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Evaluation of the green coconut fiber compatibility with cement by using different calculation methods

Using all the different methods reported in the literature, the compatibility of coconut green fiber with Portland cement in five different compounds was evaluated. For each trace, the mixtures hydration curves (temperature x time) were obtained by the use of type K thermocouples and an analog signal receiver. Based on a unique set of experiments with coconut fiber, a comprehensive critical review of the methods reported in the literature was performed to evaluate fiber-cement compatibility. Contradictory results about compatibility classification were obtained using different compatibility equations. The correlations between compatibility and compressive-strength essays results were considered to support our choice of the best method of compatibility calculation. Greater sensitivity to capture the differences in the compositions of mixtures and a higher correlation with the compressive strength of composites revealed that the CX calculation method, which considers the heat release rate as a basic parameter, presented more realistic results.

Keywords: Fiber-Cement; Composites; Compatibility Equations; Coconut Fiber; Hydration Curve.

Avaliação da compatibilidade entre fibras de coco verde e cimento usando diferentes métodos de cálculo

Utilizando diferentes métodos de cálculo relatados na literatura, avaliou-se a compatibilidade da fibra de coco verde com cimento Portland em cinco diferentes compósitos. Para cada traço, as curvas de hidratação das misturas (temperatura x tempo) foram obtidas utilizando termopares do tipo K e um receptor de sinais analógicos. Com base em um conjunto único de experimentos com fibra de coco, uma revisão crítica e abrangente dos métodos relatados na literatura foi realizada para avaliar a compatibilidade entre a fibra vegetal e o cimento. Resultados contraditórios sobre a classificação da compatibilidade foram obtidos quando se analise as diferentes equações de compatibilidade. As correlações entre os resultados de compatibilidade e o de resistência à compressão dos compósitos foram considerados na análise da escolha do melhor método de cálculo de compatibilidade. O método de cálculo CX revelou uma maior sensibilidade para capturar as diferenças nas composições das misturas e foi o que obteve uma maior correlação com a resistência à compressão dos compósitos, pois considera a taxa de liberação de calor como parâmetro básico, apresentando resultados mais realistas.

Palavras-chave: Fibra-Cimento; Compósitos; Equações de Compatibilidade; Fibra de Coco; Curva de Hidratação.

Topic: **Engenharia da Sustentabilidade e Meio Ambiente**

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INTRODUCTION

Brazil has a prominent role in green coconut production; it is the fourth largest producer with an output equivalent to two billion fruit/year according to the Brazilian Institute of Geography and Statistics / IBGE. Only 10% of all generated coconut residue is recycled, the remaining goes to garbage dumping ground in accordance with the Brazilian agricultural research agency (EMBRAPA, 2015). On the other hand, there is an increase in the number of scientific researchers focused on the increase of innovative and environmentally friendly technologies that reuse this material. The green coconut husks (Cocos nucifera L.) are agricultural waste with a possible high utilization potential in the civil construction sector. Particularly, in the Portland cement matrices, the use of forestry species bark is usually not recommended due to the high sugar content and other extractives which may interfere with cement hydration reactions (SANDERMANN et al., 1964; WEATHERWAX et al., 1964; HOFSTRAND, et al., 1984; HACHMI et al., 1990), which makes the mixture less compatible (Kared, 2010). However, some authors have recently reported the existence of certain removal treatments of the inhibitory substances that can increase the compatibility of the vegetable fibers with the binder.

Asasutjarit et al. (2007) proposed a pre-treatment based on washing fibers with hot water before being incorporated into cementitious matrices. By studying coconut fiber with cement, Ferraz et al. (2012) verified that washing treatments with NaOH and addition of $CaCl_2$ achieved the best results, taking into consideration the compatibility of the fiber, which was classified as low inhibition.

The compressive-strength of fiber cement composites is affected by presence of vegetal fiber in the cementitious matrix (LIMA et al. 2015). Vegetable fiber presence in the cementitious matrix delays the cement hydration due to the fiber's organic composed have reaction capacity with cement calcium preventing the formation of hydrated calcium silicates (C-S-H), responsible for increasing of the composite's setting time.

Aiming to measure the degree of vegetable fibers compatibility with Portland cement, several theoretical methods have been developed in recent decades. The hydration curve (temperature vs. time) of wood-cement composites is widely used to define the behavior of compatibility between lignocellulosic fibers and cement. These profiles evaluate qualitatively, in a simple way, the behavior of the cement setting process with the addition of vegetable fiber.

Several researchers have studied different methods of compatibility evaluation based on different approaches and mathematical formulations, taking account the maximum hydration temperature (SANDERMANN et al., 1964), maximum time to reach maximum temperature of hydration (WEATHERWAX et al., 1964), maximum temperature of hydration and ambient temperature (VILELA et al., 1968) and the maximum temperature of hydration and the derivative of the temporal function of temperature (HOFSTRAND et al., 1984).

Others models of calculation took into consideration, besides the previous parameters evaluated by the authors (time and maximum temperature), as the quantities and thermal system parameters (amount

of wood, specific heat of composite constituents, the total energy of hydration reaction of mixture, maximum rate of heat and the total heat released within a time interval) (HACHMI et al., 1990; KARED, 2010; ASASUTJARIT et al., 2007; FERRAZ et al., 2012; VILELA et al., 1968; HERRERA et al., 2008; PASCA et al., 2010; KAREDE et al., 2003). A compatibility evaluation scheme using the time factor of the cement setting process (TR), which is the ratio between the initial time of the hydration process of wood-cement mixing with the fresh cement was proposed (OLORUNNISOLA, 2008). An approach taking into consideration the areas under the hydration curve of wood-cement mixtures with pure cement was reported (HACHMI et al., 1990; FAN et al., 2012). However, there are still no standardized methods to measure the temperature of hydration wood-concrete mixtures, and the complexity of binder hydration process may hamper the interpretation of results (KAREDE et al., 2003).

Two open questions motivated the present work. The first relates to methods for evaluating compatibility. In general, different evaluation methods and approaches have been used in studies, separately. There were no studies to compare the information of the fiber-cement compatibility that each of these methods offers from a single experiment data. The second question concerns to the assessment of the fiber-cement compatibility in the specific case of green coconut fiber. Considering the significant volume of waste coconut fibers generated in Brazil and other tropical countries of the world, and the potential its use can represent in construction, it will be very helpful the deepening on the question about the 'compatibility' for this fiber. Thus, the objective of this study was to evaluate, compare and discuss different methods of calculations reported in the literature to measure the chemical compatibility between the green coconut fiber with Portland cement.

MATERIALS AND METHODS

Materials

Green coconut fibers were used and obtained from local business in the neighborhood called Salobrinho, in Ilhéus/BA. The fibers obtained from the coconut mesocarp cut were passed in a press for the removal of water, then they were dried in air, for forty-eight hours, milled in a ball mill for 90 minutes and classified with the passing granulometry in the sieve of 50 mesh and retained in the sieve of 80 mesh. For the use in testing, the fibers were dried in an oven at 102 ± 2 ° C for 24 hours. The cement used was CPV-ARI, with deionized water and aqueous extractive from the coconut fiber. The water/cement ratio (A/C) was set at 0,4 for maintaining an adequate amount of water, able to hydrate all the cement grains without impairing the workability of the mix when added the vegetable fibers. Only for the trace TC5, due to the incorporation of active silica, the A/C ratio used was 0,44. The proportion of coconut fiber was 5% regarding to the cement mass to ensure hydration conditions with the A/C fixed ratio (BILBA et al., 2003). The active silica, with specific mass of 2,22 g/cm³, was used in a proportion of 10% regarding to cement mass. In Table 2 are shown the compositions of the studied features.

Treatment of fibers

The treatment of the coconut fiber was carried out with hot water at 80 ° C and with water at 23 °C, ambient temperature. The coconut fibers were placed in a Eerlenmeyer of 500 ml filled with deionized water in the 7x1 proportion (water/fiber). The Eerlenmeyer was covered with aluminum foil and plunged into thermostatic bath at 80 °C for 1 hour. Then, the fibers were filtered to separate the aqueous extract (used in trace TC2). After this procedure, the fibers were washed (TC4) in deionized water at ambient temperature and filtered on a fine mesh cloth until the filtered water came out clear, then they were dried in a stove at 102 °C ± 2 °C for 48 hours.

Composites Preparation

In this work was used the cement Portland CPV – ARI, in a Blaine superficial area of 4.397 cm^2/g and specific mass of 3,09 g/cm^3. A Cement chemical analysis is shown in table 1. Five different traces were elaborated for the development of the work. The composition for each of these traces is viewed in table 2.

Table 1: Chemical composition of the cement, wt (%).

Oxides materials	**Na_2O**	**MgO**	**Al_2O_3**	**SiO_2**	**P_2O_5**	**SO_3**	**K_2O**	**CaO**	**TiO_2**	**MnO**	**Fe_2O_3**	**SrO**
CPV-ARI	0,62	2,96	3,90	15,92	0,13	4,06	0,76	67,73	0,33	0,11	3,20	0,22

Table 2: Composition of the different fiber-cement composites studied.

Sample	Composition	Mass (g)			
		Cement	Water	Fiber	Silica Fume
TC 1	CPV – ARI and deionized water (Control)	200	80	-	-
TC 2	CPV – ARI and water with extractives (No Fiber)	200	80	-	-
TC 3	CPV – ARI, deionized water and raw fiber	200	80	5	-
TC 4	CPV – ARI, deionized water and washed fiber	200	80	5	-
TC 5	CPV – ARI, deionized water, raw fiber and silica	180	80	5	20

The materials of each trace were placed in sealed plastic bags and homogenized manually during 2 minutes. Subsequently, the temperature sensor was completely introduced into the mixture, the plastic bag was sealed and wrapped with aluminum foil, as shown in figure 1. In addition, the mixtures were transferred to an adiabatic polystyrene chamber, which was sealed and placed in another chamber of the same material to avoid heat leak.

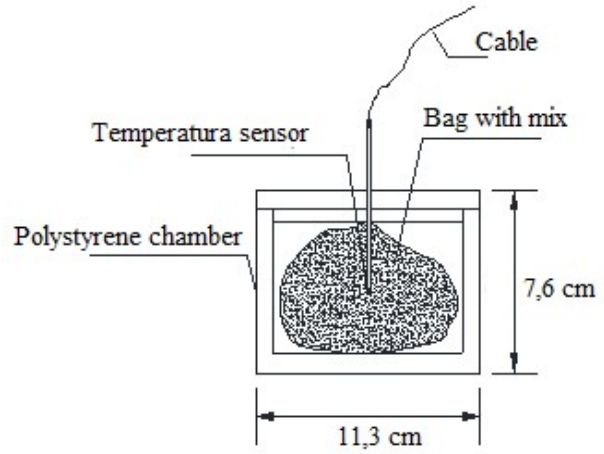

Figure 1: Sketch of temperature measure system.

Hydration test mixtures

To perform the temperature measurements, it was developed the 'SISTEMP' system, in which were used six thermocouples cables type K, connected to an analog signal receiver coupled to an analog-digital conversion board installed on a computer, where the temperature data were processed and stored. Temperature readings, in each connected cable, were programmed to be performed at pre-set time interval of 1 second for a period of 48 hours. Statistical analyzes were performed to define the precision and the accuracy of the sensors by applying the Kolmogorov-Smirnov test, with 95 % of assurance. The results of the six used sensors showed a high precision and accurate with variation <5%. The hydration tests of the five mixtures studied were performed in triplicate and the average value considered.

Calculation of the compatibility between green coconut fiber and Portland cement

The reported methods in literature for the purpose of measuring the compatibility of lignocellulosic materials and Portland cement were classified according to four different calculation approaches: temporal temperature evolution, time evolution of heat release rate, area under the curve and setting time. In Table 3 is showed a summary of all found formulas, reported in literature for calculating compatibility and inhibition indicators applied to fiber-cement composites. These formulas were implemented in the computational code FIBCEM developed in FORTRAN language.

The compatibility indexes proposed in the literature A (Aptitude (VILELA et al., 1968)), C_T (Weighted maximum temperature rate ratio (HACHMI et al., 1990)), C_H (Maximum heat rate ratio (HACHMI et al., 1990)), C_I (Compatibility index (KAREDE et al., 2003)), C_X (Cross compatibility index (PASCA et al., 2010)) and C (Area ratio (FAN et al., 2012)) have proportional behavior with fiber-cement compatibility, i.e., their values increase with greater compatibility. Indicators I_{1964} (Inhibitory index (WEATHERWAX et al., 1964)), I_{1984} (Inhibitory index (HOFSTRAND et al., 1984)) and TR (Setting time factor (OLORUNNISOLA, 2008)) have inverse behavior. Experimental tests were conducted to determine the start times of the setting time in studied mixtures according to NBR NM 65(ABNT, 2003), with A/C ratio set at 0,4. Each composite (Table 2) was tested with the SISTEMP support and with the temperature data vs. time, it was possible to construct the hydration curve of each mixture. From the hydration curves the parameters present in table 5 were calculated.

Table 3: Summary of compatibility evaluation methods used at work.

REF	CALCULATION MODEL	COMPATIBILITY CLASSIFICATION, APTITUDE OR INHIBITION RATE	
Temporal evolution of temperature		$t_{máx}$ ≤ 15 horas 15 horas > $t_{máx}$< 20 horas $t_{máx}$ ≥ 20 horas	Suitable Intermediatelyadequate Inappropriate
Weatherwax and Tarkow, (1964)	$I_{1964} = \left(\frac{t_{máx} - t'_{máx}}{t'_{máx}}\right).100$		
Sandermann and Khöler, (1964)	-	$T_{máx}$ ≥ 60°C	Suitable
		50°C≤ $T_{máx}$<60°C	Intermediatelyadequate
		$T_{máx}$< 50°C	Inappropriate
Vilela and Du Pasquier, (1968)	$A = \left(\frac{T_m - T_o}{T_M - T_o}\right).100$	A ≥ 80%,	Very good
		80% >A ≥ 60%,	Good
		60% > A ≥ 50%	Regular
		A < 50%	Bad
		I<10	Low inhibition

Hofstrand et al. (1984)	$I_{1984} = \left[\left(\frac{t_{máx} - t'_{máx}}{t'_{máx}}\right).\left(\frac{T'_{máx} - T_{máx}}{T'_{máx}}\right).\left(\frac{S' - S}{S'}\right)\right].100$	Okino, et al. 2004	10<I<50	Moderate inhibition
			50<I<100	High inhibition
			I>100	Extreme inhibition
Hachmi and Moslemi, (1990)	$R_T = \left(\frac{T_{máx} - T_R}{t_{máx}}\right).\left(\frac{m_w + m_1}{m_c}\right)$	0% - Species that completely inhibit tricalcium silicate - Not compatible 100% - Pure cement (no inhibition) - Compatible		
	$C_T = \left(\frac{R_T}{R'_T}\right).100$			
	$R_H = \left(\frac{T_{máx} - T_R}{t_{máx}}\right).(mc_w + mc_1 + mc_c + mc_d)$			
	$C_H = \left(\frac{R_H}{R'_H}\right).100$			
Temporal evolution of heat release rate				
Karede, et al. (2003)	$CI = \sqrt{\left(\frac{Q_{cmáx}}{Q'_{cmáx}}.\frac{t'_{emáx}}{t_{emáx}}\right).100}$			
Pasca, et al. (2010)	$CX = \sqrt[3]{\left(\frac{HR_{máx}}{HR'_{máx}}.\frac{H_{3,5-24}}{H'_{3,5-24}}.\frac{t'_{máx}}{t_{máx}}\right)}$	0 - Species that completely inhibit tricalcium silicate - Not compatible 1 - Pure cement (No inhibition) - Compatible		
Area under the curve				
Fan, et al. (2012)	$C = \frac{A_{wc} - A_o}{A_c - A_o}$			
Setting time		TR > 2		Inhibitor mixtures
Olorunnisola (2008)	$TR = \frac{t_{wc}}{t_{Nc}}$	1,5 < TR ≤ 2,0		Acceptable mixtures
		1 ≤ TR ≤ 1,5		Suitable mixtures

t_{max}: time to reach maximum heat rate of wood-cement mixture (h); t'_{max}: time to reach maximum heat rate of neat cement paste (h); T_m and T_{max}: maximum temperature of the mixture (cement-wood); T_o: ambient temperature; T_M and T'_{max}: maximum temperature of pure cement; S: slope of hydration curve of the mixture (cement-fiber); S': slope of the pure cement hydration curve; R_T: weighted maximum hydration rate of wood-cement; R'_T: weighted maximum temperature rate corresponding to the neat cement mixture; R_H: ratio of the maximum wood-cement heat rate; R'_H: ratio of the maximum neat cement heat rate; Q_{cmax}: maximum rate of the mixture heat evolution (cement-wood-water); Q'_{cmax}: maximum rate of evolution of the pure cement heat; t_{emax}: equivalent time required to achieve Q_{cmax}; t'_{emax}: equivalent time required to achieve Q'_{cmax}; H_{Rmax}: maximum heat rate of wood-cement mixture ($Jh^{-1}g^{-1}$); HR'_{max}: maximum heat rate of neat cement paste ($Jh^{-1}g^{-1}$); $H_{3,5\text{-}24}$: total heat released by wood-cement mixture in 3.5–24 h interval (J); $H'_{3,5\text{-}24}$: total heat released by neat cement paste within 3.5–24 h interval (J); A_{wc}: area under the curve of the fiber-cement mixture; A_C: area under the curve of the pure cement mixture; A_o: area under the curve of the ambient temperature; t_{wc}: setting time of the mixture (cement-fiber); t_{Nc}: setting time of pure cement.

X ray Diffraction (XRD)

The XRD technique was used to analyze the products formed during the hydration of the cement composites TC1, TC3 and TC4, characterized in Table 2, allowing in this way to compare the influence of the vegetal fiber in the process of composite hydration. This procedure allowed that the same amount of each composite to be monitored from the mixing until 48 hours after the beginning of the hydration process. For XRD measures the amounts of materials were reduced, keeping the same proportion of original traces. After mixing, the fresh paste was spread in aluminum mold with the surface dug into a rectangular shape and every 7 minutes the XRD readings were performed until the setting time at a rate of 10°/min and a pitch of 0.01 °. During the whole process, a total of about 60 XRD measurements were performed for each of the analyzed mixtures.

In the first 8 hours after mixing (depending on the composite analyzed), the setting process occurs, in which the paste, which is smooth and plastic, becomes solid and with defined shape and a series of physical and chemical transformations take place. In this work the MiniFlex600 X-ray diffractometer (Rigaku) was used in a range of angles (2θ) varying from 5 ° to 65 °. The conditions applied to the X - ray tube were 40 kV of voltage and 30 mA of current, with source of CuK - Beta radiation, and a nickel filter was also applied. For the

analysis of the data obtained in the test, PDXL2 software was used with mineral database available in the program.

Compressive strength and its relationship to the compatibility

The determination of the compressive strength in composites prepared with coconut fibers followed of NBR 7215 (ABNT, 1996) which addresses the mortar resistance compression. The experimental set included the preparation of 5 specimens cylindrical with dimensions of 25 mm diameter and 50 mm height, for each mixture showed in Table 2.

The samples were kept in mold covered with cloth moist and PVC sheet during first 24 hours. After this period, samples were demoded and cured during 28 days in water with temperature 18 °C. One day before the test, samples were removed from curing water to have your upper and lower surfaces polished. After 28 days all samples were tested on a press of universal trials (AI 700 M – Universal Test Machine), of Gotech under loading speed of 0.75 mm/min.

RESULTS AND DISCUSSION

Hydration test of composites.

In Figure 2, the hydration curves for the composites studied are shown. It is possible to observe a clear difference in the kinetics of hydration -reactions of mixtures due to the presence of fiber, fiber treatment, the addition of silica fume and replacement of water by aqueous extract of the fiber. These differences were used to analyze how existing calculation models respond to these differences and determine their degree of sensitivity to them.

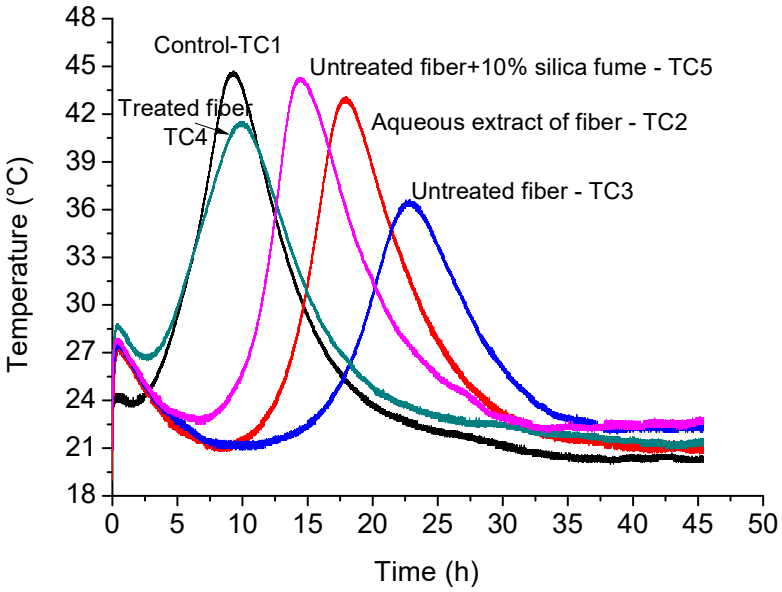

Figure 2 Hydration curves for the five mixtures tested.

In all hydration curves occur a rapid increase in temperature within the first hours, corresponding to the first peak of the curve, the hydration phase C_3A (tricalcium aluminate), followed by a drop (dormancy

period), a period with a low chemical reactivity which varies with the different composites. Hereinafter, the curves show the second and more declined peak of the curve corresponding to hydration of the C_3S (tricalcium silicate) (MEHTA et al., 2008). For TC2 curve (aqueous extract of fiber) there is an inhibition in the mixture hydration process, with a delay of 17 h in comparison to the control trace. The same delay was observed by (Fan, et al. 2012), by using extracts of tropical wood instead of distilled water as hydration solution.

By introducing the green coconut raw fiber in the composite (TC3), there is a delay in the second and highest peak of the curve in 23h, obtaining the lowest value of T_{max} of all trace, 36 °C. Ferraz, et al. (2012), by studying coconut fiber (Cocos nucifera L.), obtained T_{max} for natural fiber (no treatment) of 30,5 °C, with no peak in the hydration curve, using another cement type: CPII Z. This fact points out that regardless of the cement type used, the presence of untreated natural fiber in cement composites greatly affects the behavior of the hydration reactions in the mixture. On the other hand, it is observed that the type of cement used may slightly attenuate this effect. The dormancy period of the TC3 curve is the biggest of all the traces studied; it lasts around 5 h, and only after 12 h of the test beginning, the reactions happen again and the hydration temperature begins to rise.

The TC4 curve (washed fiber) is close to the curve TC1 (control), with no significant differences between t_{max}, with a decrease of T_{max} in 2,7 °C due to the incorporation of the fiber mass to the mixture. This observed decrease is much higher for the TC3 curve (untreated fiber), because besides the embedded fiber mass that absorbs some of the heat, there are organic compounds, such as extractives (chemical effect), that help to reduce the maximum temperature of hydration (Sadiku and Sanusi, 2014). Similar results were obtained by (Nasser and Al-Mefarrej, 2011; Sadiku and Sanusi, 2014), by studying the wood species treated in hot water.

The TC5 curve (raw fibers+ silica) also approaches the TC1 curve, but now there are no significant differences between the T_{max}, but there is a t_{max} delay of approximately 6 h. The addition of pozzolanic materials decreases and, in some cases, eliminates the inhibitory effect of extractives of the lignocellulosic material in the cement setting time, as verified by Del Menezzi, et al. (2007) that replaced part of cement for 10% of silica fume. The explanation given by Vaickelionis and Vaickelioniene, (2006) is that the specific surface of the mineral additives is much higher than the one of the cement, and the adsorption capacity of these materials is higher. Silva, et al. (2017) indicate that the pozzolan adhered to the surface of coconut fibers may provide a local pozzolanic effect in the fiber-cement interface. This pozzolanic reaction consumes portlandite (CH) and generates silicates and hydrated calcium aluminates (CSH and C_4AH_x) which decrease the local pH and increase the resistance and cohesion in the fiber-cement mixture (Gutiérrez, et al. (2005).

X ray Diffraction (XRD)

In Figure 3 two X-ray diffractograms (XRD) are showed for each composite, one before the starting setting time and one slightly after that time. The diffractograms of the composite TC1 show the phase transformations occurring at the time 2 h 35 min. At that time, which coincides with the starting setting time

of the mixture, peaks related to ettringite appear at θ = 30,4° e 30,9°, and there is also evidence of phase changes in existing peaks, compared to time of 2 h 20 min. The formation of ettringite needles, which is one of the cement hydration products and responsible for the setting process, is derived from the hydration of the aluminates (C3A and C4AF) with the gypsum (MEHTA et al., 2008).

The incorporation of 2.5% by weight of coconut fiber without treatment was sufficient to greatly delay the hydration reactions of the composite. For the composite TC3 it was not possible to observe in the XRD patterns the time of the beginning of setting process. There were no phase transformations in time 2h 30 min until time 8h, which demonstrates a slowdown in the process of hydration reactions due to the presence of untreated plant fiber.

Comparing the measurement performed at 2h35min with that carried out at 3h 14min in the diffractograms of the TC4 composite, it is observed that the mixture presented some phase transformations that indicate its setting time value, corroborating the results found in the Vicat assay. In the diffractogram at the time 3 h 14 min it is possible also to notice the formation of ettringite in the angle range of 30.9 ° (Fig 3 Table 4). Thus, the washing treatment of coconut fiber with hot water favored the emergence of the crystalline structure of the cement-fiber composite, giving it a crystalline behavior similar to that of the reference composite (TC1). Similar behavior was found by Pereira et al. (2006) to study three types of lignocellulosic materials of Portuguese origin (cork, eucalyptus and pine) by XRD and DTA techniques. The authors found that all mixes had detrimental effects on the hydration of cement.

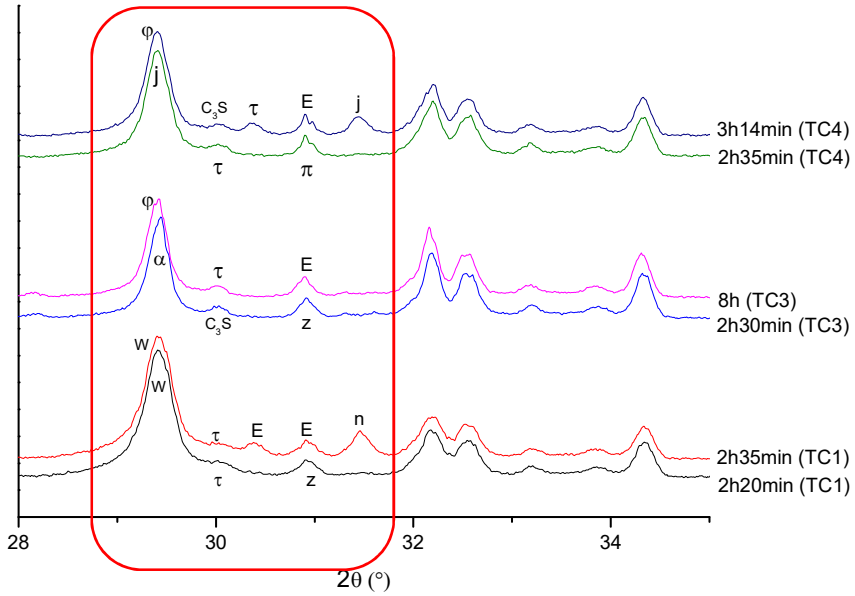

Figure 3 XRD diffractograms of the three analyzed composites.

Table 4: Identification of chemical compounds of diffractograms simbols.

Simbol	**nomenclature**	**Chemical compound**
π	-	$C_3S + C_2S$
τ	-	$C_3S + E$
φ	-	$E + C_3S + C_2S + C_4AF + C$
α	-	$C_3S + C_2S + C + E$
C_3S	Tricyclic Silicate	$2CaO.SiO_2$
E	Etringite	$3CaO.Al_2O_3.3CaSO_4.32H_2O$
j	-	$C_3S + C_2S + C$

n	-	$C+C_2S$
W	-	$C_3S+C_2S+C_4AF+E+C+P$
z	-	$C_2S + E$

Analysis of Compatibility Index by different calculation methods.

The calculations of the Compatibility Index were performed with the methods presented in Table 3. The parameters (Table 5) required to calculate these indexes were determined from the hydration curves and other experimental data obtained in this work.

Table 5: Parameters used in the compatibility indexes calculations.

	Sample				
	TC 1	TC 2	TC 3	TC 4	TC 5
T_m(°C)	-	42,4	36,3	41,3	43,5
$T'_{máx}$ (°C)	44,0	-	-	-	-
T_o (°C)	21,0	21,0	21,0	21,0	21,0
$t_{máx}$ (h)	-	17,28	22,46	10,46	15,00
$t'_{máx}$ (h)	8,71	-	-	-	-
t_{wc} (h)	-	7:54	7:23	2:51	6:22
t_{Nc} (h)	2:44	-	-	-	-
S (°C.h^{-1})	-	2,05	1,11	1,97	2,67
S' (°C.h^{-1})	3:10	-	-	-	-
A_{wc} (°C.h)	-	1222,77	1165,55	1261,25	1748,64
A_C (°C.h)	1163,90	-	-	-	-
A_o (°C.h)	920,12	1016,05	981,883	1041,73	1564,32

It was considered the specific heat of water equal to 1 cal g^{-1} °C^{-1}; of the cement, 0,2 cal g^{-1} °C^{-1}; of the aqueous extract, 0,89 cal g^{-1} °C^{-1}; and of the coconut fiber, equal to 0,34 cal g^{-1} °C^{-1} (RILEM, 1997).
Analysis of indexes variance, with a 95% confidence level, was carried out including the five composites aiming to reveal the existence of significant differences between these traces and to understand how these differences behave. The results showed that, for the indicators I1964, CX, CT and CH, all the traces are statistically different from each other, i.e., the calculation models were sensitive to capture the differences of compositions of each trace. In Table 6 are shown the obtained values in the calculation for the nine studied compatibility indicators.

Table 6: The obtained compatibility indexes.

	Sample				
	TC 1	TC 2	TC 3	TC 4	TC 5
I_{1964} (%)	0	96,95	151,87	22,30	57,70
I_{1984} (%)	0	1,22	19,97	0,47	0,01
TR	1	2,76	2,62	1,04	2,26
C	1	0,87	0,78	0,89	0,74
CX	1	0,73	0,59	0,90	0,82
A (%)	100	91,27	64,15	85,28	90,10
CI (%)	100	74,67	56,79	83,22	85,85
C_T (%)	100	46,34	27,11	74,25	67,58
C_H (%)	100	43,98	25,72	70,44	56,38

Figure 4 shows the results of the inverse indicators regarding to the compatibility. For comparative purposes, the calculated results based on the I_{1964} and on TR factor were normalized. There is a difference of numerical order between the values of I_{1964}, I_{1884} and TR by virtue of own mathematical formulation. Therefore, it was decided to standardize the results (percentage relative to the maximum value obtained) for a better visualization and thus to present a clearer comparison between the methods

It is observed a significant difference in the results of the traces assessed by the first two methods due to differences in their formulations. The fiber-cement compatibility according to method Weatherwax and Tarkow, 1964 is evaluated from the relative differences in the hydration process between the fiber-

cement blend and the standard cement mass. For this, it considers only the temporal shift of the temperature peak in the hydration curves. The method Hofstrand et al. (1984), besides this factor, considers the relative shift of the maximum temperatures and the values of the derivative of the curve. The TC5 trace has a T_{max} of 43,5 °C, very close to T_{max} of the TC1 trace, which was of 44 °C; thereby, the result of this term of the equation contributed to reduce the index to values near to zero.

The results of TR factor did not show the same behavior as the one obtained by the first two models (a greater inhibition for TC2 trace). The model showed less inhibition to TC3 trace, compared to TC2, due to the presence of fiber mass in the mixture that made the setting time to reduce. The qualitative behavior of TR (TC2) > TR (TC3), which inform us that the TC3 mixture is more compatible than the TC2 mixture, is contrary to the results obtained in all other studied methods, including both proportional as inverse compatibility indexes. This indicator is based only on the setting time and is highly influenced by the mixture composition, granulometry of the added fiber, factor A/C and type of the cement. These facts suggest that the use of TR as a compatibility indicator is quite limited, and its use may be indicating to specific practical studies.

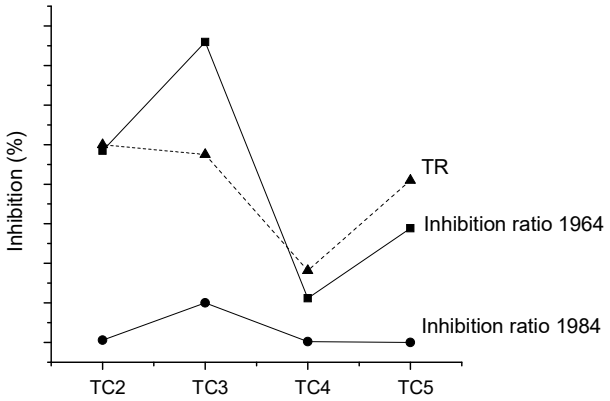

Figure 4: Results of the inverse compatibility indexes: inhibition ratio from 1964, inhibition ratio from 1984 and TR factor.

The index I_{1984}, despite considering three elements of the curve (temperature, time and derivative), shows little sensitivity to the mixture variation. The I_{1964} presents better resolution with the mixture variation, and a similar behavior (for our experiments) to the ones obtained with the proportional methods C, CX, CT and CH. This apparent better resolution of the I_{1964} indicator between the different blends may be a result valid only of the limited set of blends studied and may not necessarily be true in a larger study with different fibers and other blends. On the other hand, the fact that the I_{1964} does not contain thermal parameter information, such as T_{max}, reduces its potential to better describe fiber cement compatibility. Figure 5 illustrates the behavior of proportional compatibility indexes of the four mixtures studied according to the different calculation models. For comparative purposes, the calculated results based on the C e CX index were normalized.

In this figure we find some similarities and several differences in the relative behavior of the values of all compatibility indexes evaluated. It is observed that for all curves the value of the compatibility index decreases from TC2 trace (aqueous extract) to TC3 (raw fiber) where it shows a minimum value valley. From

TC3 to TC4 (washed fiber) all curves show steep growth peaking. However, unlike the rest of the indices for which the minimum value for TC3 is absolute minimum of compatibility, we observe that for index C the value of the lowest index is obtained for the TC5 mixture (raw fiber + silica). On the other hand, when comparing the values of the indices between the mixture TC4 and TC5, it is observed that for most indices (CX, C, CT, CH) the compatibility value is higher for TC4 (washed fiber), only for Aptitude and CI indexes, contrary behavior is observed. Some of the possible causes of obtaining these contradictory results are discussed below.

According to Aptitude index all traces were suitable for use. For the TC4 mixture (washed fiber) was obtained a peak temperature time (t_{max}) and a curve profile very close to the TC1 trace, but with T_{max} slightly lower than TC2 mixture. As the Aptitude is calculated taking into account only the maximum of the temperature, is obtained an Aptitude index for TC4 mixture lower than of the TC2 trace (aqueous extract). In this case, this contradictory result in Aptitude is a consequence of not considering the time of the hydration reaction as one of the elements of the index. Also, parameters such as the maximum temperature (T_{max}) of hydration can be influenced by the thermal absorption effect of the fiber mass incorporated to the composite.

Analysis of the C rate (Figure 5) shows a greater compatibility for TC2 trace than for TC5. However, the introduction of silica fume improves the compatibility of the composites if compared to the substitution of the kneading water by the aqueous extract of coconut fiber (VAICKELIONIS et al., 2006; SILVA et al., 2017). The C index method presented by Fan et al. (2012), which calculate the compatibility index from the area under the hydration curve, can lead to inconsistent results. This fact can be due to some factors, among them, for example, the height of the first peak of the hydration curve of the mixtures with fiber, or aqueous extract, can be greater than that of the control trace. According to Young (1972), it happens due to the presence of the fiber which can accelerate the initial hydration of C_3A before the delaying effect of C_3S starts.

CI compatibility takes into account the thermal capacity of the system, the cooling rate, the temperature variation during the hydration process and the intensity of the reaction. However, some authors Pasca et al. (2010) argue that the accelerating agents (such as silica fume) can change the CI value, since the agents only reduce the cement setting time, and thus, artificially increase the value of this index, which actually occurred in TC5 trace in our experiments. The CX index, took into account not only the thermal parameters of CI rate, but also the maximum heat transfer rate, the time to reach this maximum transfer heat rate and the total heat released during the chemical process, decreasing the artificial effect of high compatibility given by the CI index, and therefore, turning it into the most realistic CX index.

The results of CT and CH indexes (Figure 5) presented the same behavior as the CX rate obtained, despite being simpler formulations, considering only the mass of the components and the thermal capacity of the mixtures. It is noticed that the models that take into account the thermal properties of the system and the time evolution of heat quantity are more sensitive to capture the differences in the compositions of the mixtures, thus presenting more realistic results.

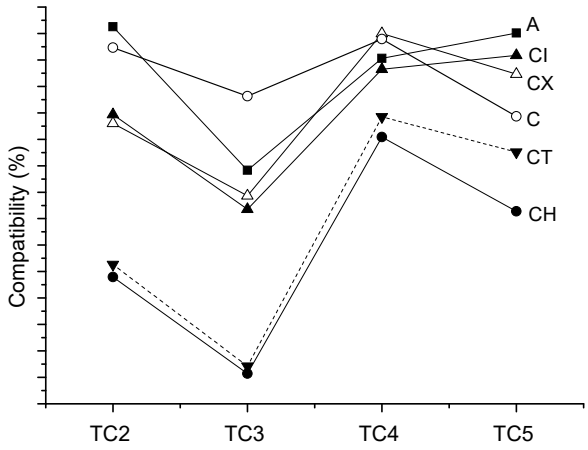

Figure 5: Comparison of the proportional compatibility indexes behavior of the four mixtures studied according to the various calculation models.

The Table 7 shows the compatibility classifications for all mixtures studied according to the various models proposed in literature. Based on results of our experiments set and according to Sanderman and Kohler (1964), which take into account only the T_{max}, the results points to the non-use of green coconut fiber in cement-based on mortars in any of the mixtures and treatments studied. In contrast, the calculation methodology applied by Vilela et al. (1968), whom consider only the relative variation of T_{max}, classifies very positively the use of this fiber. All analyzed mixes were classified as good or very good for application in cement composites. Thus, we find a clear contradiction about the compatibility classification between these authors for the same set of experimental data measured in this work.

Table 7: Compatibility classification of the studied mixtures according to different reported methods.

	Mixtures			
METHOD	TC2	TC3	TC4	TC5
Sanderman e Kohler (1964)	$T_{máx}$=42,4°C<50°C Inadequate	$T_{máx}$=36,3°C<50°C Inadequate	$T_{máx}$=40,8°C<50°C Inadequate	$T_{máx}$=43,5°C<50°C Inadequate
Weatherwax e Tarkow (1964)	15< $t_{máx}$=17,28<20 Intermediately adequate	$t_{máx}$ = 22,46 ≥20 Inadequate	$t_{máx}$ = 10, 46 ≤15 Adequate	$t_{máx}$ = 15 ≤15 Adequate
Vilela e Du Pasquier (1968)	A=93,04≥80 % Very good	80%>A=66,52≥60% Good	A=87,83≥80 % Very good	A=99,57≥80 % Very good
Hofstrand et al. (1984)	I=1,22<10 Low Inhibition	10<I=17,72<50 Moderate inhibition	I=0,47<10 Low Inhibition	I=0,22<10 Low Inhibition
Hachmi et al. (1990)	C_T=46,90%**	C_T=27,41%*	C_T=87,45%****	C_T=72,73%***
	C_H=46,90%**	C_H=26,05%*	C_H=83,12%****	C_H=60,79%***
Karede, *et al.* (2003)	CI=72,88%**	CI=54,34%*	CI=82,82%***	CI=85,84%****
Olorunnisola (2008)	TR=2,88>2 Inhibitor mixture	TR=2,69>2 Inhibitor mixture	1≤TR=1,04≤ 1,5 Adequate mixture	TR=2,32>2 Inhibitor mixture
Pasca *et al*. (2010)	CX=0,77**	CX=0,57*	CX=0,91****	CX=0,87***
Fan *et al.* (2012)	C=0,85***	C=0,75*	C=0,90****	C=0,76**

Minimum compatibility: *; Medium compatibility: **; Great compatibility: ***; Maximum compatibility:****

There are differences of classification for the analyzed traces in relation to the calculation methods used, mainly when considering the utilization of washed coconut fiber and the incorporation of silica fume. For the calculation models CT, CH, TR, CX, C and I_{1964}, the best results (highest compatibility value) were

obtained with green coconut washed fiber (TC4); while the CI methods, Aptitude and I_{1984} indicate better compatibility with the use of in natura fiber plus silica fume (TC5).

The classifications of Hachmi et al. (1990) [6] and Pasca et al. (2010) [12] are in accordance for all tested mixtures. When analyzing the extremes (traces with TC4 washed fibers and TC3 in natura fiber), most of the calculation methods coincide in the classification. The sensitivity of the compatibility indices is an important characteristic for its more precise practical use. Greater sensitivity helps to identify differences in compatibility behavior among composites in order to optimize the reuse of fibers in cement mixtures. The results showed that for the indicators I1964, CX, CT and CH, all the traces are statistically different from each other, i.e. the calculation models were sensitive to reveal the differences of compositions of each trace.

Compressive strength and its relationship to the compatibility

The variance analysis of resistance to compression results shows statistically significant differences between the five composites studied, with 95,0% level of confidence. The trace TC4 (washed fiber) presenting the best result of c*ompressive strength* (7,96 MPa) (Fig.6). The composite TC3 showed the lowest compressive strength of all the blends tested. Therefore, the addition of 2.5% of coconut raw fibers relative to the cement mass decreases the compressive strength 61.8% when compared to the reference paste (trace TC1).

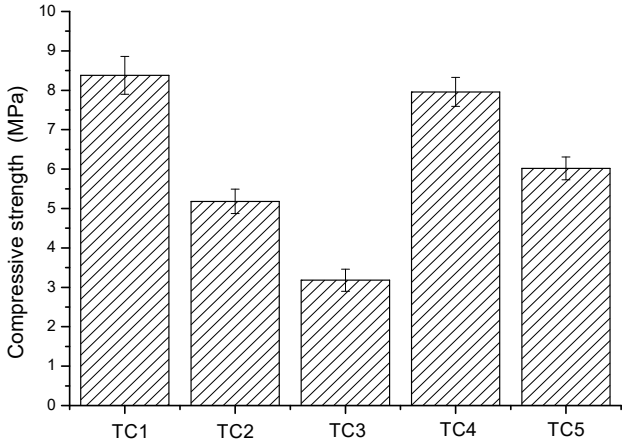

Figure 6: Compressive strength of five analyzed composite.

Bivariate analyzes using the Pearson correlation coefficient were performed to find for the five composites studied, the possible correlations between the mechanical properties of resistance to simple compression and the compatibility evaluated by different calculation methods proposed in the literature.

A greater compatibility in the fiber-cement composite should lead to greater compressive strength (SEDAN et al., 2008; MACÊDO et al., 2011). The compatibility favors the formation of a network of silicates and aluminates that interconnect giving greater resistance to the composite (MEHTA et al., 2008). In this way the indicator that measures the compatibility should show a positive correlation with the resistance.

The correlation found between the compressive strength and the compatibility calculated by different calculation models was positive, which indicates the direct proportionality between them. The highest correlation between resistance and compatibility was significant (95% confidence level) for the CX

(0,987)[1] and CH (0,934)[1] models. The results of various models showed an intermediate level of correlation of the compatibility indices / inhibition) with the compressive strength: C (0,85)[1], CT (0,83)[1] and CI (0,764)[1]. The smallest correlations were obtained between resistance and several indexes: for I_{1964}, TR and the I_{1984} model (0.649)[1] and for Aptitude (0.647)[1]. All correlations were negative for inhibition calculation models, indicating that these parameters are inversely proportional to compatibility properties. Based in our results, from analysis of the models proposed in the literature it can be concluded that the CX index was the compatibility calculation model that best reflects the interaction behavior of vegetable fibers with cement. This formula appears more complete and well as consider the system characteristics (calorimeter), the maximum exothermic effects, and the heat generated during the composite hydration process. CX also showed a higher sensitivity in identifying differences in mixtures and high correlation with the compressive strength of the composites.

CONCLUSIONS

All studied calculation models indicate a lower compatibility to the composite prepared only with untreated coconut fiber and by the majority of the models, the greater compatibility was obtained for composites prepared with treated coconut fiber. The delay in the composite setting time was visualized in the XRD patterns, which allowed to identify the exact time when the phase transformations responsible for gaining strength occurred and to verify the influence of the fiber (and its treatment) in the hydration process of the composite.

From the behavior of the TC2 hydration curve, it is possible to conclude that the presence of organic extractives in solution has an important effect, delaying the hydration reaction. However, the calculation models showed that heat emission rates and the compatibility are less affected. The effect of the use of silica in the composite compatibility leads to similar results to those ones obtained by the fiber washing treatment. Therefore, they are alternatives that can be chosen considering other factors, such as costs, energy and water consumption, in each case. The CX compatibility index was the compatibility calculation model that obtained the highest correlation with the compressive strength. Also, taking into account its high sensitivity and the physicochemical quantities included, we consider it the best index to evaluate fiber-cement compatibility.

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The values in parentheses correspond to the correlation between resistance and compatibility. The closer to 1, the higher is this correlation.

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