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.

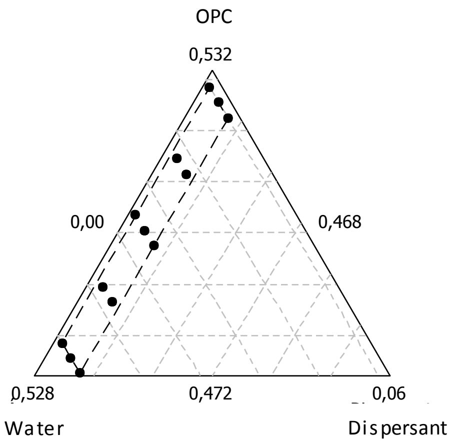


Figure 1. Experimental formulations for the dispersant dosage study

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.

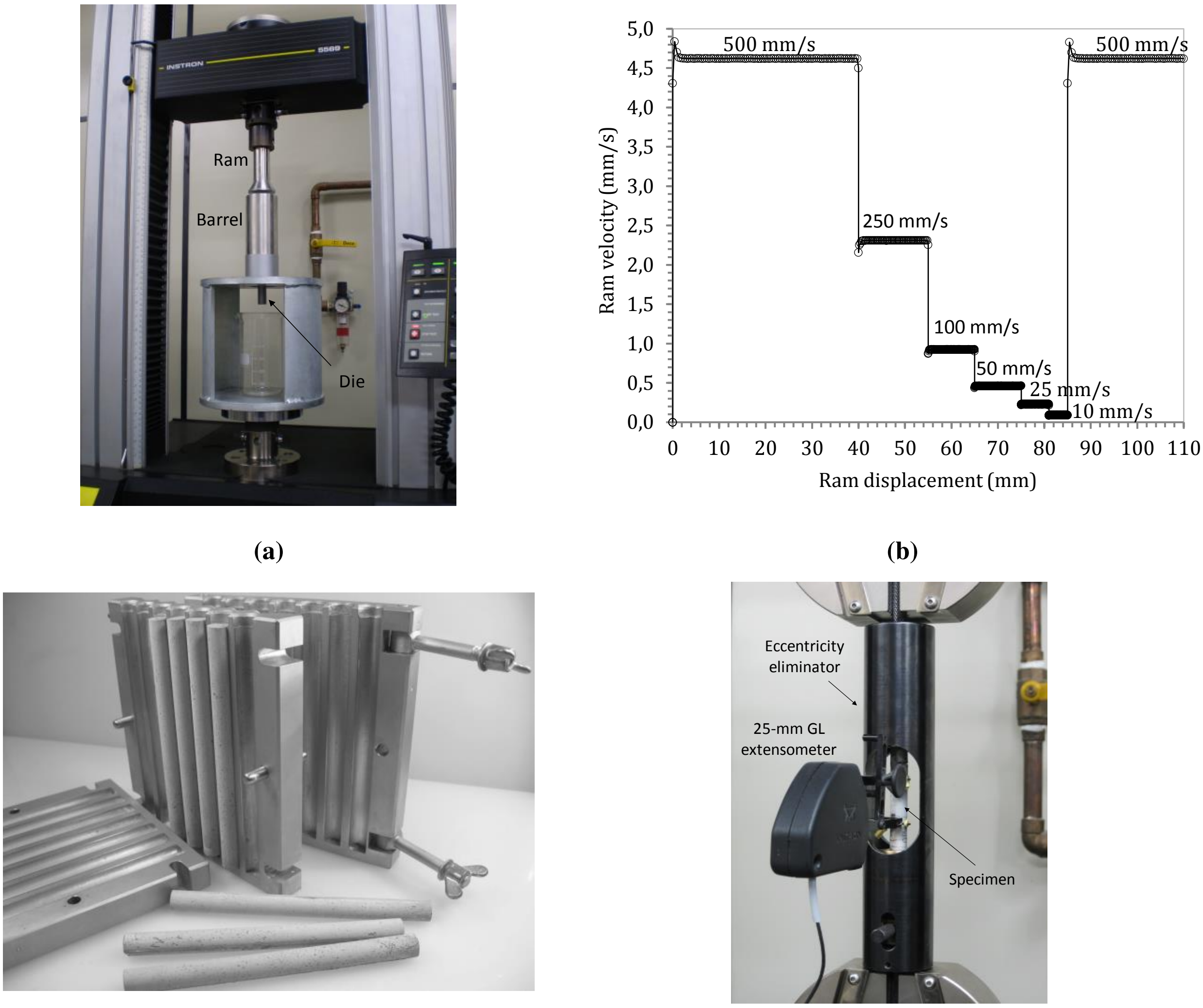
For PVA composites series, the PVA fiber contents varied from 1 to 3% by volume, whereas the content of ARGF varied from 2 to 4%, by volume, for ARGF composites series. Mixtures of PVA and ARGF series were subjected to high-shear-rate extrusion tests (Figure 2a) performed with three different die dimensions 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.

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

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

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.

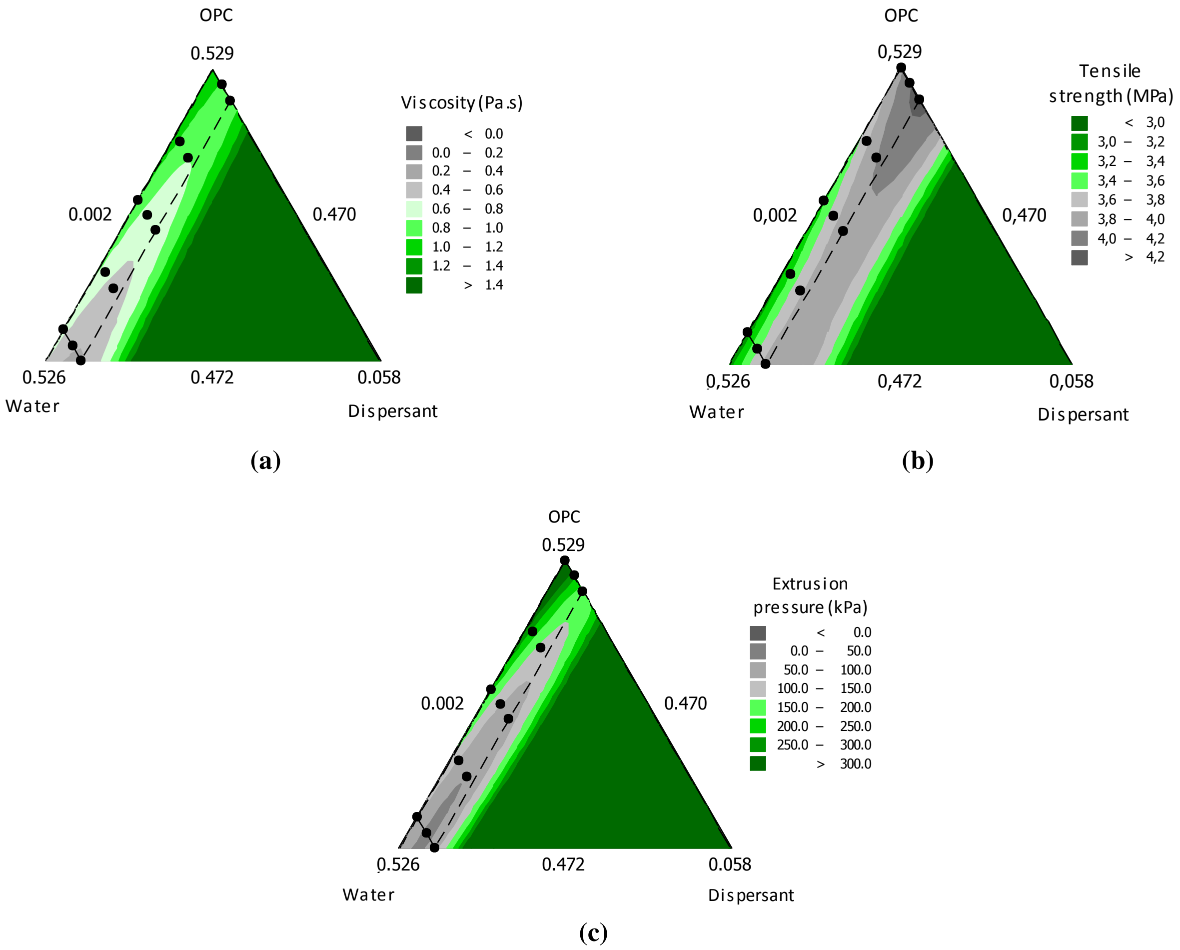


Figure 3. Dispersant dosage study: a) viscosity at shear rate of 1000 s^{-1}, b) tensile strength in Brazilian test, and c) extrusion pressure in a 15-mm-length die with a diameter of 2 mm with a ram velocity of 2 mm/s

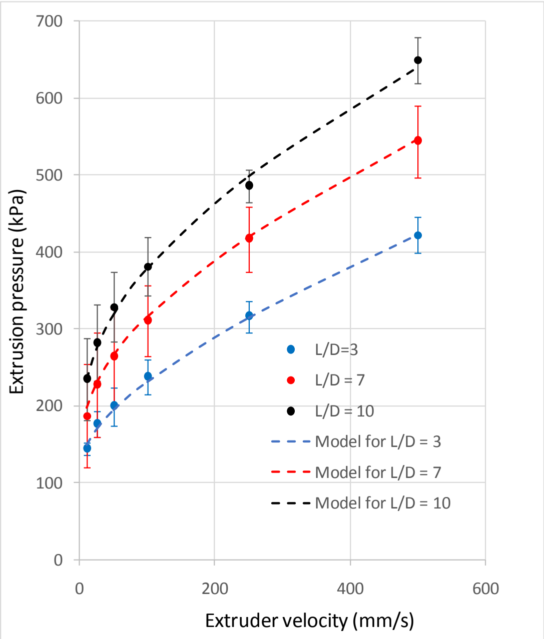
Table 1. Optimization of the matrix performance

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

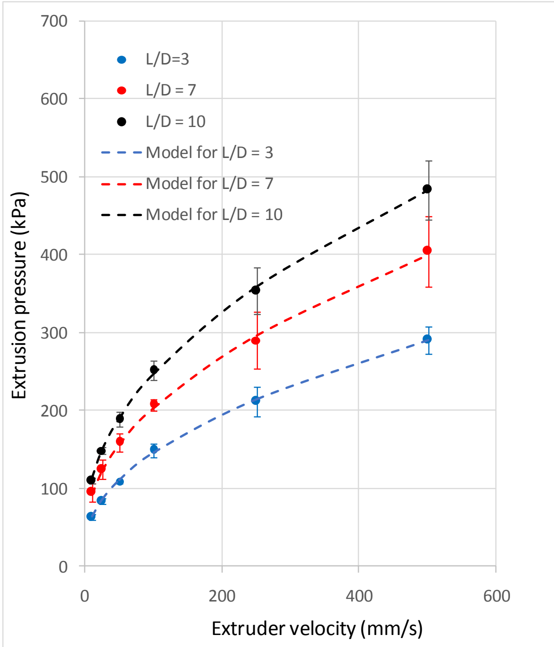
The procedure developed by Derringer and Suich (1980) for multiresponse optimization consists in maximize the geometric mean of the individual desirabilities for the experimental responses. Desirability ranges from zero to one and corresponds a weight given for the property according to its value. The 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.

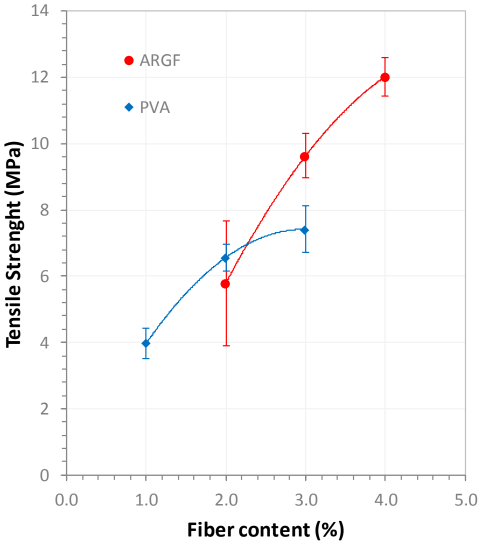
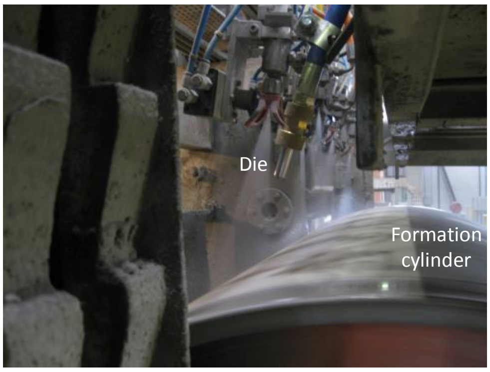


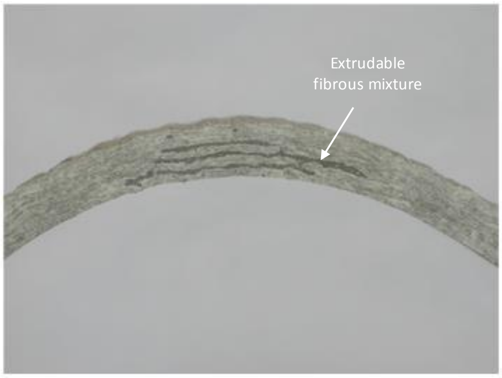
Figure 5. Direct tensile strength of specimens containing PVA fibers and specimens with ARGF

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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REFERENCES

Akers, S. A. S. “Cracking in fiber cement products”. 10th Int. Inorganic-Bonded Fiber Composites Conference. São Paulo, Brazil. 2006.

Amada, S., Ichikawa, Y., Munekata, T., Nagase, Y., Shimizu, H. “Fiber texture and mechanical structure of bamboo”. In: “Composites part B 28B. p13-20. 1997

Benbow, J., Bridgwater, J. “Paste Flow and Extrusion”. Clarendon Press, Oxford (1993).

Bentur, A., Mindess, S. “Fibre reinforced cementitious composites”. London and New York. Elsevier Applied Science, 1990.

Bever, M. B., Duwez, P. E. “Gradients in composite materials”. Materials Science and Engineering 1972; 10(1): 1-4.

Bruck, A., Evans, J.J., Peterson, M.L. “The role of mechanics in biological and biologically inspired materials”. Experimental mechanics 2002; 42(4): 361-371.

Cornell, J. A. “Experiments with mixtures”. John Wiley & Sons. New York. 1981.

Derringer, G.; Suich, R. “Simultaneous optimization of several response variables”. Journal of Quality Technology 12 (4). (1980).

Dias, C. M. R., Savastano Jr., H., John, V. M. “Exploring the potential of functionally graded materials concept for the development of fiber-cement”. Construction and Building Materials 24(2) (2010) 140-146.

Dias, C. M. R., Savastano Jr., H., John, V. M. “The FGM concept in the development of fiber-cement components”. American Institute of Physics Conference Proceedings, 973(1) (2008) 525-531.

Hansen, K. F.; Stang, B. D. “Safety against formation of through cracks of profiled fibre-reinforced cement sheets for roofing”. Construction and Building Materials V 23, I1, (2009) p. 334-339.

Miyamoto, Y., Kaysser, W.A., Rabin, B.H., Kawasaki, A., Ford, R.G. “Functionally graded materials: design, processing and applications”. Ed. Kluwer Academic Publisher. 1999.

Neubrand, A., Rödel, J. “Gradient Materials: An overview of a novel concept”. Z Metallkd V88, N5, p. 358-371. 1997.

Scheffé, H. “Experiments with mixtures”. Journal of the Royal Statistical Society B 20 (1958). 344-360.

Shen, B., Hubler, M., Paulino, G. H., Struble, L. J. “Functionally-graded fiber-reinforced cement composite: Processing, microstructure, and properties”.Cement and Concrete Composites 30(8) (2008) 663-673.

Stroeven, P., Hu, J. “Gradient structures in cementitious materials”. Cement and Concrete Composites 29(4) (2007) 313–323.

Toader, N., Sobek, W., Nickel, K. G. “Energy Absorption in Functionally Graded Concrete Bioinspired by Sea Urchin Spines”. Journal of Bionic Engineering 14(2) (2017) 369–378.