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# Thermal and Chemically Modified Plant Fibres as Reinforcement In Cementitious Matrices

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#### ABSTRACT

The effect of hornification and hydrophobic silica (Si) coatings on the performances of plant fibre cement composites were assessed. Three macro fibres and a micro fibre, all from plant sources were used as reinforcement. Flexural results indicated that Si coated fibres increased the modulus of rupture (MOR), limit of proportionality (LOP) and modulus of elasticity (MOE) of cement composites but reduced specific energy (SE) while hornification decreased the above properties. Likewise, decreased water absorption, increased bulk density, pore volume and diameter were observed in Si coated fibre composites. Overall Si coatings on control and hornified fibres showed better pre peak behaviour in composites compared to purely hornified fibres which had a slightly better post peak performance.

#### **1.0 INTRODUCTION**

Plant-based fibres offer a sustainable alternative to plastic in fibre cement composites. However, the alkaline environment of cement is aggressive to these fibres and limits their use. For enhanced fibre cement composite durability, plant fibres have been modified in several ways as revealed by other researchers. Sisal fibres were subjected to a series of wetting and drying cycles (hornification) which improved the strength, strain and bonding of fibre with the matrix [1]. Methaacryloxypropyltri-methoxysilane and aminopropyltri-ethoxysilane were used as coatings on pulps [2] and were found to influence fibre matrix interface and fibre mineralization. Si deposited on pulp as a function of synthesis time and precursor content [3] indicated increased deposition and bonding with time and enhanced thermal stability. Furthermore, it was observed that the Si modified pulps accelerated hardening with no inhibition to cement paste hydration [4].

This study subjected plant fibres to hornification and silica (Si) coatings. Hybrid fibres made of three macro fibres and a micro fibre were fabricated to assess the short-term physical and mechanical performances of the plant fibre cement composites.

#### 2.0 MATERIALS AND METHODS

Macro fibres from hemp, flax and palm plants; and micro pinus fibres were used in a hybrid combination. 4 mm long hemp, flax and palm fibres were subjected to 2, 4 and 4 cycles respectively of hornification as proposed by [1], whereas the micro pinus fibres were not. As received (control) fibres and hornified fibres were immersed in Si suspensions synthesized via the sol-gel method with 1.6, 0.4, 0.1 and 0.00025 mol of water, ethanol, tetraethoxysilane and nitric acid with 1% of hexadecyltrimethoxysilane by total volume of other reagents for 30 minutes. After which the fibres were oven dried at 80°C for 10 minutes. Fibres were incorporated in matrices of Portland cement and limestone. A mix design as

indicated in Table 1 was used to produce 200 x 200 mm hybrid fibre composite pads with the slurry dewatering process accompanied by pressing method. An average drainage time of 82 s was recorded for composites during vacuum extraction of water. The pads were compressed at 3.2 MPa for 5 minutes and cured in a saturated atmosphere for 28 days.

Sample ID	Cement	Limestone	Fibre Mass (g)			Fibre trea	atment	
	(g)	(g)	Pinus	Hemp	Flax	Palm	Туре	coating
UCHYC	264.00	48.50	5.00	3.80	4.30	4.30	Control	-
CHYC	264.00	48.50	5.00	3.80	4.30	4.30	Control	silica
UCHNHYC	264.00	48.50	5.00	3.80	4.30	4.30	Hornified	-
CHNHYC	264.00	48.50	5.00	3.80	4.30	4.30	Hornified	silica

Table	1. Mix design	of hybrid	plant fibre	cementitious	composites
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Each pad was cut into four prismatic specimens 165 x 40 mm in dimension and saturated in water for 24 hours before test. Four-point bending was conducted on a universal testing machine Emic DL-30,000 with a 1kN load cell at a deflection rate of 1.5 mm/min and span of 135 mm. Composite pores were measured with a Thermo Scientifc Mercury Porosimeter Pascal 440. Optical and scanning electron microscope images were obtained with a VHX-6000 digital microscope and JEOL JSM-6480LV respectively.

#### 3.0 RESULTS AND DISCUSSION

The mechanical and physical performances of composites are summarized in Table 2. Si coated hybrid composites possessed higher modulus of rupture (MOR), limit of proportionality (LOP), modulus of elasticity (MOE) and lesser specific energy (SE) compared to uncoated hybrid composites. Hornification reduced all mechanical properties and bulk density (BD) but increased water absorption (WA) of composites. The reductions were seen to be greater in uncoated fibre composites compared to coated fibre composites.

Sample ID		Mecha	Physical			
	MOR (MPa)	LOP (MPa)	MOE (GPa)	SE (kJ/m²)	WA (%)	BD (g.cm <sup>-3</sup> )
UCHYC	5.92 ± 1.27	5.00 ± 1.21	8.30 ± 0.44	0.42 ± 0.15	16.41 ± 0.32	1.65 ± 0.02
CHYC	6.33 ± 0.95	5.52 ± 1.49	9.35 ± 1.85	0.28 ± 0.17	16.36 ± 0.74	1.72 ± 0.04
UCHNHYC	5.04 ± 1.16	$4.09 \pm 0.80$	6.71 ± 2.34	0.38 ± 0.24	17.52 ± 1.41	1.56 ± 0.06
CHNHYC	6.16 ± 0.87	5.52 ± 0.71	8.59 ± 1.30	0.31 ± 0.13	17.04 ± 0.65	1.70 ± 0.02

**Table 2.** Average values and standard deviations of the mechanical and physical properties of silica coated and uncoated hybrid fibre composites

The SE of coated hornified fibre composites were found to be greater than the coated control fibre composites. Likewise, coated hybrid fibre composites absorbed less water and possessed higher densities than uncoated composites. Table 3 gives the pore properties of hybrid composites. The Si coatings increased porosity, pore volume, pore diameter but decreased the pore surface area of composites; while hornification increased porosity, pore volume, surface area but decreased pore diameters of composites. Composites with coated hornified fibres had the highest pore volume and porosity. Optical images in Figure 1(a) show the dispersion of most plant fibres in the lower half of the composites. This might be due to the higher density of plant fibres compared to the cement paste slurry and the negative vacuum applied. The lower performances of coated fibre composites could be attributed to the large pores or cracks seen running along the side of the fibres. Figure 1(b) shows the debonding between fibre and matrices as indicated by the black carbon (resin) around the fibres. All fibres, control, hornified and coated fibres, lost water and shrunk away from the cement matrix. A significant number of pores were observed in coated fibre composites composites composites compared to uncoated fibre composites.

Table 3: Total pore volume (TPV), total pore surface area (TPSA), average pore diameter
(APD), porosity of silica coated and uncoated hybrid fibre composites

Sample ID	TPV (mm³/g)	TPSA (m²/g)	APD (nm)	Porosity (%)
UCHYC	134.69	10.77	50.05	22.85
CHYC	142.17	9.79	58.09	24.21
UCHNHYC	166.69	17.32	38.50	27.09
CHNHYC	180.79	14.25	50.75	28.96

There was no obvious silica interface between fibre and matrix which supports the assumption that coatings reacted with cement to form hydration products. Alternatively, the coatings might have been washed off the fibres during the fabrication process. Air pockets might have formed about fibres and coatings with voids/depressions during production.

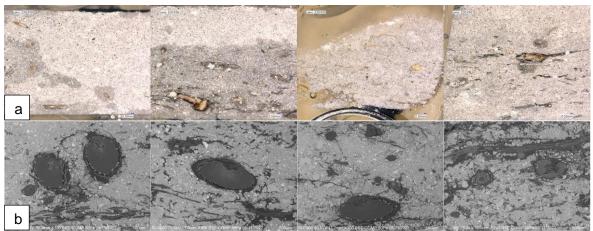


Figure 1: (a) Optical and (b) SEM of UCHYC, CHYC, UCHNHYC and CHNHYC (left to right)

#### 4.0 CONCLUSION

Stresses induced in fibres during hornification and matrix stiffness resulted to decreased mechanical performance, while Si coating did the reverse. The lower post peak performance of the coated fibre composites relative to control composites can be attributed to the fabrication method which left very large pores along plant fibres resulting in quicker debonding from matrices. The use of silica coatings on control and hornified plant fibres in composites is viable and significantly enhances properties within the elastic zone.

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#### References:

1. Ferreira, S.R., et al., *Effect of Sisal Fiber Hornification on the Fiber-Matrix Bonding Characteristics and Bending Behavior of Cement Based Composites.* Key Engineering Materials, 2014. **600**: p. 421-432.

2. Tonoli, G.H.D., et al., *Cellulose modified fibres in cement based composites.* Composites Part a-Applied Science and Manufacturing, 2009. **40**(12): p. 2046-2053.

3. Raabe, J., et al., *Evaluation of reaction factors for deposition of silica (SiO(2)) nanoparticles on cellulose fibers.* Carbohydr Polym, 2014. **114**: p. 424-431.

4. Raabe, J., et al., Effect of Nano-silica Deposition on Cellulose Fibers on the Initial Hydration of the Portland Cement. Bioresources, 2018. **13**(2): p. 3525-3544.