

IC-NOCMAT 2007 Maceió
*International Conference on Non-Conventional Materials and Technologies:
Ecological Materials and Technologies for Sustainable Building
Maceió, Alagoas, Brazil, 14th-17th October 2007
In Honour of Professor R.N.Swamy*

**PROSPECTIVE STUDY ON VEGETABLE WASTES AS
REINFORCEMENT IN EXTRUDED FIBRE-CEMENT**

Y.J. M. SOTO¹; G.H.D. TONOLÍ²; R.S. TEIXEIRA³, S.F. SANTOS³; H. SAVASTANO JR.³

¹*Escola Politécnica, Universidade de São Paul, Brazil.*

²*Escola de Engenharia de São Carlos, Universidade de São Paulo, Brazil.*

³*Faculdade de Zootecnia e Engenharia de Alimentos, Universidade de São Paulo - Avenida Duque de Caxias Norte, 225, 13635-900 – Pirassununga, SP, Brazil. E-mail: holmersj@usp.br*

ABSTRACT

The extrusion process involves the formation of cohesive fibre-cement composites by forcing it through a die that can be adjusted to the various shape configurations. These sections are then cut to the desired length. This method can produce composites with high-density matrix and fibre packing, low porosity, and strengthening of the fibre matrix bond. This process also permits the use of residual fibres from the agro industry as raw materials for the production of cost-effective construction elements.

In the present work four different types of waste fibres under 4 mm length were used as reinforcement with concentration of 1% by weight. The fibres were originated from the following different vegetable plants: coir, sisal, banana, and sugar cane. Tailoring of the composites were performed in an auger extruder using ordinary Portland cement, high range water reducer (HRWR), rheological modifier and water soluble lubricant. The mechanical performance of the composites was evaluated by four-point bending test at 28 days of cure. Physical characterization by Archimedes procedure involved the determination of bulk density, apparent porosity and water absorption at the same age. Air permeability evaluation was conducted and the extruded composites presented unimportant incidence of inter-connected pores. This result indicates good performance under weathering because it permits to associate the resistance to the penetration of degradation agents with the structure properties (e.g. particle packing and permeable pores). The extruded composites reinforced with coir and banana fibres maintained their mechanical properties after 200 cycles of soak/dry accelerated aging test.

KEYWORDS: Agro industry; residues; banana fibre; sisal fibre; coir fibre; sugar cane lignocellulose fibre; cellulose; fibre cement; extrusion process.

INTRODUCTION

Natural fibres as reinforcement of fragile matrices based on cementitious materials have induced great interest in developing countries based on their low cost, availability, economy of energy, and also as asbestos substitutes regarding environmental concerns [1]. According to Swamy [2], the use of composites in flat sheets, roofing tiles and pre-manufactured components, can represent significant contribution for the infrastructure of these countries.

The available technologies for asbestos-free fibre-cement are well accepted in the developed world. However, they require large investments that are many times impractical, considering the reality of the low income countries. The developing societies have to look for alternative raw materials. Appropriate fibres and binders, different of the traditional raw material, frequently involve higher costs and great consumption of energy in their processing [3]. Another concern is the durability of the alternative products, as well as their compatibility to the usefulness of the life of the components that are destined to build the homes of the low income population [4].

Tropical countries like Brazil are indeed the domain of natural fibres mainly directed to cordage, textile and papermaking production. The heterogeneity and perishing of such fibres and also the market of restricted uses lead to the intense generation of residues with a high potential of pollution. For example for each ton of commercially used sisal fibres, three tons of residual fibres have been dumped causing hazard to the environment Also the coir fibres, extracted from the mesocarp of the coconut fruit, and banana fibres cut from the pseudo-stem of the plant, are examples of others widely available fibres [4].

The extrusion technique is a current forming process for a wide range of material such as polymers, metals, foods and ceramic pastes [5-7]. Extrusion is the compression process in which materials are forced to flow through a die orifice to provide long continuous product. Cross-sectional shape of component is assured by the shape of the orifice (die). A common type of auger extruder is shown at Figure 1.

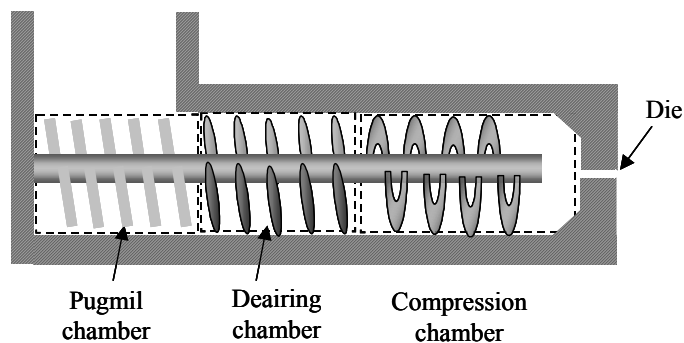


FIGURE 1 - Auger Extruder

The research on extrusion of fibre-reinforced cement-based composites (FRCC) has been started at Northwestern University, using an auger extruder [8]. Several types of products were successfully extruded including fibre-cement flat sheets and tubes proving that extrusion is a promising alternative tool to manufacture FRCC. Extrusion provides a great improvement in the mechanical performance compared to traditional casting methods [9]. This improvement can be attributed to the low porosity and high fibre-matrix bond strength of extruded fibre cement

products. This technique opens a new reality for the cementitious composites performance using vegetal fibre residues.

This work reports on macrostructural behaviour of fibre-cement composites produced by extrusion. Vegetable fibres from agricultural residues (banana, coir, sisal, sugar-cane bagasse) were used as the sole reinforcement of extruded cement based composites.

EXPERIMENTAL

Fibres preparation

Sisal (*Agave sisalana*) field by product was provided by the Associação de Desenvolvimento Sustentável e Solidário da Região Sisaleira (Apaeb), Brazil. Sisal strand fibres originated from residues of the cordage industry with 0.187 mm of average diameter were cut around 4 mm long. Also residuary coir fibres with 0.223 mm average diameter, banana fibre with 0.154 mm, and sugar cane bagasse with 0.24 mm were cut to under 4 mm length to be used as reinforcement.

Properties of the fibres extracted from the literature [10] are presented in Table 1.

TABLE 1 - Macro fibres properties [10]

Properties	Specific mass (kg/m ³)	Tensile strength (MPa)	Modulus of elasticity (GPa)
Coir (<i>Cocos nucifera</i>)	1177	95 a 118	2.8
Sisal (<i>Agave sisalana</i>)	1370	347 a 378	15.2
Banana (<i>Musa cavendishii</i>)	1031	384	20 a 51
Sugar Cane (<i>Saccharum officinarum</i>)	750	170-290	15 a 19

Aiming to determinate their water absorption ability, all of the fibres were immersed for 24 h and shortly after cooled for the same amount of time. 15 g of each fibre were weighted using a thermo-balance and dried at 70°C until constant mass, with maximum variation of 1% . Solid contents obtained were 42% for banana, 28% for sisal, 32% for coir and 30% for sugar cane fibres. These data allows calculating percentage of water absorption of 312% for coir, 357% for sisal 238% for banana, and 333% for the sugar cane fibres.

Matrix

A Portland cement matrix was used with the purpose of observing fibre behavior in a highly alkaline environment.

The matrix material used in this study includes the following ingredients: ordinary Portland cement (CPV ARI, NBR5733 [11]), water soluble polymers, high range water reducer HRWR and a lubricant. The portland cement particles characterization were performed by a laser particle size analyzer. Cement Particles showed 50% of its mass less than 10 microns. Hydroxypropyl methylcellulose was used as rheological modifier, with average molecular weight 86,000 and viscosity at 2% in water at 20°C of 5,385 cPs. A polyether carboxylic with commercial name of ADVA FLOW was used like HRWR. Propylene glycol (PG) with viscosity of 60 cPs at 20°C with average molecular weight of 76.10 was used as water soluble lubricant.

Composite extrusion

Cement composite pads measuring 7 mm x 35 mm and reinforced by the different fibre residues were prepared in the laboratory using an auger extruder without vacuum, Figure 2.

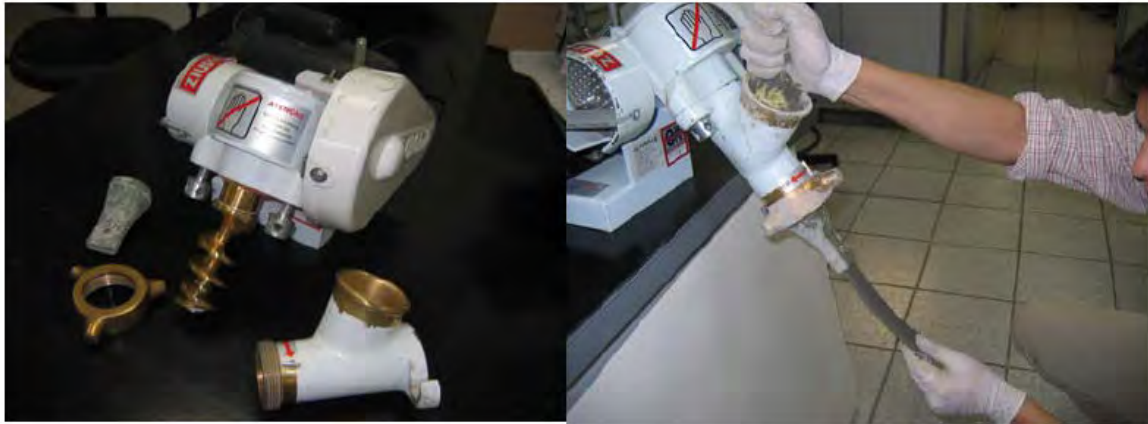


FIGURE 2- Auger Extruder Without Vacuum

The mix-designs evaluated are presented in the Table 3, each using a 0.26 water to cement ratio. The mixtures were accomplished by mechanical stirring, in a planetary mixer EMIC, at low velocity, 140 rpm, during 5 min and finally 15 min at high velocity, 285 rpm. The slurry was transferred to the extruder and recirculated for 5 min before the tailoring of the composites. The composites were sealed wet in a plastic bag to cure at room temperature for two days, and immersed in lime saturated water during 26 days. Specimen thickness was approximately 7 mm. On completion of the curing, specimens were tested at 28 days after production. Prior to mechanical test under saturated condition, specimens were immersed in water for 24 h. At least 6 specimens were tested to obtain the average and standard deviation in each batch.

TABLE 3 – Composites Formulations

<i>Composite</i>	<i>Cement-(%-mass)</i>	<i>Fibres (%-mass)</i>	<i>HPMC-(%-mass)</i>	<i>HRWR-(%-mass)</i>	<i>PG-(%mass)</i>
<i>Coir(Cocos nucifera)</i>	99%	1%	0.5%	0.5%	0.001%
<i>Sisal(Agave sisalana)</i>	99%	1%	0.5%	0.5%	0.001%
<i>Banana (Musa cavendishii)</i>	99%	1%	0.5%	0.5%	0.001%
<i>Sugar Cane (Saccharum officinarum)</i>	99%	1%	0.5%	0.5%	0.001%

Physical characterization tests

Water absorption (WA), bulk density (BD) and apparent void volume (AVV) results were obtained from the average of 12 specimens for each formulation, following the procedures specified by ASTM C [12].

Experimental evaluation of air permeability at room temperature (25 – 30°C) was conducted in a permeameter, as described elsewhere [13]. The testing sample was fixed between two chambers, leaving a circular passing area ($4.91 \times 10^{-4} \text{ m}^2$). The experiments evaluated the easiness of airflow through the sample's thickness (around 8 mm) by measuring the exiting air velocity in response to the variations of the applied inlet pressure. Permeability tests were performed on at least three specimens for each treatment. Samples were previously dried in a ventilated oven at 100°C. The moisture was removed primarily to prevent any influence of water on the measurements. The permeability constants k_1 and k_2 are obtained by fitting the experimental data using Forchheimer's equation (Eq.1), expressed for compressible fluids flow [14]:

$$\frac{P_i^2 - P_o^2}{2P_o L} = \left(\frac{\mu}{k_1} \right) v_s + \left(\frac{\rho}{k_2} \right) v_s^2 \quad (1)$$

where P_i and P_o are the absolute inlet and outlet air pressures, respectively; v_s is the fluid velocity; L the sample thickness; μ the fluid viscosity (air viscosity $\sim 1.8 \times 10^{-5} \text{ Pa.s}$); and ρ the fluid density (air density $\sim 1.08 \text{ kg/m}^3$). The parameters k_1 and k_2 are, respectively, the Darcian and non-Darcian permeability constants.

The permeability constant k_1 , or Darcian constant, represents the viscous effect of the shearing (friction and interactions amongst fluid and porous media); the permeability constant k_2 , or non-Darcian constant, reflects the tortuosity of the porous media when the shearing velocity is high [15]. The higher the k_1 and k_2 values, higher was the permeability of the porous media.

Soak/dry accelerated ageing cycles

The soak/dry accelerated ageing test involved comparative analysis of the physical and mechanical performance of the composites before and after soak/dry cycles. Specimens were successively immersed in water at $20 \pm 5^\circ\text{C}$ during 170 min, followed by the interval of 10 min, and then exposed to the temperature of $70 \pm 5^\circ\text{C}$ for 170 min in a ventilated oven and with the final interval of 10 min. This procedure was based on recommendations of the EN 494 [16] Standards. Each soak/dry set represents one cycle and was performed for 200 times (i.e., 200 cycles).

Mechanical characterization tests

Mechanical tests were performed in a materials testing machine Emic DL-30,000 equipped with 1 kN load cell. A four-point bending configuration was employed in the determination of modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE) and

specific energy values. A span of 135 mm and a deflection rate of 1.5 mm/min were adopted in the bending test. Eqs. 2, 3 and 4 define MOR, LOP and MOE respectively:

$$MOR = \frac{P \cdot L_v}{b \cdot h^2} \quad (2)$$

$$LOP = \frac{P_{lop} \cdot L_v}{b \cdot h^2} \quad (3)$$

$$MOE = \frac{276 \cdot L_v^3}{1296 \cdot b \cdot h^3} \cdot \left(\frac{P}{\delta} \right) \quad (4)$$

where P is the maximum load, P_{LOP} is the load at the upper point of the linear portion of the load-deformation curve, L_v is the major span between the supports, b and h are the specimen width and depth respectively, δ is the deflection of the composite.

Specific energy (Eq. 5) was defined as the energy absorbed during the flexural test and divided by the specimen cross-sectional area. The absorbed energy was obtained by integration of the area below the load-deflection curve to the point corresponding to a reduction in load carrying capacity to 70% of the maximum reached. The deflection during the bending test was collected by the deflectometer positioned in the down side of the specimen.

$$Specific \ energy = \frac{absorbed \ energy}{b \cdot h} \quad (5)$$

TEST RESULTS AND DISCUSSION

28 days-old composites did not show air permeability up to 1 MPa of pressure. This behavior is linked to the fact that there is not a significant net of connected pores. That is due to the low water cement ratio of the mixture and to the die effect on the external surface of the sample, which mitigate water migration throughout the process. High water cement ratio mixtures drained in a process such as Magnani or Hatschek can promote connected nets in the composite.

Exception made to sugar cane composites, water absorption in 28 days does not show relevant difference, as it can be observed in Figure 3. After 200 cycles of aging all of the composites show water absorption reduction, due to the continuous hydration of cement phases and to calcium hydroxide carbonation, all of which close existing pores in the composites.

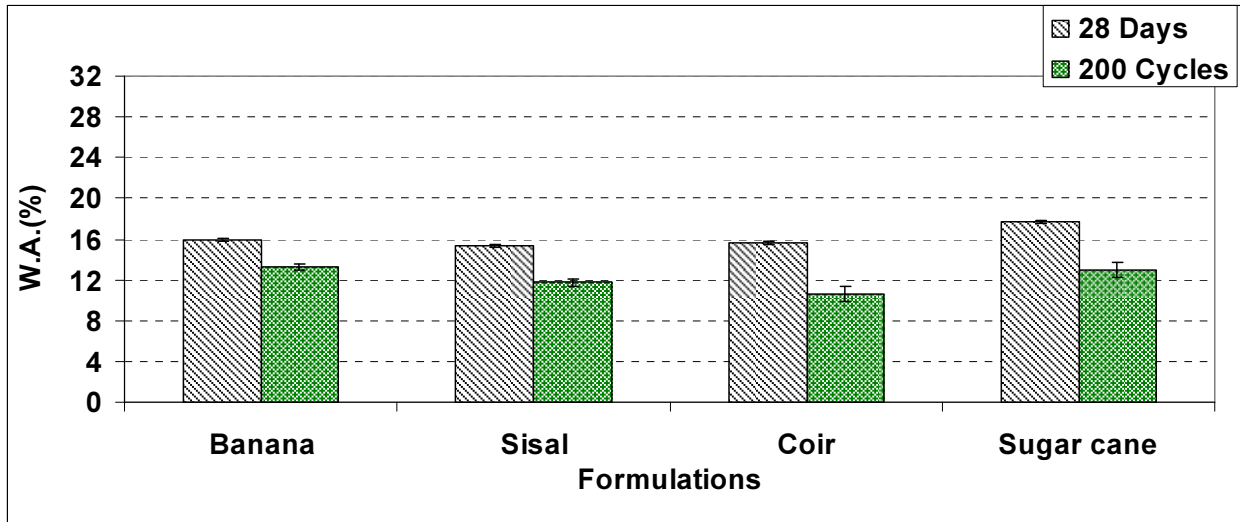


FIGURE 3 - Average values and standard deviations for water absorption (WA) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles.

As demonstrated in Figure 4, composites bulk density do not show pronounced variations in 28 days. That is probably because the various fibres tested do not have enough differentials in matrix imperfections and voids to distinguish them. The 200 cycles of aging was not enough to change the bulk density of the composites.

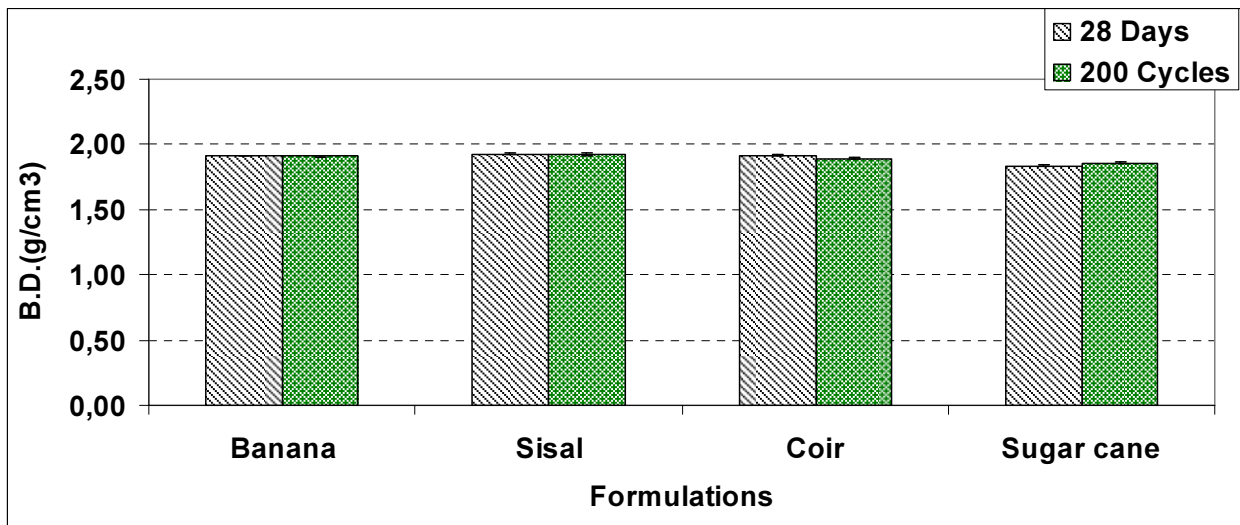


FIGURE 4- Average values and standard deviations for bulk density (BD) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles.

As it can be observed in Figure 5, apparent void volume in 28 days do not differ significantly among composites, except for sugar cane composites, with 17.72% apparent porosity is probably due to this fibre's higher water sorption ability. After aging all composites undergo decreasing in absorption percentage as cement components hydrate and calcium hydroxide carbonatation continues.

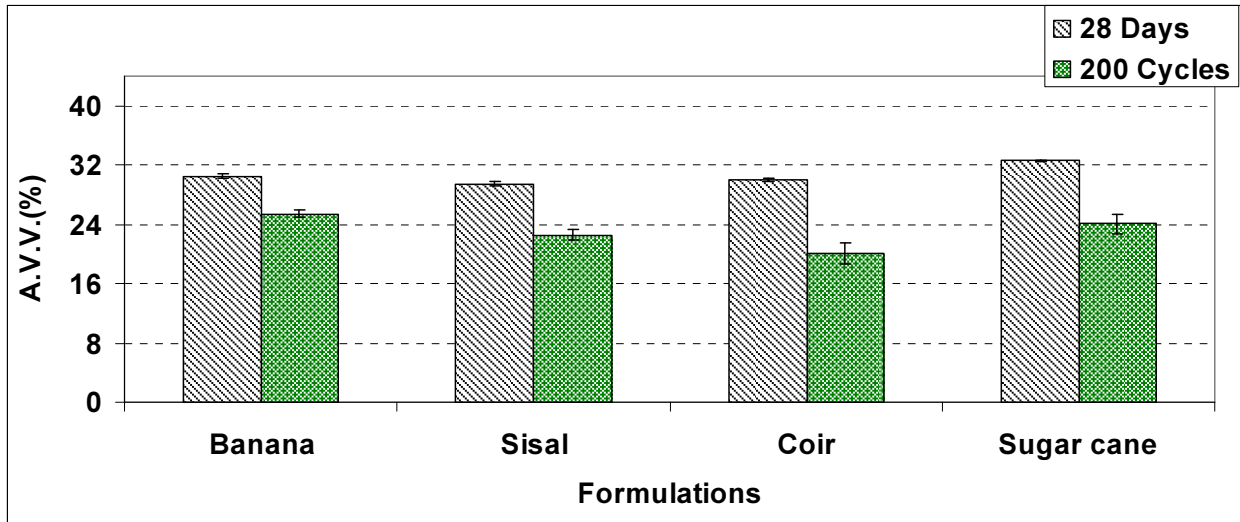


FIGURE 5- Average values and standard deviations for apparent void volume (AVV) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles.

Modulus of rupture averages for 28 day-old composites reinforced with different fibres do not show statistically significant variation. Modulus of rupture varied from 6.78 MPa for banana reinforced composites and 7.95 MPa for coir fibre reinforced composites, as shown in Figure 6. Accelerated aging of 200 soak/dry cycles negatively affects sugar cane and sisal reinforced composites, but the same effect was not observed in banana or coir reinforced composites. Loss of composite mechanical properties are probably due to the presence of calcium hydroxide, as the matrix has no added component capable of consume it and decrease medium alkalinity and protect fibre from degradation.

Banana reinforced composite shows slightly increase in its rupture module, with no statistical significance after 200 cycles. Coir reinforced composites show statistically insignificant reduction of that parameter after 200 cycles. Coir and banana fibres have the lowest water absorption percentage with 238% to banana and 312% to coir fibres. That improves the composites performance after aging, as fibre hygroscopic characteristics provide dimensional variations, lower in these fibres than in the other two. Dimensional variations deteriorate fibre mechanical properties, because its structure changes after absorbing and losing water, and weakens matrix fibre interface [10]. Another condition affecting performance in composite accelerated aging is the content of lignin in fibres. Literature [17-19] present contents of lignin of 10% for sisal, 24% for banana, 29% for coir and 20% for sugar cane fibres. Mohr et al. [20] state that lignin can improve composite durability even tough it is vulnerable to alkalinity. That improvement is achieved for lignin in cell walls restrict dimensional changes in fibre through soak/dry cycling, reducing the fibre-cement debonding. Additionally, it claims that lignin works as a physical barrier. Having that in consideration, it is possible to affirm that the high level of lignin of coir and banana fibres improve their mechanical performance with aging. Sugar cane reinforced composites still present a chemical incompatibility [17] with cement, which compromises matrix/fibre interface, heading to a significant reduction in their mechanic properties while aging.

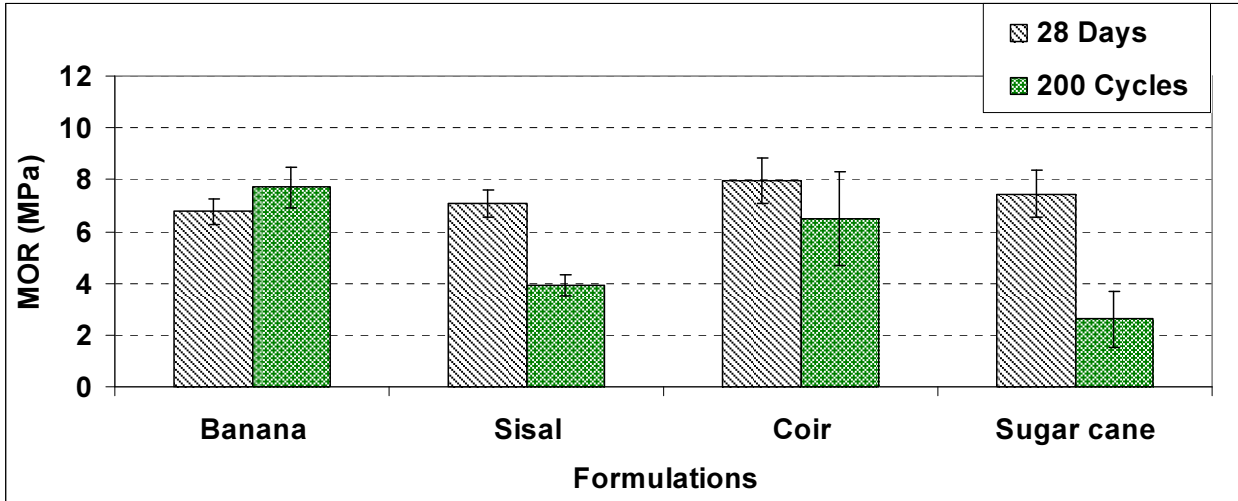


FIGURE 6 - Average values and standard deviations for limit of modulus of rupture (MOR) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles.

Figure 7 presents results for specific energy in 28 days and after 200 soak/dry cycling. Composites do not show statistically significant variation of specific energy for 28-day-old composites.

Specific energy shows no statistically significant variation for 200 aging cycles in composites reinforced with banana or coir fibres. That is probably due to those fibre's high lignin content and lower water absorption ability. On the other hand, specific energies for sisal and sugar cane composites are negatively affected by water absorption and lignin content. Moreover, composites reinforced with sugar cane present chemical incompatibility, which weakens their matrix-fibre interface.

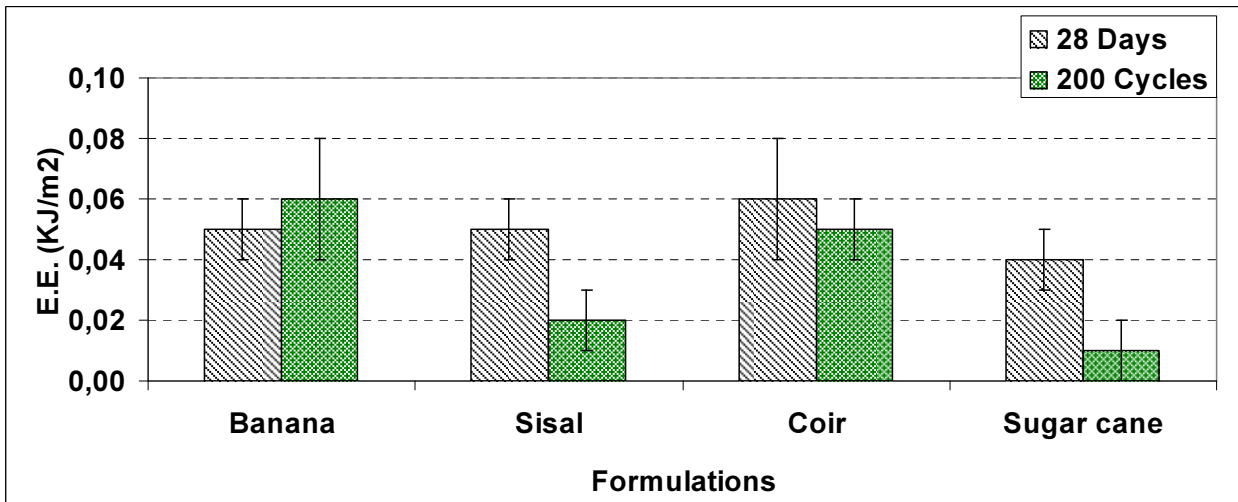


FIGURE 7 - Average values and standard deviations for limit of specific energy (SE) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles.

The performance of limit of proportionality of 28-day-old composites show no significant variation, as presented in Figure 8. Once aged in a 200 soak/dry cycle process, banana and coir

fibres show no variation statistically significant. Sisal and sugar cane fibres have their performance decreased.

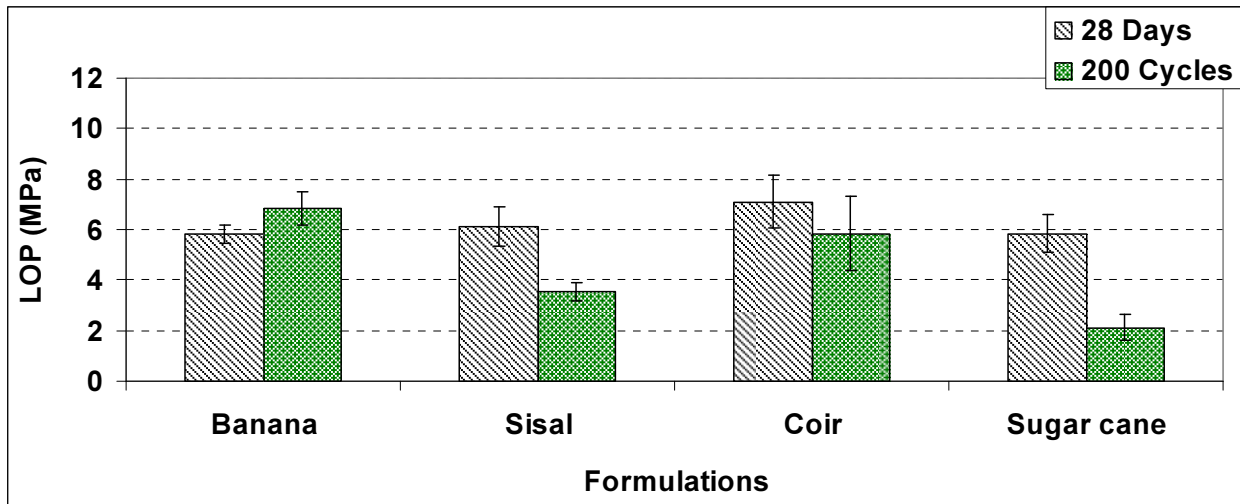


FIGURE 8 - Average values and standard deviations for limit of proportionality (LOP) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles.

Modulus of Elasticity in 28 days do not present significant variation for any of the four composites, as it is seen in Figure 9. After accelerated aging composites containing sisal and coir show no significant variation.

Composites reinforced with banana present an increasing modulus of elasticity, which can be related to the densification of the matrix caused by the continuous cement hydration and calcium hydroxide carbonation. Densification can be evidenced in Figure 3 with a reduction of water absorption and in Figure 5 with the reduction of apparent voids volume. Reduction in the modulus of elasticity of composites reinforced with sugar cane can be related mainly to chemical incompatibility with the cement.

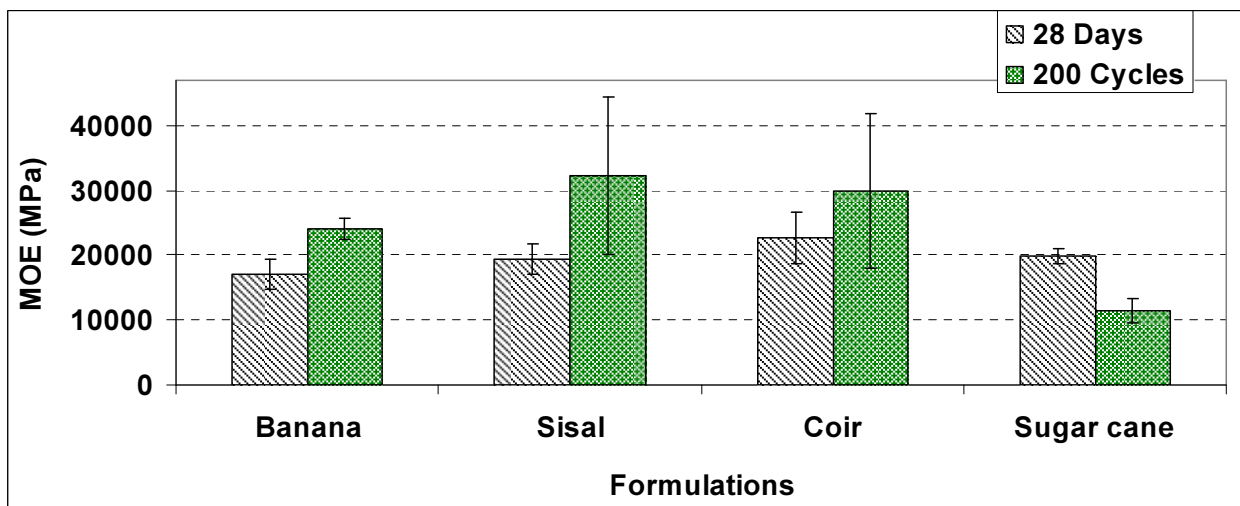


FIGURE 9 - Average values and standard deviations for modulus of elasticity (MOE) of the extruded composites reinforced with the different fibres, at 28 days and after 200 ageing cycles

Conclusions

Extruded composites of an alkaline cementitious matrix, reinforced with 1% by mass of banana, sisal, coconut and sugar cane fibres had both their mechanical and physical performances evaluated, leading to the following conclusions:

- The mechanical performance for the four fibre cements is similar at 28 days.
- At 28 days of aging, sugar cane reinforced composite shows higher water absorption and higher volume of apparent voids.
- After 200 soak/dry accelerated ageing cycles banana and coir reinforced composites maintain their mechanical properties.
- Sisal and sugar cane reinforced composites do not maintain their mechanical properties after 200 wet/dry aging cycles.
- Composites at 28 days of age do not present significant permeable pore net at a pressure of 1 MPa.

Acknowledgements

The authors would like to thank the Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP) and to the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), Brazil for the support to the development of this work.

REFERENCES

1. MacVicar, R, Matuana LM, Balatinecz JJ. Aging mechanisms in cellulose fibre reinforced cement composites. *Cement & Concrete Composites* 1999; 21(3):189–96.
2. Swamy RN. Influence of slow crack growth on the fracture resistance of fibre cement composites. *The International Journal of Cement Composites* 1980; 2(1) : 43-53.
3. Coutts, RSP. A review of Australian research into natural fibre cement composites. *Cement & Concrete Composites* 2005; 27(5):518-526.
- 4 Savastano Jr H, Agopian V, Nolasco AM, Pimentel L. Plant fibre reinforced cement components for roofing. *Constr Build Mater* 1999; 13(2): 433-8.
5. Groover MP *Fundamentals of Modern Manufacturing*. New Jersey: Prentice Hall, 1996.
6. A. Cheyne; J. Barnes; D.I. Wilson, Extrusion behaviour of cohesive potato starch pastes: II. Microstructure–process interactions. *Journal of food Engineering* 2005; 66(1): 1-12..
7. Burbidge AS, Bridgwater J, *The Single Screw Extrusion Of Pastes Chemical Engineering Science* 1995; 50(16): 2531-2543..
8. Mu B. *Short Fibre-Reinforced Cementitious Composites Manufactured by Extrusion Technology*. Ph.D. Thesis, Hong Kong, Hong Kong University of Science and Thecnology, 1999.
9. Shao Y, Shah SP, Mechanical properties of PVA fibre reinforced cement composites fabricated by extrusion processing. *ACI materials Journal* 1997: 94(6): 555-564 .

10. Savastano Jr H, *Materiais à Base de Cimento Reforçados com Fibra Vegetal: Reciclagem de Resíduos para a Construção de Baixo Custo*. Livre Docência Thesis, São Paulo, Escola Politécnica da Universidade de São Paulo, 2000.
11. Associação Brasileira de Normas Técnicas. NBR5733, Cimento Portland de Alta resistência inicial, Rio de Janeiro, RJ, Brazil. 1991.
12. American Society for Testing and Materials. C 948-81, Test Method for Dry and Wet Bulk Density, Water Absorption, and Apparent Porosity of Thin Sections of Glass-Fibre Reinforced Concrete. West Conshohocken, PA, USA. 1981.
13. Innocentini, MDM, Pardo ARF, Pandolfelli VC. Modified Pressure–Decay Technique for Evaluating Highly Dense Refractories Permeability, *J. Am. Ceram. Soc.* 2000; 83(1): 220–22.
14. Innocentini MDM, Pardo ARF, Menegazzo BA, Bittencourt LRM, Rettore RP, Pandolfelli, V C. Permeability of high-alumina refractory castables based on various hydraulic binders. *J. Am. Ceram. Soc.* 2002; 85(6), 1517–1521.
15. Salomão R, Cardoso FA, Innocentini MDM, Bittencourt LRM, Pandolfelli VC.. Polymeric fibres and the permeability of refractory castables. *Cerâmica* 2003; 49(310): 23-28.
16. European Committee for Standardization, EN 494. Fibre-cement profiled sheets and fittings for roofing – Products specification and test methods. 1994. BSI – British Standards Institution, London, UK. 1994.
17. Sarmiento R. C. *Argamassa de Cimento Reforçada com Fibras de Bagaço de Cana-de-açúcar e sua Utilização Como Material de Construção*. D.M. Thesis, Campinas, Universidade estadual de campinas, 1996.
18. Bilba K, Arsene M.A., Ouensanga A. Study of banana and coconut fibres botanical composition, thermal degradation and textural observations. *Bioresource thecnology* 2007; 98 (1): 58-68.
19. Lima LRP. *Análise teorica e experimental de compósitos reforçados com fibras de sisal*. Phd Thesis. Rio de Janeiro, Universidade Federal do Rio de Janeiro,2004.
20. Mohr BJ, Biernacki JJ, Kurties KE. Microstructural and chemical effects of wet/dry cycling on pulp fibre-cement composites. *Cement and Concrete Research* 2006: 36(7):1241-1251.