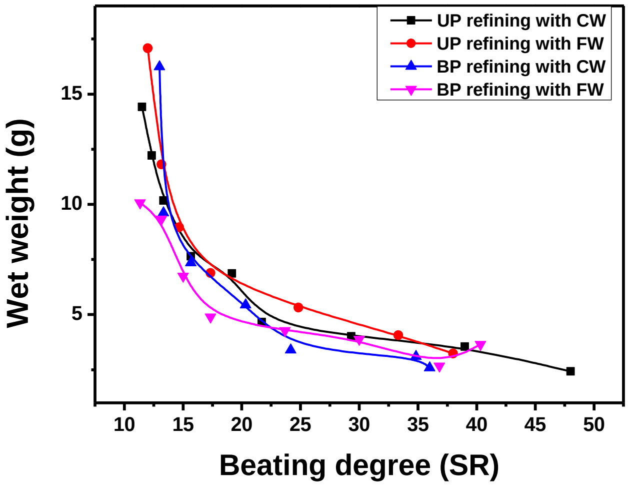

Figure 3 – Beating degree versus wet weight of different fibers.
UP: Unbleached pulp. BP: Bleached pulp. CW: Circulation water. FW: Fresh water.

pH value of water used for refining has no obvious influence to the relationship between wet weight and beating degree. Meanwhile, the relationship of beating degree and wet weight is the same during refining unbleached pulp and bleached pulp with Hollander, which is a typical beating machine used for both asbestos and wood fiber. The relation is affected by refining method. Refining process of unbleached and bleached pulp can be divided into 3 stages according to the wet weight value:

(1) In first stage, wet weight value is larger than 8. The beating degree increased a little, but wet weight drops seriously. The increasing of beating degree mainly relies on the short cutting (as shown in Figure 4) of fibers in this stage. A certain amount of fibers with suitable length is obtained for second stage, and here is lowest effect on the increasing of beating degree.

(2) In second stage, wet weight value is larger than 4 and lower than 8. Wet weight of fibers declines slower than first stage, and the beating degree increases quickly. The increasing of beating degree does not only rely on the short cutting of fibers, but also on the friction between fibers. A certain amount of fibers with suitable length and fineness is obtained for second stage, so this stage has great effects on the increasing of beating degree.

(3) In third stage, wet weight value is lower than 4. Wet weight of fibers declines much slowly, and the beating degree increases quickly. The increasing of beating degree mainly relies on the friction between fibers, and separation and fibrillation is realized. It has the most important effect on the increasing of beating degree.

Therefore, the beating degree increased mainly by the interaction (friction and collision) between fibers, and the fiber cutting does not have obvious effects on beating degree.

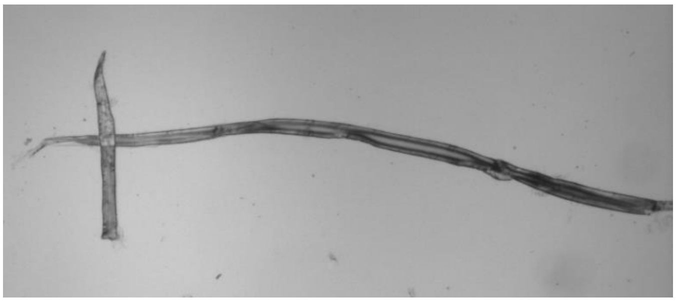

Figure 4 – Contrast of typical short cutting fiber (vertical) and normal fiber (horizontal) observed by microscope.

ESTABLISHMENT OF WATER FILTRATION RESISTANCE

Water plays an important role in the production of calcium silicate boards, and it makes the cementitious materials, fibers and different kinds of additives uniformly dispersed to form the slurry. The slurry dehydrates smoothly through filtration water fabric (felt) and forming drum to form a cement layer, and then stacks into boards. As the medium of component reactions, water also provides better way to transfer the energy to composite silicate boards and offers needed substances for the reactions.

Water acting as the carrier is not required during the reaction, while the water acting as reaction medium remains. Thus, unnecessary part of water will be dehydrated in the production process. During the process of dehydration, the water suffers certain resistances while it goes through the cement layer and filtration water fabric (felt) because of flow and filtration resistance. Dynamic resistance and inherent resistance exist in dehydration process. The main cementitious materials in the slurries are silica and calcium hydroxide. Dynamic resistance is formed when silica, calcium hydroxide, cement with the amount of less than 10%, fibers, and different kinds of additives gather together to form a cement layer, while inherent resistance is the dehydration resistance origins from filtration fabric or felt in the equipments. The filtration system is comprised of bottom web (pure polyester) and fabric, which used pure nylon needle woven felt with the surface density of 700 g/m^2. By adjusting the percentage of fiber in formula under certain beating degree and the time for filtrating same volume slurry, effects of fiber on filtration resistances can be verified, as shown in Figure 5.

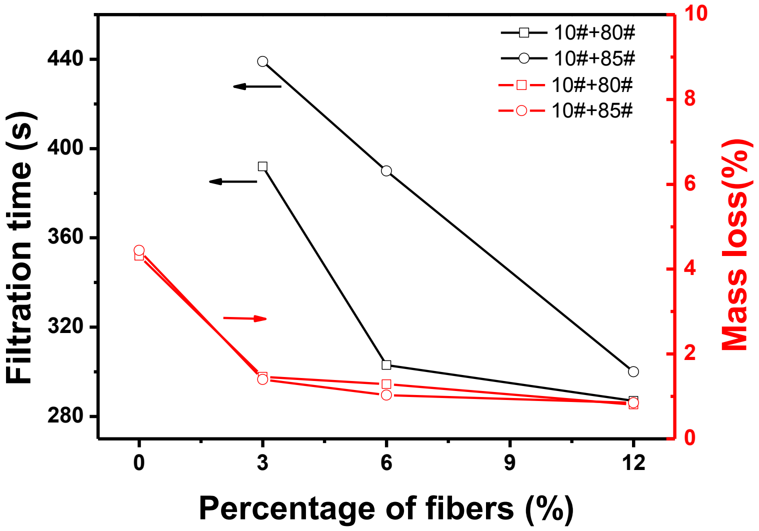

Figure 5 –Filtration time and mass loss amount versus percentage of fibers.
10#-filtration number. 80#, 85#-fabric number.

If there is no fiber in this process, the filtration resistances of cementitious materials are insufficient. Most of the slurry (cementitious materials) will pass the felt, and the loss amount is almost equal to the additive amount in the process. Quite little slurry remains on the felt. If there is no fiber in practical manufacturing, it is impossible for the felt to retain the cement layer. Therefore, during the process of dehydration of slurry, the fiber plays a key role in the loss of cementitious materials and establishment of cement layer.

The percentage of fiber in formula has effects on the filtration as well. More time is required to filtrate the same volume of water with less fiber. It can be explained that less fiber means more cementitious materials. More cementitious materials stick to the fiber, and higher sedimentation rate for this cementitious and fiber materials will be improved. Fiber can hardly pass but remain on the felt. When cementitious materials fail to stick to the fiber, they pass the felt as well as passing the fiber which sticks with cementitious materials. But the space between them is too small to get through. Cementitious materials will remain on the felt, which makes a smaller space for the water to get through. Less fiber, higher sedimentation rate will be for this cementitious and fiber materials and tighter dispersion on the felt with longer filtration time; more fiber, shorter filtration time. The percentage of fiber also affects the loss of slurry. Higher percentage of fiber will decrease the amount of cementitious materials that are not absorbed on the fibers. So the loss amount of cementitious materials decreases before the fiber which sticks cementitious materials arrive to the felt.

The change of beating degree will affect dehydration effect under the same conditions of concentration of slurry, percentage of formula, and particle size of cementitious materials. The result is shown in Figure 6.

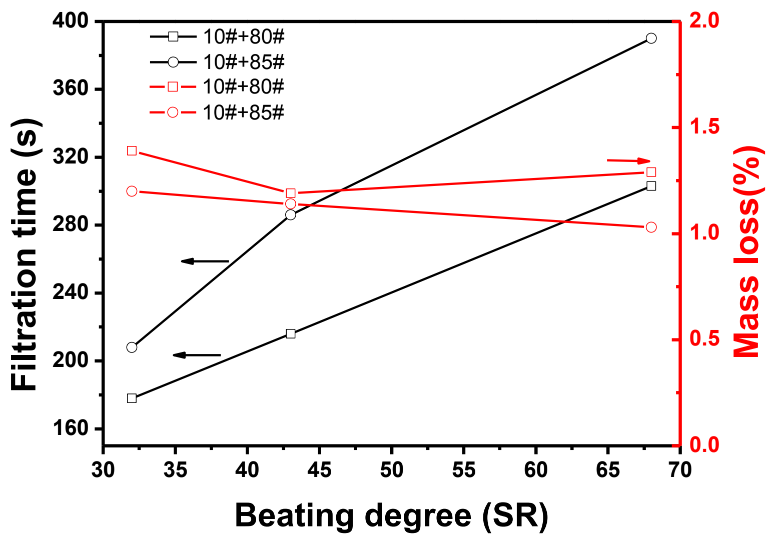

Figure 6 –Filtration time and mass loss amount versus beating degree of fibers.
10#-filtration number. 80#, 85#-fabric number.

Higher beating degree makes longer filtration time and less mass loss, because higher beating degree means higher specific surface area of fiber and stronger ability to absorb cementitious materials. There are less free cementitious materials that are not absorbed on fibers, and it accelerates the sedimentation rate. On the other hand, the change of the bore diameter of the felt also influences the dehydration process, which can be seen in Figure 5 and Figure 6.

Smaller diameter of filtration fabric means fewer amounts of mass loss and longer filtration time for same volume of water. More solid residue stays on the fabric, which makes less space for running off of water and bigger resistance. Therefore, the filtration is decided by filtration felt and the inherent property of slurry, and the property of the slurry depends on the percentage of fibers and its beating degree. It is deciding effects on the establishment of powder layer and boards formation in Hatscheck production. When the cement layer arrives at the forming drum, it is pressed by the forming drum and breast roll, and solid residue will be dehydrated again.

The filtration resistance of slurry is mainly affected by its property and the felt. In the slurries, fibers are covered by cement. When the slurries dropped on the felt, the cement on the surface facing to felt may be detached from the fibers according to the pressure. It leads to the insufficient encapsulation of the fibers, which will cause the formation of fiber layers. When the vacuum degree is enough high, or water content of slurries is over 80 %, the phenomenon is significant (Figure 7). Meanwhile, fiber layers and cement layers are difficult to join together. Different contents of fibers in different layers can be easily observed. Therefore, formula, proportion, particle size, fiber beating degree and wet weight, filtration fabric and felt are the main source of filtration resistance and restrict each other in the manufacturing process of composite silicate boards.

Figure 7 – Typical boards with separated fiber layers and cement layers.

CONCLUSION

Fibers from radiata pine and spruce were analysed to characterize the content and morphology. The effects of fiber properties on manufacture process and properties of the boards were also considered. The results are listed as follows:

(1) Alkaline refining water may increase cellulose content in radiata pine fibers, and which has little effect on that of spruce fibers. The increasing of beating degree may decrease lignin content in fibers.

(2) In refining process, neutral water is beneficial for refining efficiency of unbleached pulp, and alkaline water can be used in refining bleached pulp to improve water utilization rate. Improving of beating degree can decrease the mass loss of cementitious materials, which is achieved through the interaction (friction and collision) between fibers but not short cutting of fibers.

(3) In filtration process, formula, proportion, particle size, fiber beating degree and wet weight, filtration fabric and felt are the main source of filtration resistance, and the properties are influenced each other. Meanwhile, high vacuum degree and water content may lead to separation of fiber layers and cement layers.

Through these researches, the utilization of fibers can be controlled to improve the properties of composite silicate boards.

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FUNCTIONALLY GRADED FIBER-CEMENT: EXTRUDABLE MIXTURES FOR GRADATION OF HATSCHEK-MADE CORRUGATED SHEETS

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ABSTRACT

In this work, the functionally graded materials concept is linked to the Hatschek technology to enhance the performance-to-cost ratio of corrugated fiber-cement sheets. Extrudable composites at a high shear rate were developed for application between the layers of Hatschek-made corrugated sheets. Mixtures with a polyvinyl alcohol (PVA) fiber content of 1, 2, and 3% or alkali resistant (AR) glass fiber content of 2, 3, and 4%, by volume, were subjected to high-shear-rate extrusion tests and used for the preparation of specimens. After 28 days, the specimens were subjected to direct tensile tests. The content and type of fiber played an essential role in the extrusion pressure. The composites with 4% AR glass fiber content exhibited an average tensile strength of 12.0 MPa, whereas those with 3% PVA fiber content exhibited and an average tensile strength of 7.5 MPa. These mixtures were successfully applied in an exploratory preindustrial test.

KEYWORDS:

Fiber-cement; functionally graded materials; high-shear-rate extrusion; PVA fibers, AR glass fibers

INTRODUCTION

Functionally graded materials (FGMs) present at least one property which gradually vary through their volume, in a controlled way to reach the desired performance. Bamboo is a classic example of FG biomaterial which is structured by a natural process so that the fiber content gradually varies along its thickness, conferring greater mechanical strength in the regions where the stresses are more intense under wind loads (Bruck et al., 2002; Amada et al., 1997).

About 50 years ago, Bever and Duwez (1972) recognized that human-made composites could be designed and produced by locally varying the characteristics or content of the dispersed phases and the composition or microstructure of the matrix to obtain composites with improved performance in several applications. Because of numerous advantages over homogeneous materials, FGMs have been employed in several branches of science and technology (Miyamoto et al., 1999; Neubrand and Rödel, 1997). Nowadays, the FGM concept has been applied to cementitious composites (Stroeven and Hu, 2007; Dias et al., 2008; Shen et al., 2008; Dias et al., 2010; Toader et al., 2017). The FGM concept has a high potential to improve the performance-to-cost ratio of fiber-cements. However, little attention has been paid to the application of this concept in the commercial products.

Reinforcing fibers are the most prominent cost component in asbestos-free (AF) fiber-cement products, and so the rational distribution of fibers through products could make a significant difference in their performance-to-cost ratio. The static load capacities of corrugated sheets, produced with polyvinyl alcohol (PVA) or polypropylene (PP) fibers, are approximately 30 to 50% lower than those of conventional asbestos-cement (AC). This decrease in strength has led to an increase of issues associated with cracking and loss of water tightness (Akers, 2006; Hansen and Stang, 2009).

The mechanical properties of fiber-cement composites are mostly related to their fiber content, orientation, and geometric parameters (Bentur and Mindess, 1990). Therefore, by locally adjusting one of these parameters it is possible to vary in a controlled way the mechanical properties of a constructive component. Simply increasing the fibers volume fraction in highly stressed regions of a corrugated sheet and reducing its content in low-stress regions, the performance of AF products can be improved without a significant increase in their cost (Dias et al., 2008; Dias et al., 2010). In this paper, we explore the possibility of introducing mechanical properties gradation in corrugated sheets produced by Hatschek machines applying a fiber-rich cementitious mixture between the layers in specific points of the product.

EXPERIMENTAL PROCEDURES

In a preliminary industrial experiment, it was observed that the surface of a Hatschek formation cylinder rotates with velocity of approximately 1.5 m/s. To perform the application of the fiber-rich mixture on the surface of the formation cylinder, we estimated a shear rate of approximately 2400 s^{-1} for extrudate using an extrusion die 5 mm in diameter. This shear rate is exceptionally high when compared with conventional extrusions. So, the experimental work was divided into three stages: a) dosage of the dispersant by using a statistical mixture design, b) evaluation of the extrudability of the composites at high-shear-rate extrusion, and c) evaluation of the mechanical performance of the composites.

In the first stage, a carboxylate-based dispersant dosage study was carried out to determine the content capable of generating the lowest viscosity for the matrix that, in the fresh state, allowed the addition of high fiber contents, besides mechanical performance optimized in the hardened state. In the sequence, the matrix formulation was maintained, and cementitious composites for a high-shear-rate extrusion have been developed to be applied between the layers of Hatschek-made corrugated sheets. The flow characteristics and mechanical performance of these cementitious composites were investigated. Mixtures with PVA fiber content of 1, 2, and 3% or alkali resistant glass fiber (ARGF) content of 2, 3, and 4%, by volume, were subjected to high-shear-rate extrusion tests and used for the preparation of the specimens. After 28 days, the specimens were subjected to direct tensile tests. The highest strength mixtures were applied in a preindustrial test to validate the technique created to vary the properties of Hatschek-made corrugated fiber-cement sheets locally.

Materials

The following materials were used in this work: a) OPC with a skeletal density of 3120 kg/m³, b) 6-mm long Kuraray Kuralon PVA fibers with a skeletal density of 1356 kg/m³ and an equivalent diameter[1] of (14.38 ± 2.09) μm, c) Vetrotex anticrack HD ARGF, (13.9 ± 0.49) mm long with a diameter of (14.6 ± 0.90) μm, d) carboxylate-based dispersant (Melflux 2651 F), and e) methyl hydroxyethyl cellulose (MHEC) water retention agent (Tylose MN 60001 P6). A 15.5% of zirconia (ZrO_2) by mass, which is characteristic of ARGFs, was detected by X-ray fluorescence in the glass fibers utilized in this work.

Dosage of the dispersant by using a statistical mixture design

A statistical mixture design was applied for the dispersant dosage. In this mixture design, the volumetric content of the OPC varied from 47.25% to 52.85%. The water content varied from 47% to 52%, while the content of dispersant ranged from 0.15% to 0.75% by volume. Figure 1 shows the formulations. In this stage, the mixtures were evaluated in a rotational rheometer, in a ram extruder and used to prepare specimens for Brazilian split-cylinder tests. The rheological tests were performed on a TA Instruments AR 2000 rheometer, applying concentric cylinders with diameters of 40 mm (internal) and 41 mm (external). In these tests, the shear rate ranged from 0 to 1000 s^{-1} for 5 s and was maintained at 1000 s^{-1} for 30 s when the viscosity was measured. The mixtures were subjected to high-shear-rate extrusion tests (Figure 2a) performed in a ram extruder with a 52.4-

[1]The PVA fibers employed had no perfect circular cross-section. The equivalent diameter corresponds to that for a circular area numerically equal to the real cross-section area.

mm diameter barrel. A 15-mm length die was used with an internal diameter of 2 mm. The ram velocity was held equal to 2 mm/s.

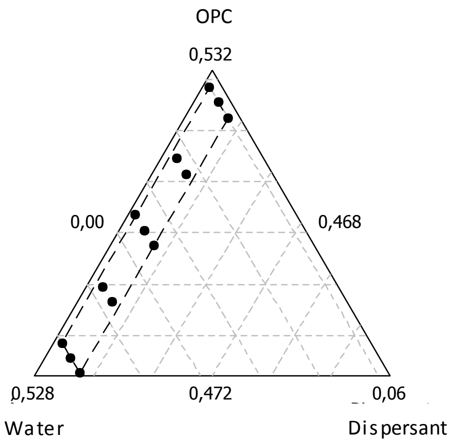


Figure 1. Experimental formulations for the dispersant dosage study

Production and evaluation of the composites

After choosing the optimal proportion between the ingredients for the matrix, the composites were produced as follows: a) mixing the OPC and dispersant for 1 min in a 5-L planetary Hobart mixer, b) addition of water gradually over a 2-min interval while the mixer was kept on, c) addition of fibers and mixing for 5 min in low rotation, and d) addition of MHEC and mixing for 1 min at high rotation. MHEC was used in a volume content equal to 0.70% to avoid segregation during extrusion. Immediately after mixing, the mixture was placed in the barrel of the ram extruder (Figure 2a), where it rested for 1 min and was tested in extrusion.

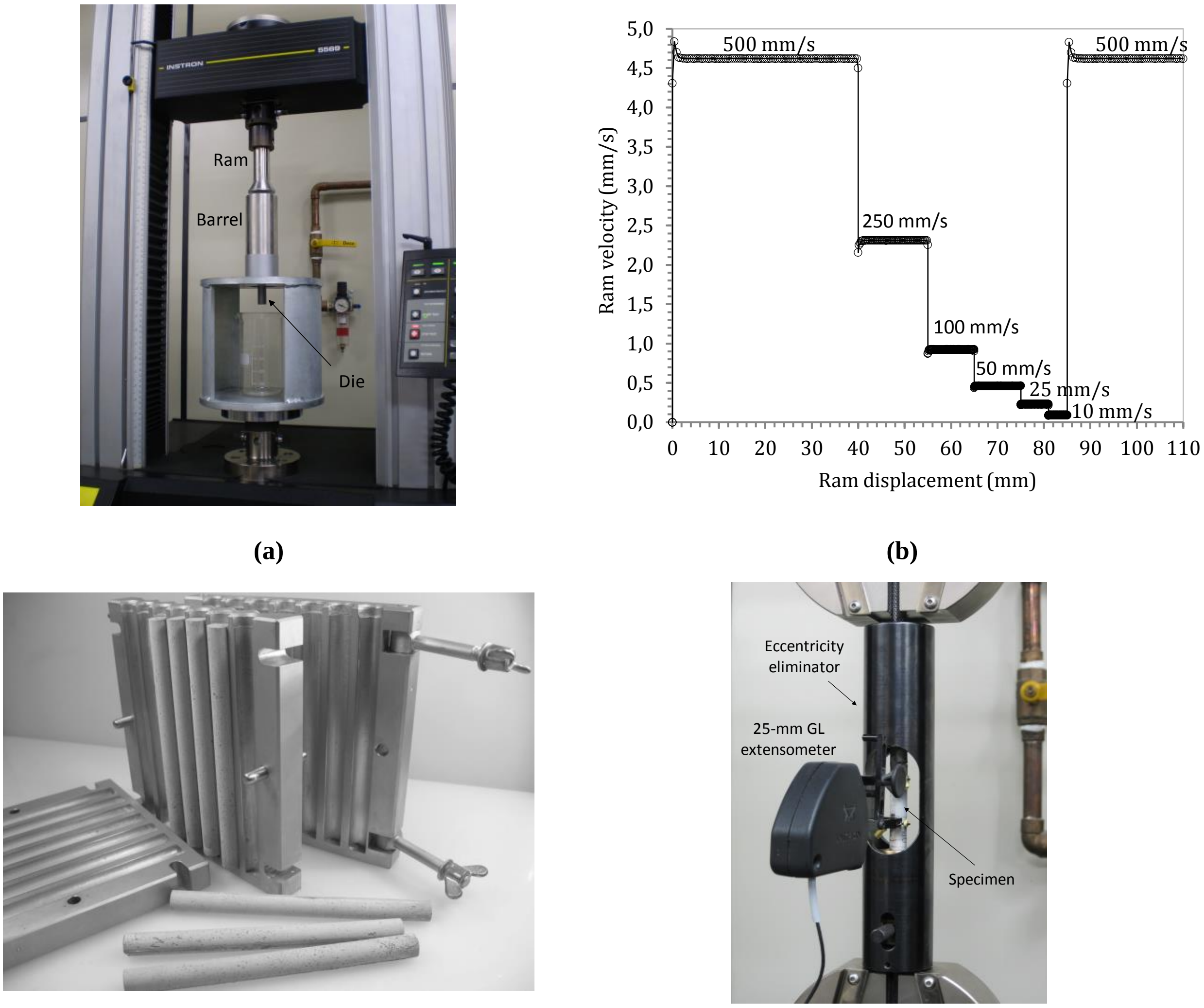
For PVA composites series, the PVA fiber contents varied from 1 to 3% by volume, whereas the content of ARGF varied from 2 to 4%, by volume, for ARGF composites series. Mixtures of PVA and ARGF series were subjected to high-shear-rate extrusion tests (Figure 2a) performed with three different die dimensions with lengths of 15, 35 and 50 mm, and with an internal diameter of 5 mm. The extrudate velocity ranged from 10 mm/s to 500 mm/s as shown in Figure 2b. These mixtures were injected into a metallic mold for the preparation of 100-mm-length specimens with a diameter of 10 mm (Figure 2c). After 28 days, the specimens were subjected to direct tensile tests (Figure 2d). The loading rate was controlled at 0.20 mm/min, and the longitudinal strains were measured with a 25-mm GL axial clip extensometer.

The procedure developed by Benbow and Bridgwater (1993) has been the most used to evaluate the rheological properties of mixtures for extrusion. This method consists of performing extrusion tests on a ram extruder (Figure 3a) using dies with different lengths and determining the extrusion pressures for a set of extrudate velocities. The primary objective of this procedure is to evaluate the influence of the formulation on parameters related to the rheological properties of the mixture. Eq. 1 relates the extrusion pressure *P* with these parameters (Benbow and Bridgwater, 1993). In cases where there is no linearity between pressure and extrudate velocity, Benbow and Bridgwater (1993) recommend the use of Eq. 2 with six parameters.

$$P = 2(\sigma_0 + \alpha V) \ln\left(\frac{D_0}{D}\right) + 4(\tau_0 + \beta V)(\frac{L}{D}) \quad \text{Eq. 1}$$

$$P = 2(\sigma_0 + \alpha V^m) \ln\left(\frac{D_0}{D}\right) + 4(\tau_0 + \beta V^n)(\frac{L}{D}) \quad \text{Eq. 2}$$

Where, *V* is the extrudate velocity in the die land, σ_0 is the yield stress extrapolated to zero velocity, α is a factor characterizing the effect of velocity, D_0 is the diameter of the barrel, *D* is the diameter of the die, τ_0 is the wall shear stress extrapolated to zero velocity, β is the wall velocity factor, *m* and *n* are constants.



(a) (b)

(c) (d)

Figure 2. a) Extrusion test, b) ram velocity with correspondent extrudate velocities, c) mold for specimen's preparation for tensile tests, and d) direct tensile tests with an extensometer and eccentricity eliminator

RESULTS AND DISCUSSION

Mixture adjustments

In the dispersant dosage study, the contents of OPC, water, and dispersant were varied (see Fig. 1). Figure 3 depicts the response surfaces for the viscosity, tensile strength, and extrusion pressure. The results show that as the dispersant and water contents increase, the viscosity of the mixture reduces, so the point of minimum viscosity is the one with the highest water and dispersant contents (Fig. 3a). The response surface for pressure in high-shear-rate extrusion behaves like that for the viscosity.

The highest tensile strength obtained in Brazilian split-cylinder test was obtained with the lowest water content and the highest dispersant content (Figure 3b). For the matrix of extrudable composites, the mixture has low viscosity in the fresh state to allow the inclusion of high fiber contents. The choice of the matrix formulation was made using multiple optimizations (Derringer and Suich, 1980), using the parameters presented in Table 1.

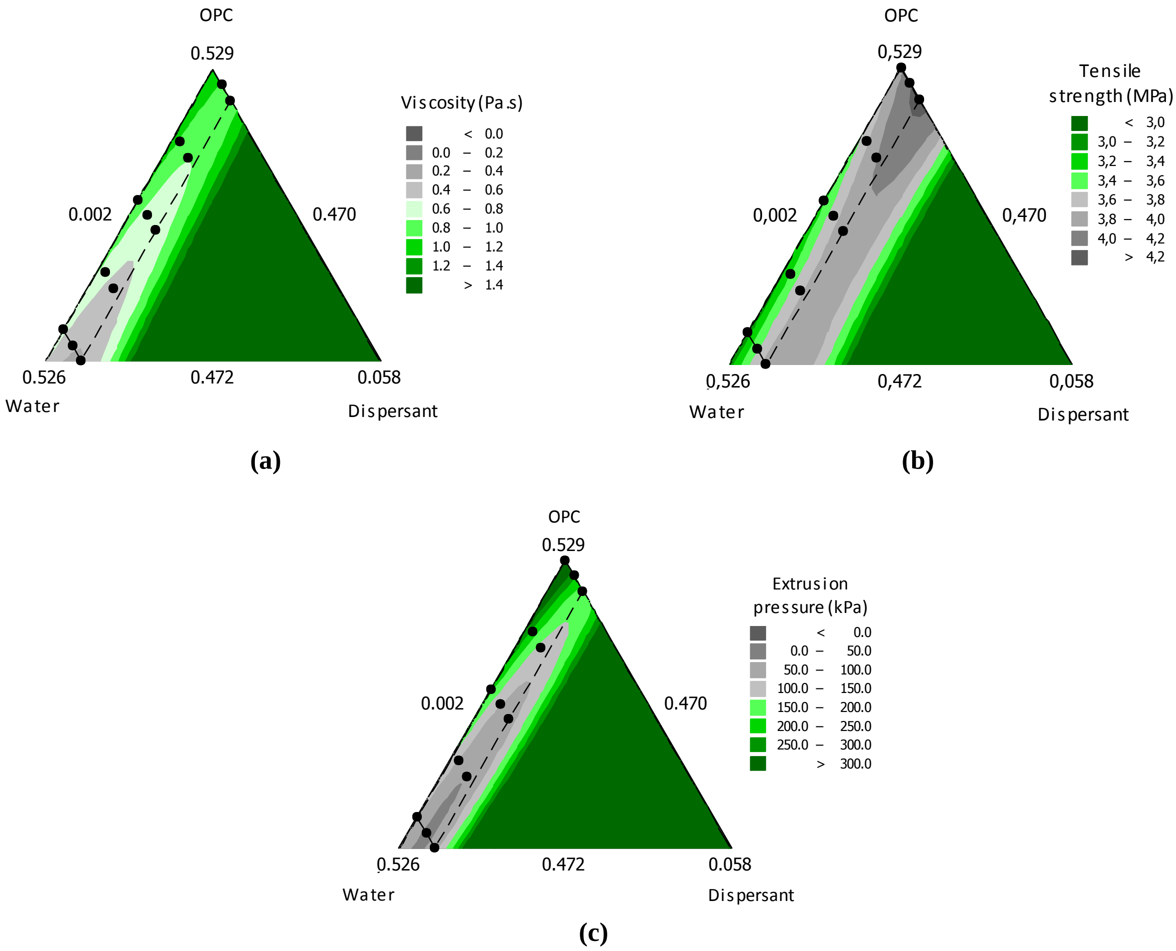


Figure 3. Dispersant dosage study: a) viscosity at shear rate of 1000 s^{-1}, b) tensile strength in Brazilian test, and c) extrusion pressure in a 15-mm-length die with a diameter of 2 mm with a ram velocity of 2 mm/s

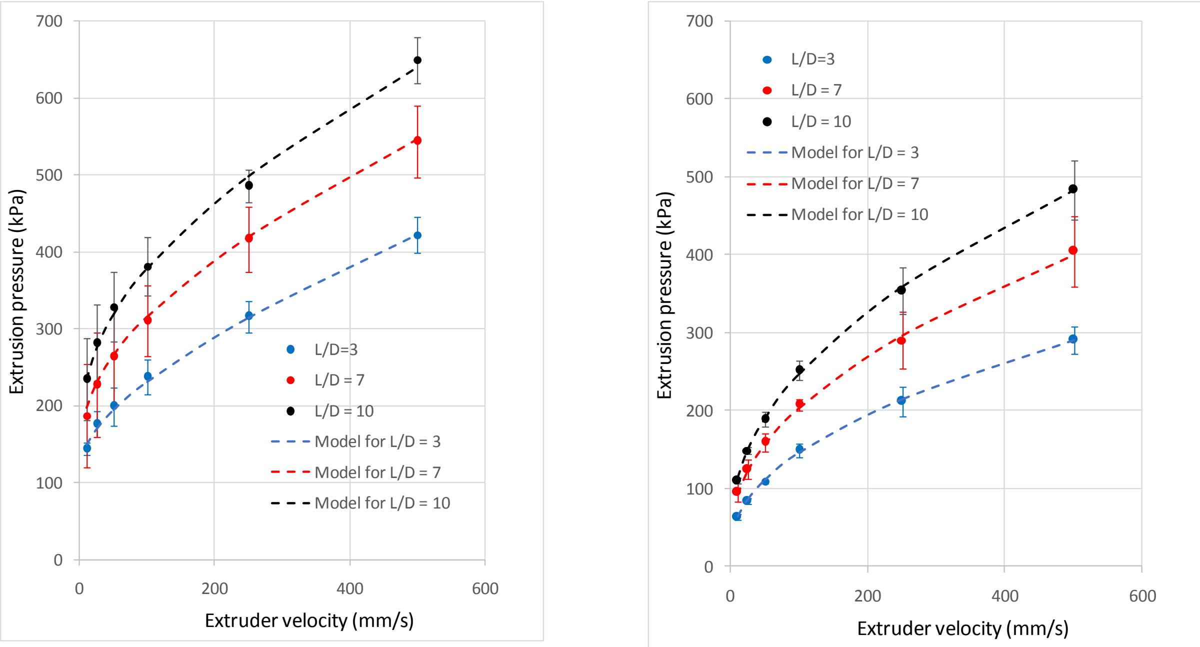
Table 1. Optimization of the matrix performance

Parameters	Minimum	Target	Maximum	Predicted	Desirability
Extrusion pressure (kPa)	50	50	200	57.1	0.952
Tensile strength (MPa)	3	4	4	3.8	0.796
Viscosity	0.5	0.5	1	0.5	1.000

The procedure developed by Derringer and Suich (1980) for multiresponse optimization consists in maximize the geometric mean of the individual desirabilities for the experimental responses. Desirability ranges from zero to one and corresponds a weight given for the property according to its value. The setup in Table 1 minimizes the viscosity and the pressure in the extrusion, while the tensile strength is maximized. The formulation that best fitted the defined parameters and that had an overall desirability of 0.91 was composed of 48.50% cement, by volume, 50.81% water, and 0.69% dispersant. Table 1 provides the predictable properties for this formulation.

Optimization of the extrusion parameters

In the sequence, mixtures with PVA fiber content of 1, 2, and 3% or ARFG content of 2, 3, and 4% by volume were subjected to high-shear-rate extrusion tests. Figure 4 shows the extrusion pressure versus extrudate velocity curves for composites with a fiber content of 3%. As expected, the extrusion pressures are higher for longer dies. Also, as the extrudate velocity increased, the pressure became higher. This behavior is similar for different types of fibers: PVA (Figure 4a) and AR glass (Figure 4b). By comparing composites with the same fiber content, at the same rate of application, those with PVA fibers had higher extrusion pressures, although the glass fibers applied in the present experiment were more than twice the length of the PVA fibers. A plausible hypothesis is that the stiffness of the fibers affects their alignment during the flow, directly interfering with the extrusion pressure. Moreover, because of the geometric characteristics, for the same volumetric content of fibers, the number of glass fibers is smaller than that of PVA fibers.



(a) (b)

Figure 4. Pressure versus extrudate velocity: a) for the PVA3% series and b) for the ARGF3% series

The parameters of the Benbow and Bridgwater model (Eq. 2) for the mixtures are shown in Table 2. The six-parameter equation (Eq. 2) was used because the linearity deviation between pressure and velocity. The curves obtained through the models for the mixtures PVA 3% and ARGF 3% are presented in Figure 7. The models fitted by the least-squares method were statistically significant and explained the experimental data thoroughly. However, an apparent tendency of variation of the parameters of the models with the variation of the fiber contents was not observed. Thus, the equations were used only for the sizing of the applicator in the preindustrial test. When, for example, the PVA 3% mixture was applied at a speed of 1.5 m/s, with an extruder with a 76-mm-diameter barrel and a 5-mm-diameter and 50-mm-length die, it required pressure of approximately 1.1 MPa.

Table 2. Benbow and Bridgwater (1993) extrusion parameters

Volumetric content of fibers (%)	Type of fibers	σ_0(kPa)	α (kPa.(s.m^{-1})^m)	m	τ_0(kPa)	β (kPa.(s.m^{-1})^n)	n	R^2
1	PVA	0.00	33.82	0.548	0.78	6.46	0.434	1.000
2		12.01	42.67	0.575	0.00	8.48	0.453	0.996
3		21.45	82.63	0.761	0.00	9.16	0.240	0.997
2	ARGF	3.79	61.31	0.562	0.37	7.29	0.410	0.998
3		3.75	57.96	0.509	0.76	8.42	0.466	0.999
4		3.48	54.20	0.546	0.76	8.98	0.496	0.992

Mechanical characterization

Figure 5 shows how the tensile strength of the composites with PVA or AR glass fibers varies with the fiber content. In both series, the tensile strength of the composites increased with increasing fiber content. In this study, composites with 3% of PVA fibers showed an average tensile strength of 7.4 MPa, an excellent value for cementitious composites with this type of fiber. Composites with 4% glass fibers had a tensile strength of 12 MPa. The composites with different types of fiber showed entirely different mechanical behavior. The composites with PVA fibers had a pseudoplastic behavior with high deformation capacity, improved with the increase of fiber content. The composites with AR glass fibers showed brittle behavior.

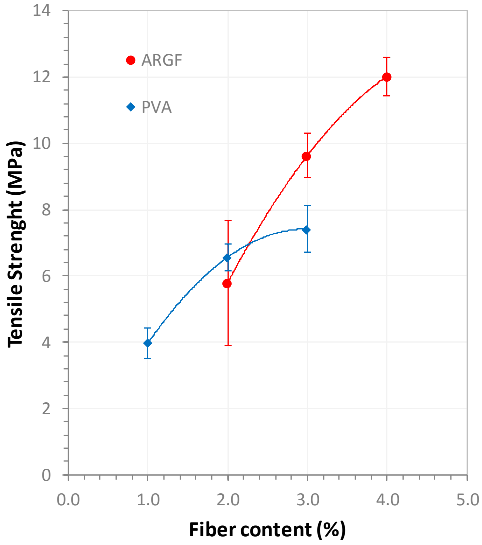
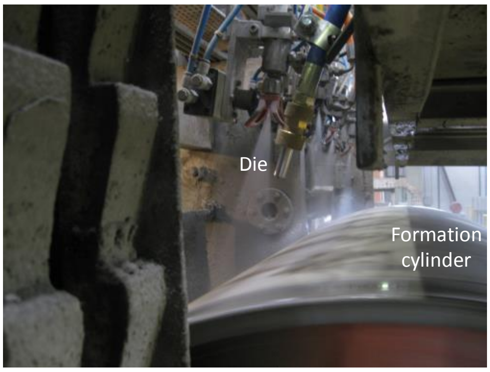


Figure 5. Direct tensile strength of specimens containing PVA fibers and specimens with ARGF

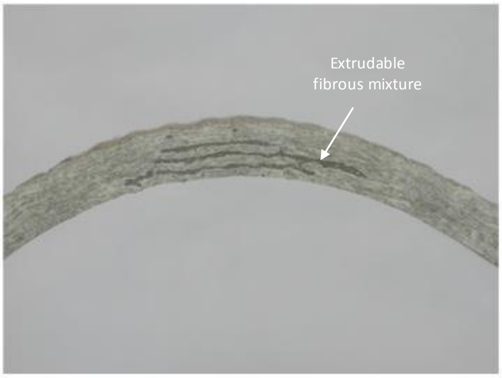
Preindustrial application

A preliminary preindustrial experiment was carried out on a Hatschek machine, which was producing approximately 10 t/h of green fiber-cement sheets. All the parameters of production process were preserved, including the formulation in the vats. The fibrous mixture applicator consisted of an engine, a removable cell into which the fresh fibrous mixture is placed, a ram that is pushed by the screw, and a control panel. This system enabled the ram displacement and flow rate of the mixture to be controlled carefully. A hose with a die having a 5-mm-diameter circular hole was attached to the end of the extruder and positioned above the formation cylinder of the Hatschek machine, as shown in Figure 6a. During the manufacturing of the sheets, synchronization between the applicator and the formulation cylinder was needed. A lack of synchronization can result in damage to the surface of the finished product, as well as discontinuities in the fibrous mixture layers, variation in the number of layers, and shapeless accumulations. Once controlled, the extrusion of the fibrous mixtures between the layers does not disturb the corrugation of the sheet (Figure 6b).

The PVA3% and ARGF4% mixtures were successfully applied in a preliminary preindustrial study. The application of the mixture containing 4.0% ARGF provided the local increase of the limit of proportionality (LOP) of the composite by about 10%, i.e., from (9.65 ± 0.76) MPa to (10.6 ± 0.54) MPa, without significant changes in the modulus of rupture (MOR). The application of the mixture with 3% PVA fibers provided the local increase of the MOR by 10%, i.e., from (19.4 ± 0.77) MPa to (21.2 ± 0.89) MPa, without significant changes in the LOP. The technique of localized application of extrudable fibrous mixtures has proved to be efficient to modify the properties of fiber-cement corrugated sheets locally. Experiments should be carried out to reduce the fiber content in the vats and to evaluate the performance of the entire corrugated sheets.



(a)



(b)

Figure 6.a) Position of the die in the Hatschek machine and b) ideal cross-section of a 6-mm thicker corrugated sheet after application of PVA3% mixture

CONCLUSIONS

The content and the type of fiber play a crucial role in the extrusion pressure. By comparing composites having the same fiber content, at the same extrusion shear rate, those with PVA fibers had higher extrusion pressures, although the glass fibers were more than twice the length of the PVA fibers. A plausible hypothesis is that the stiffness of the fibers affects their alignment during the flow, directly interfering with the extrusion pressure. Moreover, because of the geometric characteristics, for the same volume of fibers, the number of glass fibers was about 60% smaller than that of PVA fibers. Composites with 4.0% glass fibers, by volume, had average tensile strengths of 12.0 MPa, while those with 3.0% PVA fibers showed 7.5 MPa. The ARGF 4% and PVA 3% mixtures were successfully applied in a preliminary preindustrial study. Once controlled, the extrusion of the fibrous mixtures between the layers does not disturb the corrugation of the sheet. The proposed technique of localized application of rich-fibrous mixtures presents a high potential for producing functionally graded corrugated fiber-cement sheets. New experiments should be carried out to evaluate the possibility to reduce the fiber content in the vats and improve the mechanical performance of an entire corrugated sheet.

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