

## **THE INFLUENCE OF SILICA FUME ON THE DRYING SHRINKAGE OF FIBER CEMENT REINFORCED WITH PVA FIBERS**

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### **ABSTRACT**

The main purpose of this paper is to study the influence of silica fume on the drying shrinkage and flexural resistance of fiber cements produced by the Hatscheck process. Samples of fiber cement reinforced with PVA and cellulose fibers were tested with two different compositions, one sample with silica fume and the other without silica fume. Two test series were performed: shrinkage and flexural resistance were measured. In spite of the change in formulation happening just in the cementitious matrix, there were no changes of LOP and E of the composite. However, the results of MOR of the composite with silica fume were larger than for the other composite. The fiber cement with silica fume presented greater shrinkage than the other, for the same mass loss. That occurred due to the refinement of pores brought about by the addition of silica fume, measured for MIP.

### **KEYWORDS:**

Fiber cement; drying shrinkage; silica fume; PVA; MIP.

### **INTRODUCTION**

In Brazil, PVA and PP fibers have been widely used to reinforce fiber cement. Moreover, silica fume has been used as pozzolanic addition to improve the matrix properties (Devasto, 2006), leading to an improvement in the performance of fiber cements without asbestos.

However, asbestos free fiber cement components frequently present edge crackings which appears during storage. The drying differential, a major cause of shrinkage, can give birth to tensile stress that can cause cracking (Akers *et al.*, 1983).

Several factors influence the risk of cracking, including surface drying velocity, which depends on the micro-environment, tensile strength, Young's modulus, the magnitude of drying shrinkage of fiber cement and stress relaxation. These factors are related to hygroscopic movement (drying) or fiber cement properties; however the phenomenon that governs the emergence of cracking is shrinkage. Drying shrinkage is influenced by the presence of capillary pores (capillary pores are those with diameters between 5 and 50 nm, Mehta, 2008), as these are associated with the shrinkage mechanism called capillary depression (Wittmann, 1982). The magnitude of the generated stress depends on three factors: pore fluid surface tension, contact angle (Mora-Ruacho *et al.*, 2009) and the distribution of pore diameters (Bentur, 1979). The first two factors are related to the fluid (fluid surface tension) and the affinity between the fluid and the capillary walls (contact angle); and they can be changed by the use of admixtures (Mora-Ruacho *et al.*, 2009). The third factor is related to the composite properties, thus the pore size distribution of cement based materials depends on several factors, including cement type or mineral admixtures, curing and compacting conditions,

amount of mixing water, and content of filler, among other factors. The capillary pressure is significant only in pores with diameters below 35 nm, according to Young (1988.).

The cementitious matrix has macropores, capillary pores (cause of drying shrinkage) and pores smaller than the capillaries (size less than 5 nm), which also generates shrinkage. These pores are present between the layers of C-S-H, and may have up to 15Å (Mehta, 2008). Between C-S-H lamellae (hydrophilic solid surfaces), separated by a thin film of water, the resulting force is repulsive and is called disjoining pressure (Feldman; Sereda, 1970). With the release of water trapped between the lamellae, the disjoining pressure is canceled, resulting in shrinkage. In this way, the use of pozzolanic materials, which increases C-S-H content, produces a potential to increase shrinkage. Nevertheless, there is little discussion about it in the bibliography.

The main point of this paper is to study the influence of silica fume on the drying shrinkage and flexural resistance of fiber cement, produced by the Hatscheck process.

## EXPERIMENTAL

The effects on shrinkage, porosity and the mechanical properties of PVA reinforced fiber cement produced in Hatscheck machine, and having the limestone filler replaced with silica fume, were evaluated.

Two formulations of fiber cement were produced as shown in Table 1. The first formulation (sample FC) is composed of cement, limestone filler, fiber and water; the second formulation (sample FC+SF) is composed of cement, limestone filler (minus 3%), water, fiber, and the addition of 3% silica fume.

**Table 1 – Composition of samples of fiber cement (by volume)**

	Composition (%)	
	FC+SF (fiber cement with silica fume)	FC (fiber cement without silica fume)
Water	54.3	54.7
Cement	26.6	26.8
Limestone filler	12.3	14.7
Silica fume	3.0	0.0
Fibers	3.7	3.8
	100	100

## Materials

The Portland cement used was type CII-F-32<sup>1</sup>, with a unimodal distribution of particles and a higher frequency of particles with a diameter of 40 µm. Magnesium type limestone from southern Brazil, not significantly different in comparison with cement, except for a larger amount of coarse particles, was also used. The silica fume was non-densified, and had Chapelle reactivity of 727 mg of CaO/g. Figure 1 shows that this material was finer than the cement and limestone-filler, but much coarser than expected, suggesting the presence of large particle aggregates due to insufficient dispersion in the plant (Romano *et al.*, 2008). The organic fibers used were hardwood pulp and synthetic fiber of polyvinyl alcohol (PVA).

<sup>1</sup> The CII-F-32 cement is composed of 90-94% clinker + gypsum, and 5-10% of limestone filler, according to the classification of the Brazilian Association of Technical Standards – ABNT.

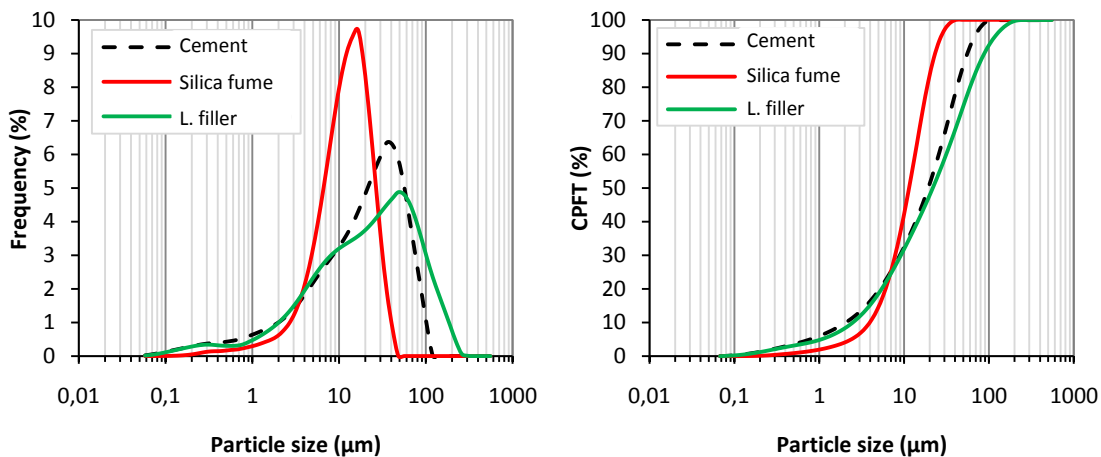


Figure 1 – Discrete and cumulative particle size distribution (Mastersizer S model) of the cement, limestone filler and silica fume.

## Samples

The samples of fiber cement were produced by the Hatschek process in a Brazilian fiber cement factory. Figure 2 shows the cutting of still fresh fiber cement to form the flat plates used in the study. The samples were subjected to an initial curing of 8 hours in an environmental chamber with high relative humidity and temperature of 60°C.



Figure 2 – Sample of fiber cement produced by the Hatschek process.

## MOR and Young's modulus

The tests were performed on 28 specimens of 160x40x6 mm (14 in each formulation). This sample was subjected to an additional cure of 17 days in an environmental chamber with a temperature of 23°C and a relative humidity of 90%. The samples were then immersed for two days in water saturated with lime. The specimens were tested after completing 20 days of hydration.

The flexural test was conducted with a 4-point method using an Instron universal machine (model 5569) with a load cell of 1 kN and displacement sensor Solatron positioned exactly in the center of the span to measure deflection. The download speed was constant and equal to 5.0 mm/min. The mechanical indices calculated from this test were: maximum flexure load (MOR), Young's modulus (E) and limit of proportionality (LOP).

**Drying shrinkage**

Eighteen specimens of 160x40x6 mm (9 in each formulation) were used to measure drying shrinkage. After an initial 24h of curing the specimens were prepared for testing by: protecting the sides with polyurethane varnish and fixing the concave metal pins to the tops with epoxy adhesive and curing for an additional 5 days in an environmental chamber at a temperature of 23°C and a relative humidity of 90%.

The drying shrinkage test started after 6 days of curing in an environmental chamber - temperature 23°C and relative humidity 50%. Both length and mass of the samples were periodically recorded. The mass was determined using a digital balance (precision  $\pm 0.001$  g) and shrinkage which included a support, comparison bar and extensometer.

**Pore size distribution**

A set of 4 specimens of 1.0 g (2 in each formulation), were produced using a small diamond saw to measure distribution of pore size by mercury intrusion test (Micromeritics equipment, model Auto pore III). This sample was subjected to an additional cure of 17 days in an environmental chamber with temperature at 23°C and the relative humidity, 90%. After curing, the samples were dried in a vacuum drying oven for 48 hours at a temperature of 60°C.

**Statistical test of hypothesis - Student's t-test**

The test results obtained were analyzed by applying a statistical test of hypothesis. All results presented are from the averages of several repetitions, so we applied the Student's 't' test as a test of significance when comparing two means. This test is suitable for making comparisons between treatments where the response shows if there are any significant differences between means.

The parameters adopted in the calculations were hypothesis of zero difference and a probability of less than 5% in the distribution curve 't'. This tool considers in its analysis the number of repetitions and the variability of the testing technique.

**RESULTS AND DISCUSSIONS****Mechanical strength**

The only difference between the fiber cement formulations studied was the addition of silica fume to one of the samples, in other words, the difference occurs only in the cementitious matrix. However, the test results of flexural strength showed no significant difference between the samples, including indices that characterize the matrix (LOP and E). From the results shown in Figure 3, we observe that some curves (flexure stress versus deflection) are superimposed for both samples. Considering all the curves in Figure 3, we observe that the curves in red (FC+SF, fiber cement with silica) are concentrated slightly above the curves in blue (FC, fiber cement without silica), indicating greater resistance to bending.

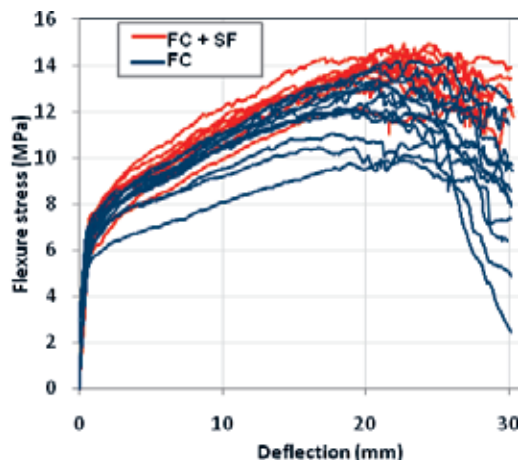


Figure 3 – Flexural strength results. One outlying curve with exceptionally low strength was removed.

The curves in Figure 3 show some indices of mechanical strength such as MOR (indicates the maximum flexure load), E (Young's modulus) and LOP (indicates the elastic limit), and the last two reflect changes in the cementitious matrix. The results show that the values of MOR vary widely in the samples without silica. Despite this, the MOR is statistically different between the samples, and the fiber cement with silica is tougher than the other. The same was not true in the other indices (E and LOP), which are statistically equal. Although some changes were expected in these indices, especially because silica fume modifies the cementitious matrix, that did not happen. A possible explanation for that may be the young age of the samples when submitted to rupture, seeing that chemical reactions resulting from the hydration of silica fume only occur in the long term.

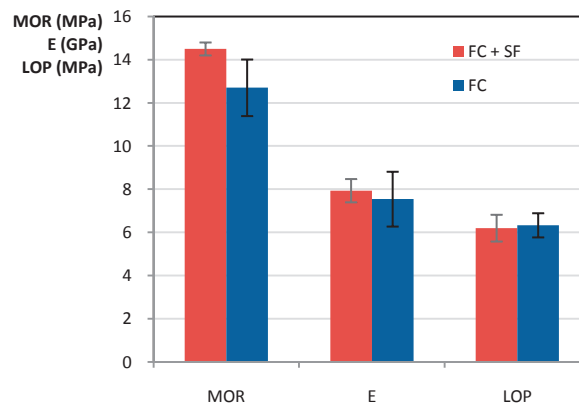


Figure 4 – Values of flexure stress at maximum flexure load (MOR), Young's modulus (E) and limit of proportionality (LOP)

### Influence of silica fume on the drying shrinkage

The silica fume particles have smaller average diameters (higher frequency of particles with diameter 17  $\mu\text{m}$ ) than those of cement and limestone filler (higher frequencies of particles with diameters 40  $\mu\text{m}$  and 50  $\mu\text{m}$ , respectively). With this smaller average diameter, the silica fume may promote a denser packing of particles, and consequently, reduce porosity and pore size (pore refinement).

According to this presented hypothesis, the results of drying shrinkage were consistent. Sample FC+SF (cement with silica fume) showed 23% greater (2.06 mm/m) drying shrinkage than sample FC (cement without silica) (2.53 mm/m) (Figure 5). The cause of the greater shrinkage is the reduction in pore size, which according to the data presented in the literature review, increases capillary tension.

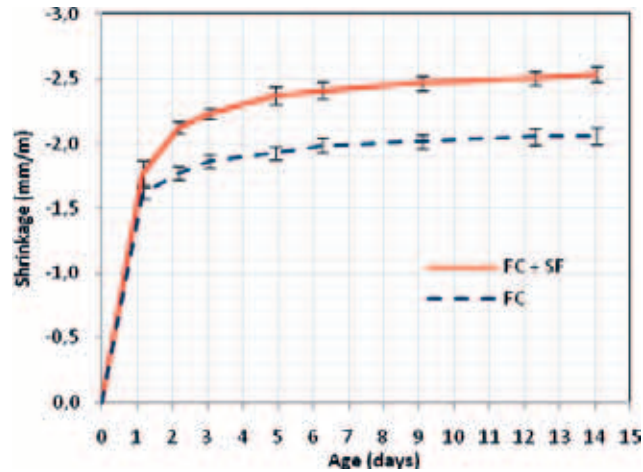


Figure 5 – Effect of silica fume on drying shrinkage

The hygroscopic movement is a major cause of shrinkage, so it is important to measure the sample's water loss during drying. The mass loss (indicates the amount of water evaporated during drying) of the two samples was statistically the same, as shown in Figure 6.

The relationship between shrinkage and hygroscopic movement is linear for both samples (Figure 7), showing that the shrinkage is caused by the loss of water, however, the biggest shrinkage in sample FC+SF produces lines that do not overlap on the graph. Figure 7 illustrates that for any given mass loss the shrinkage in the sample with silica is higher, making this fiber cement more susceptible to cracking.

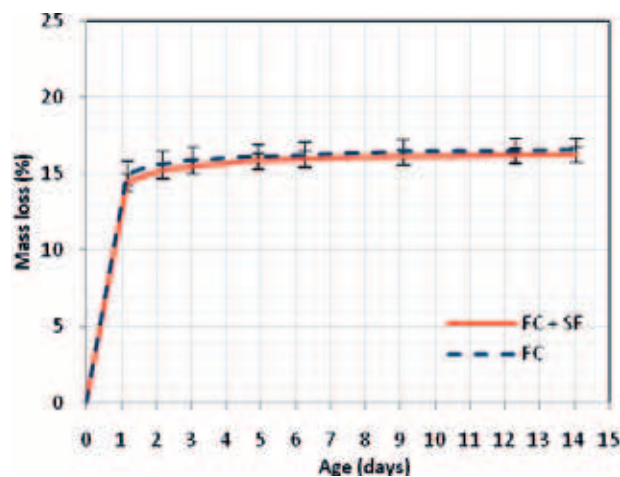


Figure 6 – Mass loss due to drying (water loss)



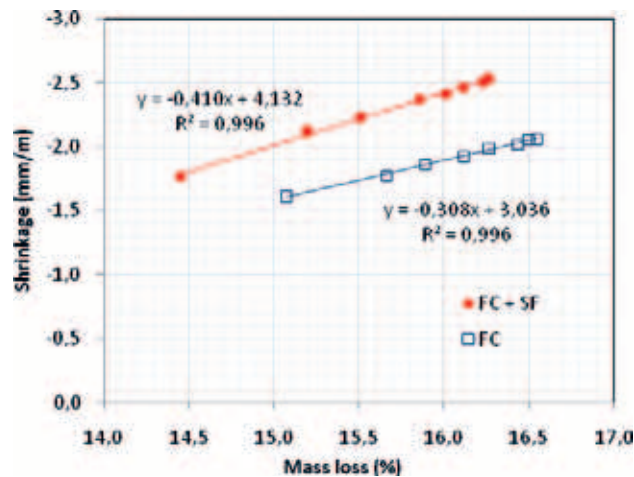


Figure 7 – Relationship between drying shrinkage and mass loss

The refinement of the pores due to the addition of silica fume is confirmed by mercury intrusion porosimetry. The results of this technique have shown a greater volume of pore diameters smaller than 0.05  $\mu\text{m}$  (mesoporous, according to Young, 1988) in samples with silica fume (Figure 8). Pores of this size are equivalent to intergranular voids, also classified as capillary pores. Thus, it can be stated that the addition of silica fume increased the amount of capillary pores, those pores susceptible to changes in capillary tension. The increase in the volume of capillary pores was 74%, increasing from 0.0019 ml/g to 0.0033 ml/g, when the silica fume was added. For the range of macropores (diameters larger than 0.05  $\mu\text{m}$ ) there was no significant difference.

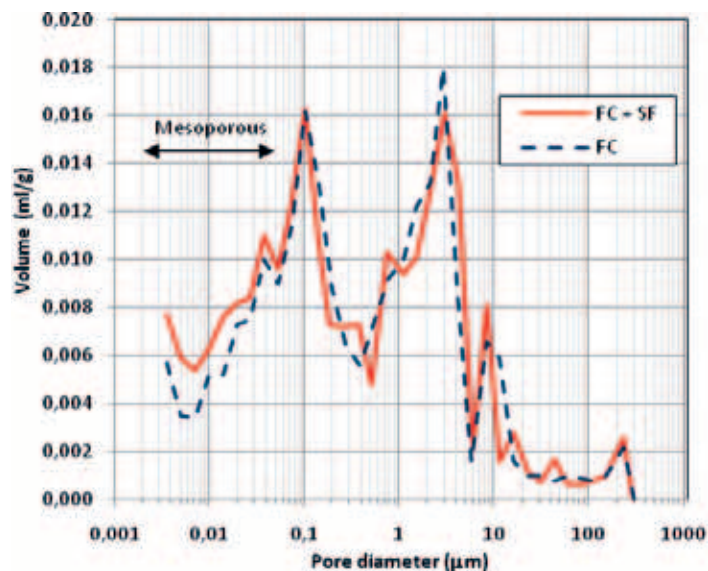


Figure 8 – Size distribution of pores in the samples of fiber cement

In summary, the addition of 3% silica fume by volume in place of limestone filler caused an increase of 74% in the volume of capillary pores in the fiber cement (equivalent to an increase of the capillary pores of 0.82% to 1.49% of total porosity). This change was reflected by a 23% increase in drying shrinkage, which is significant, and can cause damage to fiber cement products, especially in early ages.

An estimation of the fiber cement's internal tension was calculated considering a hypothesis of restrained shrinkage by using Hooke's law ( $\sigma = E \cdot \varepsilon$ ), where shrinkage was equal to the deformation ( $\varepsilon$ ), and flexural strength test was used to measure the Young's modulus ( $E$ ) equal to the internal tension ( $\sigma$ ). The stress relaxation was not considered in this estimate.

The results in Figure 9 show that for differences in shrinkage of around 0.5 mm/m, the difference in internal stress of the composite can be higher than 4 MPa. In the results obtained after 14 days of testing the flexural stress is 28% higher in the sample of fiber cement with silica fume (20.24 MPa for sample FS+SF, and 15.86 MPa for sample FS).

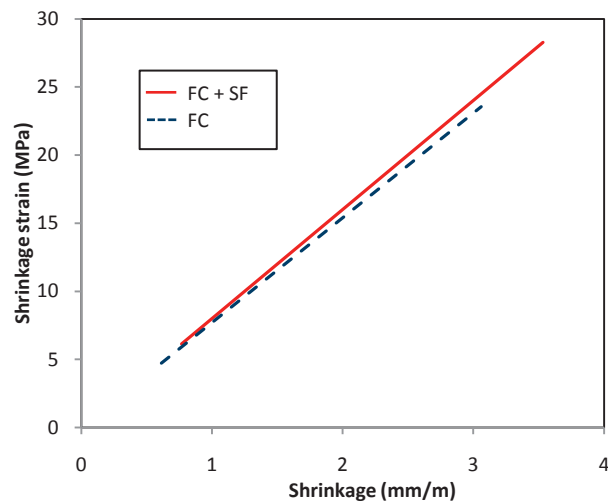


Figure 9 – Shrinkage strain calculated from values of drying shrinkage.

## CONCLUSIONS

The influence of silica fume on the composition of fiber cement was negligible in affecting the mechanical strength of the composite, with no differences in  $E$  and LOP between samples, but the MOR was slightly higher in those samples with silica fume. Further, silica fume significantly influenced the drying shrinkage of the composite, increasing it by 23% when added to fiber cement in place of limestone filler. The addition of silica fume refines the pores of the matrix as evidenced by the results of mercury intrusion porosimetry, and the increase in the volume of capillary pores generates a large increase in capillary pressure which, consequently produces an increase in shrinkage.

With advanced studies on asbestos-free fiber cement, where mechanical properties and durability are increasingly improved, the use of silica fume in the formulation should be adopted with attention. The use of silica fume can cause increased shrinkage, which is currently one of the drawbacks in developing this product, especially considering the cases where silica fume is not well dispersed in suspension, and therefore does not confer the expected increase in mechanical performance. The addition of silica fume to fiber cement should occur in conjunction with other actions to reduce drying shrinkage, such as improvements in the resistance of the matrix, or reducing the rate of drying.

Finally, the importance of determining the size distribution of pores in this type of work should be highlighted, since even small differences in the total porosity of the composite, whether or not this difference occurs in the range of capillary pores, results in shrinkage and the tendency for cracking can be significant.

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## REFERENCES

- Akers, S. A. A. e Partl, M. 1990. Hygral and thermal expansion / shrinkage properties of asbestos-free fibre cement. *Cement and Concrete Research*. 1990, Vol. 12, 1.
- Akers, S. A. S. e Garrett, G. G. 1983. Observations and predictions of fracture in asbestos cement composites. *Journal of Materials Science*. 1983.
- Bastos, P. K. X. 2001. Retração e desenvolvimento de propriedades mecânicas de argamassas mistas de revestimento. *Tese (doutorado)*. São Paulo : Escola Politécnica da USP, 2001.
- Baroghel-Bouny, V. 1994. Caractérisation dès pâtes de ciment et des bétons. *Paris Laboratoire Central dès Ponts ET Chaussées*. 1994.
- Bentur, A. 1979. Effect of curing temperature on the pore structure of tricalcium silicate pastes. *Journal of Colloid and Interface Science*, 1980, Vol. 74, 2, PP. 549-560.
- Devasto, S. 2006. Report about experiences with cement based corrugated sheets vacuum processing. *In: The 10th International Conference on Inorganic-Bonded Composite Materials*. São Paulo, Brazil. 2006, PP. 33-48.
- Diamond, S. 2000. Mercury porosimetry: An inappropriate method for measurement of pore size distributions en cement-based materials. *Cement and Concrete Research*. 2000, Vol. 30, PP. 1517-1525.
- Dias, C. M. R. 2005. Efeitos do envelhecimento na microestrutura e no comportamento mecânico dos fibrocimentos. *Dissertação (mestrado)*. São Paulo : Escola Politécnica da USP, 2005.
- Feldman, R. F.; Sereda, P. J. 1970. A new model for hydrated Portland cement and its practical implications. *Engineering Journal*. 1970, Vol. 53, PP. 509-20.
- Ferreira, J. P. M. 2004. Tensão superficial - sua natureza e seus efeitos. *Boletim da Sociedade Portuguesa de Química*. 2004, 93, PP. 43-48.
- Hua, C., Acker, P. e Ehlacher, A. 1995. Retrait d'autodessiccation du ciment - Analyse et modélisation macroscopique. *Bulletin de Liaison des Laboratoires des Ponts er Chaussées*. 1995, 196, PP. 79-89.
- Hwang, C. L. e Young, J. F. 1984. Drying shrinkage of Portland cement pastes I. Microcracking during drying. *Cement and Concrete Research*. 1984, Vol. 14, 4, PP. 585-594.
- Lobo, C. e Cohen, M. D. 1992. Effects of silica fume on expansion characteristics of expansive cement pastes. *ACI Materials Journal*. 1992, Vol. 89, 5, PP. 481-490.
- Mehta, P. K.; Monteiro, P. J. M. 2008. Concreto – Microestrutura, propriedades e materiais. São Paulo : Ibracon, 3ed, 2008, 674p.
- Mora-Ruacho, J.; Gettu, R.; Aguado, A. 2009. Influence of shrinkage-reducing admixtures on the reduction of plastic shrinkage cracking in concrete. *Cement and Concrete Research*. 2009, Vol. 39, PP.141–146.
- Rao, G. A. 1998. Influence of silica fume replacement on expansion and drying shrinkage. *Cement and Concrete Research*. 1998, Vol. 28, 10, PP. 1505-1509.
- Romano, R. C. O.; Schreurs, H.; John, V. M.; Pileggi, R. G. 2008. Influência da técnica de dispersão nas propriedades de sílica ativa. *Cerâmica*. 2008, Vol. 54, PP.456-461.
- Sato, N. M. N., 1998. Análise da porosidade e de propriedades de transporte de massa em concreto. *Tese (doutorado)*. São Paulo : Escola Politécnica da USP, 1998.
- Savastano Junior, H. e Agopyan, V. 1999. Transition zone studies of vegetable fibre-cement paste composites. *Cement and Concrete Composites*. 1999, Vol. 21, 1, PP. 49-57.
- Savastano Junior, H. 1992. Zona de transição entre fibras e pasta de cimento Portland: caracterização e inter-relação com propriedades mecânicas do composto. *Tese (doutorado)*. São Paulo : Escola Politécnica da USP, 1992.

- Taylor, H. F. W. 1997. Cement chemistry. London : *Thomas Telford*, 2ed, 1997, 459p.
- Toledo Filho, R. D. e Sanjuán, M. A. 1999. Effects of low modulus sisal and polypropylene fibre on the free and restrained shrinkage of mortars at early age. *Cement and Concrete Research*. 1999, Vol. 29, PP. 1597-1604.
- Van der Heyden, L. 2010. Technical specifications of matrix raw materials for Hatschek technology based fibrecement – A pragmatic approach. *Construction and Building Materials*. 2010, Vol. 24, PP. 147-157.
- Wittmann, F. H. 1982. Creep and shrinkage mechanisms. In: Z.P. Bazant, F.H. Wittmann (Eds.), *Creep and Shrinkage in Concrete Structures*, Wiley, Chichester, 1982, PP. 129-161.
- Young, J. F. 1988. Physical mechanisms and their mathematical descriptions. *Mathematical modelling of creep and shrinkage of concrete*. Chichester : Wiley, 1988, PP. 63-98.