Effect of treatments on high initial strength portland cement

and mosso bamboo compatibility

Efecto de los tratamientos sobre la compatibilidad entre el
bambu moso y el cemento portland de alta resistencia inicial

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Abstract

The objective of this study was to evaluate the effect of six different types of treatment on mosso bamboo biomass (Phyllostachys pubescens) in order to verify its compatibility with Portland cement. The treatments adopted for mosso bamboo were: immersion in different solutions as to know: cold water, hot water, sodium hydroxide, sodium silicate, silane and calcium chloride. The degree of compatibility was determined through the study of the hydration heat rates in the first 24 hours and the compressive strength after 7 days of the bamboo-cement composites. The influence of the crystallinity of the fibers was analyzed using X-Ray Diffractometry. Results indicated that the addition of 4% calcium chloride to the blend is the most efficient treatment to decrease the species inhibitory capacity, while treating the raw material with sodium hydroxide adversely affected to the binder. The treatment with cold water presented the highest composite compressive strength and the treatment with silane presented the highest fiber crystallinity index.

Keywords: Vegetable Biomass, composites, compatibility, Mosso bamboo, treatments

Resumen

El objetivo de este estudio fue evaluar el efecto de seis diferentes tipos de tratamiento sobre la biomasa de mosso bambú (Phyllostachys pubescens) con el fin de verificar su compatibilidad con el cemento Portland. Los tratamientos adoptados para mosso bambú fueron: inmersión en diferentes soluciones como saber: agua fría, agua caliente, hidróxido de sodio, silicato de sodio, silano y cloruro de calcio. El grado de compatibilidad se determinó mediante el estudio de las tasas de calor de hidratación en las primeras 24 horas y la resistencia a la compresión después de 7 días de los compuestos de bambú-cemento. La influencia de la cristalinidad de las fibras se analizó mediante difractometría de rayos X. Los resultados indicaron que la adición de cloruro de calcio al 4% a la mezcla es el tratamiento más eficaz para disminuir la capacidad inhibidora de la especie, mientras que el tratamiento de la materia prima con hidróxido sódico se ve afectado adversamente al aglutinante. El tratamiento con agua fría presentó la resistencia compresiva compuesta más alta y el tratamiento con silano presentó el mayor índice de cristalinidad de la fibra.

Palabras clave: Biomasa Vegetal, compuestos, compatibilidad, bambú moso, tratamientos

1. Introduction

Residues produced by timber industry constitute an important source of raw material for the production of construction components, allowing their application as reinforcement for cement paste. This kind of residue has created great interest due to low cost, availability, energy savings as well as for being a solution as far as environmental issues are concerned. The use of prefabricated tiles, panels and other building components can represent a significant contribution to the growth of the infrastructure in developing countries (Savastano Junior and Santos, 2008).

The wood-cement composite is generated by the junction of wood particles or fibers with cement paste. The organic part of the composition, that is, the wood and fibers may also be obtained from non-wood agricultural fibers (such as sisal, bamboo, flax) and other lignocellulosic residues (such as bagasse, wheat, newspaper, among others) (Moslemi, 1997; Cheumani et al., 2011).

The main disadvantage of using these compounds is the inadequate interaction between wood and cement. Wood

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is a lignocellulosic material, and it is susceptible to degradation in a high alkalinity environment, as it is in the cement paste (Moslemi, 1989).

Substances such as hemicelluloses, starches, sugars, phenols, among other extractives, may inhibit the cement hydration process.

According to Biblis and Lo (1968), comprising the organic components of the wood, the carbohydrates are the ones that have the greatest negative influence on the cure of the cement, being glucose and cellulose considered the most inhibitory ones. In the phenolic groups, lignin and terpenes do not present an inhibitory effect, whereas tannins, mainly the ones which are hydrolysate, have a great influence on the inhibition of the cement paste, as well as organic acids of high molecular weight, such as aliphatic acids.

The hydrated cement paste has four main solid phases: hydrated calcium silicate (C-S-H), calcium hydroxide (portlandite), calcium sulfoaluminates and unhydrated clinker grains. The calcium hydroxide crystals (Ca (OH)₂) are responsible for the high alkalinity of the cement matrix, resulting in a pH of 12.5 (Senff et al., 2005).

The components of the wood can be degraded by the calcium hydroxide formed during the cement hydration. This alkaline environment is capable of removing part of the extractives and dissolving components of the wood as the



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hemicelluloses, thus causing dimensional changes in the cement-wood composite (Miller and Moslemi, 1991; Fan et al., 1999).

Wood carbohydrates react with calcium, aluminum and iron cations, retarding the cement hydration reaction, reducing its crystallinity and strenght (Hachmi and Campbell, 1990). The water present in the pores of the cementitious matrix presents high pH, which can break the chemical bonds of the wood fibers, allowing the migration of the wood extractives to the surface of the cement paste (SILVA et al., 2009).

According to Miller and Moslemi (1991), some substances such as resins and fats can migrate to the wood particles surface during the drying process, and they form a hydrophobic layer that reduces the hydrogen bonds between the cement and the wood, which ends up reducing Interfacial strength.

Hachmi and Moslemi (1990), found that the wood extractives are the main responsible for the inhibition of the cement paste, among these there are resins, fat acids, terpenes, terpenoids, simple sugars, salts, phenolic compounds and aliphatic compounds.

According to this compatibility level between cement and wood, this interaction is known as compatible or non-compatible, that is, if the chemical cement hardening process is not disturbed or disturbed to a small extent, it is said that these are compatible. If the presence of the wood affects the hardening of the cement, it is said that these are incompatible (Jorge et al., 2004).

The hydration reaction of the cement is exothermic, that is, there is heat release when it occurs. By measuring the total heat released over time, it is possible to monitor the hydration of the cement, so that this method can be used to analyze the compatibility between the cement and the wood by checking its temperature in the hydration reactions (Hachmi and Moslemi, 1989; Hachmi et al., 1990).

In order to solve the problem of inhibition, chemical additives or accelerators are usually used in diluted solution, adopting pre-treatments for the wood, as aqueous extraction, alkaline extraction, use of blocking agents and binders to decrease the inhibitory content substances. Other strategies are: use of wood preservatives, enhancing prolonged wood storage, wood drying, by employing use of the initial high-strength cement or aluminous cement, CO injection, among others (Moslemi et al., 1983; Simatupang et al., 1988; Pehanich et al., 2004; Na et al., 2014).

These treatments aim at search to eliminate the chemical components that cause cement paste inhibition, and the same time increasing cement hydration to neutralize the hydration retardation effect caused by the presence of wood extractives (Na et al., 2014).

To meet the need for renewable materials, agricultural by-products such as rice hulls, coconut, sisal and bamboo fibers have been used, thus minimizing energy consumption, conserving non-renewable natural resources, reducing pollution and keeping the environment healthy. Bamboo is a material that offers economic advantage as it reaches its full growth in only a few months and its maximum mechanical strength in just a few years. In addition, it exists in abundance in tropical and subtropical regions of the globe (Ghavami, 2005).

Bamboo is characterized as a renewable natural resource, biodegradable, energetically efficient and presents great potential to be used as a building material, however its

applications were limited due to its dimensional instability, inadequate adhesives for its use, difficult connections due to its irregular shape, and fast deterioration due to the anatomical constitution rich in starch (Yushun et al., 2012). Its use in composites has advantages in terms of increasing the properties of strength, elasticity and dimensional stability. In Asian countries, it is widely used for the construction of housing, furniture manufacturing and utensils for daily living, due to its high strength and surface hardness, and flexibility (Hiziroglu et al., 2005; Sumardi et al., 2007; Obataya et al., 2007).

The main focus of this research is to analyze the compatibility between Portland cement and the mosso bamboo biomass (Phyllostachys pubescens), analyzing the effects of the treatments on the compatibility of the bamboo and cement.

2. Materials and Methods

2.1 Materials

Mosso bamboo biomass (Phyllostachys pubescens Mazel ex J.Houz) was employed as raw material. The mossô bamboo particles were donated by a company located in the city of São José dos Pinhais (Paraná, Brazil). After this material is received in the company, it is transformed into particles with the aid of hammer mills, after which, the classification of its granulomentry occurs. The particles maximum size is 1.18 mm according to ASTM C 136/1 (2001b).

The binder used was the high initial strength Portland cement - CPV-ARI, which was chosen due to its initial setting time being faster than the other Portland cements and reach high resistance levels in the first days after the application. In order to analyze the effect of the treatments on the hydration of the CPV-ARI cement, six treatments were used applied to on the mosso bamboo biomass, these treatments were adopted based on the literature. (Castro et al., 2014; Na et al., 2014; Jorge et al., 2004; Pehanich et al., 2004)

- Soaking in the sodium hydroxide (NaOH), sodium silicate and silane, in which the biomass remained immerse in diluted solution at 1%, for a period of one hour, after which the bamboo particles was rinsed in cold water and subsequent dried at room temperature;
- Solubilization in cold water, in which the biomass remained immersed in cold water for 48 hours, after that period it was dried in room temperature (Castro et al., 2014; Pehanich et al., 2004);
- Solubilization in hot water, in which the biomass remained immersed in water at 100° C for 3 hours, after that period it was dried in room temperature, and (Castro et al., 2014; Pehanich et al., 2004);
- Use of calcium chloride based accelerator additive with 4% content, which is added to the composite mixture made with the in natura biomass (JorgE et al., 2004;).

To evalute the heat of hydration of the Portland cement with and without mosso bamboo biomass, according to Hachmi et al. (1990), a calorimeter adaptation was used, as a data acquisition device for WARME Master Logger A 202,



thermocouple type K (nickel - chrome) 8 channel ANSI 2 x 24 (0.51 mm) AWG in silicone.

To analyze the effects of the treatments on the crystallinity of the bamboo mosso fibers, according to Segal et al. (1959). The crystallinity index of the fibers is directly related to the mechanical properties of the composites, so the more crystalline the fiber with the index closer to 100%, the better the performance will be when used as reinforcement in cement-wood panels (Hu and Hsieh, 2001).

2.2 Method

2.2.1 Chemical characterization of mosso bamboo

This biomass of mosso bamboo was chemically characterized employing TAPPI recommendations. Regarding solubility in cold and hot water (TAPPI T 207 cm, 1999), sodium hydroxide (TAPPI T 212 om, 2002a), total extractives (TAPPI T 204 cm, 1997), lignin (TAPPI T 222 om, 2006), ashes (TAPPI T 211 cm, 2002b), moisture (TAPPI T 264 cm, 2008) and pH (TAPPI T 252 om, 2002c).

2.2.2 Compatibility Index

To evaluate the hydration heat of the Portland cement when mixed with the biomass of mosso bamboo, moulds were made using mixes of Portland cement slurry with water: cement (w/c) ratio of 0.4 and cement: and composites of cement wood (w/c) of 1:13 and w/c of 0.4. And for one of the composite mix with in natura bamboo, 4% of calcium chloride was added.

Mixtures were placed inside the calorimeter where the silicone insulation type K2x24 compensation cable was introduced into the mix. The compensation cable was connected to a signal receiver where the data was taken and converted to temperature values by a computer program. The

readings were collected every second for a period of 24 hours. Three replicates were performed for each mixture.

From the hydration curves, it was possible to determine the maximum temperature reached by each mixture, the time in which this temperature was reached and the maximum rate of variation of the temperature over time, thus allowing to determine the compatibility index for mosso bamboo with and without treatment. The results were statistically analyzed using the Tukey multiple means comparison test at 95% level of probability.

For the compatibility index calculation, the equation elaborated by Hofstrand et al. (1984) was employed, which considers the maximum temperatures of the hydration reaction of mixtures that contain or not the bamboo particles, and also takes into account the maximum rates of temperature variation over time, as indicated in Equation [1]:

$$I = \left[(t2-t^2/t^2)(T2-T2/T2)(S'-S/S') \right] * 100$$
 (1)

Where:

t: time to reach the maximum temperature (cementwood-water);

T; maximum hydration temperature (cement-wood-water);

S: maximum rate of temperature variation over time (cement-wood-water);

t': time to reach the maximum temperature (paste);

T': maximum temperature of hydration (paste), e;

S': maximum rate of change of temperature over time (paste).

According to Equation, wood can be classified into four categories, according to Table 1 (Okino et al., 2004; Nasser et al., 2016).

Table 1. Classification of cement hydration inhibition according to the inhibitory index

Inhibitory index (%)	Classification		
I < 10	Low Inhibition		
<i>I</i> = 10 − 50	Moderate inhibition		
<i>I</i> = 50 − 100	High Inhibition		
I > 100	Extreme Inhibition		

2.2.3 Compressive strength

In order to analyze the effects of the treatments on mosso bamboo biomass, selected a cylindrical test specimen with a ratio of 1: 13: 0.4 (cement: bamboo particles: water) for the composites, and reference paste with ratio 1:0.4 (cement: water). Cylindrical test specimens were molded, with internal diameter of 50 mm and height of 100 mm. The

mortar was placed in the mold, divided in four layers of the same height, every layer added was thickened with thirty blows of socket (ASTM C 39, 2001a). After moulding, specimens were cured in a humid chamber (temperature of 23 °C and relative humidity> 95% RH) for 7 days. For compressive strength, a universal tests machine with computer data acquisition was used, the specimen, duly capped with neoprene pieces garnished with steel helmets, was placed on the lower plate of the press, so that it was fully centered to the load axis. After this the compression load to the specimens was transmitted. The compressive strength of the composites was measured quadruplicated and the results were statistically analyzed using Tukey multiple means comparison test at 95% level of probability.

2.2.4 Crystallinity Index

The crystallinity index was obtained by the X-ray diffraction method established by Segal et al. (1959). This is an empirical process that uses adjustment and transmission techniques, measuring the interference intensity in the crystalline plane 002 and the amorphous scattering. The crystallinity index is given by Equation (2).

$$c = \left(\frac{I002 - Iam}{I002}\right) * 100 \tag{2}$$

In which:

Ic: crystallinity index;

lam: intensity of the minimum diffraction, related to the amorphous part $(2\theta = 18.8 \text{ °})$, and;

1002: intensity of the maximum diffraction, related to the crystalline part ($2\theta = 22.4$ °).

For the X-ray diffraction analysis, the sample was sieved and then, the finer grain fraction was subjected to an X-ray beam using Shimadzu XRD-7000 X-ray Diffractometer, using a Cu- $K\alpha$ radiation tube (40 kV, 30 mA), with a diffraction angle (2 θ) ranging from 15° to 25°, as indicated in the literature.

3. Results

3.1 Chemical characterization of mosso bamboo

Table 2 presents the chemical characterization of mosso bamboo biomass.

 Table 2. Chemical characterization of mosso bamboo

Test	Observed value	
Solubility in cold water (%)	2.32	
Solubility in hot water (%)	3.93	
Solubility in NaOH (%)	20.30	
Total extractives (%)	4.27	
Lignin (%)	46.63	
Ashes (%)	2.24	
Moisture (%)	10.93	
pH (2h)	9.0	
pH (24h)	9.3	
*Mean values for three repetitions.		

Based on the results presented, it can be noted that the solubility in NaOH eliminated a higher percentage of biomass substances; this is due to the fact that the sodium hydroxide solution eliminates not only the extractives present



in the wood but also other components such as degraded cellulose and hemicelluloses.

It also shows that this species does not present significant content of total extractives, this being a key factor for the compatibility between the cement and the biomass, hence the higher the extractive content, the greater the incompatibility between the cement and the lignocellulosic material.

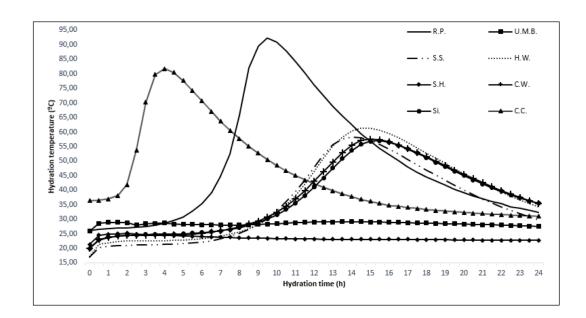
Mosso bamboo presents high ash content (organic matter) in its composition, and this high content can affect the process of hydration of the cement. According to Yu (2007), bamboo presents on average 2-3% of silica in its composition, which reacts with the calcium hydroxide present in the cement matrix, forming an extra amount of hydrated

calcium silicate and, consequently, decreasing the alkalinity of the composite (BIRICIK et al., 1999).

It is also observed that the biomass shows alkaline pH, that is, the nature of its extractives is alkaline. According to Hachmi and Moslemi (1990), alkaline extractives tend to react with the calcium hydroxide present in the cement matrix and form permanent complexes with it, not allowing it to reach the necessary core size for the crystallization of the cement.

3.2 Compatibility Index

The hydration curves of the inhibition tests are shown in Figure 1.



R.P.: Reference paste; S.S.: Sodium silicate; S.H.: Sodium hydroxide; Si.: Silane; U.M.B.: Untreated Mosso bamboo; H.W.: Hot water; C.W.: Cold water; C.C.: 4% calcium chloride.

Figure 1. Hydration curves for the reference paste and mosso bamboo composites

The treatments made with hot water, sodium silicate, cold water and silane were those that were able to raise the maximum cement hydration temperature, when compared with the composite made with untreated bamboo.

Sodium hydroxide affected negatively the cement hydration temperature when compared to the untreated bamboo particles composite. In addition, it can also be seen that the 4% of addition of calcium chloride was the treatment which most approached the hydration temperature to of the reference paste.

Table 3 shows the maximum hydration temperature, the time at which this temperature occurred, the compatibility index as well as its classification in terms of the compatibility with cement.

Table 3. Average temperature values, time and compatibility index for mosso bamboo composites

	Maximum temperature (°C)	Time (h)	I (%)	Inhibitory index
Reference paste	92.33	9.5°	-	-
Untreated Mosso bamboo	29.13°	14.0°	31.77	Low inhibition
Sodium hydroxide	25.13°	2.0	57.65 ^a	High inhibition
Sodium silicate	58.30°	14.0°	9.62	Low inhibition
Cold water	57.33°	15.5°	16.42°	Moderate inhibition
Hot water	61.30°	15.0°	11.65	Moderate inhibition
Silane	57.00°	15.5	17.17°	Moderate inhibition
Addition of 4% calcium chloride	81.40	4.0	5.44∘	Low inhibition

*Mean values for three repetitions.

Based on the compatibility indexes, it was verified that the treatment that significantly increased the compatibility between the CPV-ARI cement and the bamboo particles was the one made with sodium silicate, followed by hot water, cold water and silane. The treatment with sodium hydroxide (NaOH), strongly increased the incompatibility between the cement and the bamboo particles.

For the addition of calcium chloride to the mixture, it was noted that there was a significant improvement in the compatibility index, which almost came close to the maximum hydration temperature of the cement reference paste.

The Tukey test at 95% (α = 0.05) confidence was performed to statistically compare the influence of the treatments on the cement hydration temperature. Thus, it can be stated that the treatments performed applied to in the biomass were a significant factor for the increase of the hydration temperature of the CPV-ARI mixtures.

3.3 Compressive strength

The average values of the compressive strength of the composites and the reference paste are shown in Table 4.

Table 4. Compressive strength after seven days Mean values

	Ratio (cement: bamboo particles: water)	Compressive strength (MPa)
Reference paste	1: 0: 0.4	44.66
Untreated mosso bamboo composite	1: 13: 0.4	11.65°
Sodium hydroxide	1: 13: 0.4	13.50≈
Sodium silicate	1: 13: 0.4	17.18=
Hot water	1: 13: 0.4	16.28⊭
Cold water	1: 13: 0.4	19.49
Silane	1: 13: 0.4	<i>17.55</i> ⊭
Untreated mosso bamboo composite and addition of 3% calcium chloride	1: 13: 0.4	18.49

*Mean values for four repetitions.



^{**}Means followed by the same letter do not differ from each other, according to the Tukey test at 95% level of probability

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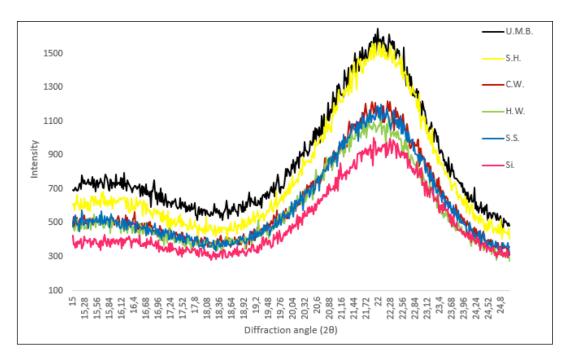
Regarding the treatments adopted, it was noted that all increased the compressive strength of the composites when compared to those the composite made with untreated bamboo.

The Tukey test at 95% (α = 0.05) confidence was performed to statistically compare the influence of the treatments on the compressive strength. It can be stated that the bamboo particles treatments were determinant for the increase in the

compressive strength of the composites. The treatment with cold water increased the compressive strength of the composites the most.

3.4 Crystallinity index

The X-ray diffractometer for the different treatments performed on the bamboo fibers is presented in Figure 2.



U.M.B.: Untreated Mosso bamboo; S.H.: Sodium hydroxide; C.W.: Cold water; H.W.: Hot water; S.S.: Sodium silicate; Si.: Silane.

Figure 2. X-ray diffractometer for the different pretreatments applied to of mosso bamboo fiber

Table 5 presents the values of the crystallinity index for

the different treatments carried out on mosso bamboo particles.

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	Crystallinity index (%)
Untreated Mosso bamboo	58.63
Sodium hydroxide	65.59
Sodium silicate	64.02
Hot water	63.32
Cold water	64.80
Silane	68.13

All of the treatments carried out on mosso bamboo particles increased the fiber crystallinity. Among the treatments, those with silane allowed obtained the highest crystallinity index, increasing it by 16%. This occurred because the silane acts as a coupling agent in the fiber, modifying its interface, making it more crystalline.

The fiber crystallinity index can be related to the compressive strength of the composite made with silane treated bamboo particles, since it presents the third higher compressive strength.

On the other hand, the treatment with hot water was the one that had the least influence on the crystallinity index, increasing it by 8%. In addition, it is also possible to verify a correlation between the compressive strength of the composites from bamboo treated with hot water and the index of crystallinity, as these reached the fifth lower compressive strength rate.

4. Discussion

As reported, the chemical composition of the biomass disturbs the hydration process of the cement. However, bamboo particles does not have a high extractive content, and presents a high amount of ash in its composition.

According to for the inhibition test, it can be observed that the treatment with sodium hydroxide negatively affected the hydration temperature, and the compatibility index of the composite, decreasing the compatibility by 18% when compared to the composite reference. This is due to the fact that the sodium hydroxide solution eliminates not only the wood extractives but also other components such as cellulose and hemicelluloses, thus influencing the hydration of the cement. It is also noted that this treatment delayed the setting time of the cement when compared to the composite made with untreated bamboo particles.

The treatment with silane showed an increase of the compatibility of 86% between the cement and the bamboo particles, acting as a binding agent between the cement and the biomass, thus increasing the cement heat of hydration. Regarding to treatment with sodium silicate, this acts as a blocking agent over the bamboo particles.

The pre-treatment carried out with cold water removes organic components, tannins, gums and sugars, and the pre-treatment with hot water, in addition to these components, eliminates starch, all of which affect the hydration process of the cement. Thus, the treatment with hot water becomes more efficient to increase the compatibility between the mosso bamboo particles and Portland cement, as this eliminates the starch present in the mosso bamboo particles. The treatments with hot and cold water, when compared to the composite with untreated bamboo particles, proved helpful to increase the compatibility, in 63% and 48%, respectively.

For the composites with the addition of 4% calcium chloride, this increases the compatibility between the vegetal bamboo particles and cement. This occurs due to the fact that the calcium chloride acts as a catalyst for the mixture, accelerating the reactions hydration of the cement and

preventing delay in the hydration process the biomass to affect this process.

The treatment with sodium silicate equaled the time of hydration of the reference paste, when compared to the composite with untreated bamboo particles. For the treatments with cold water and silane these increased the setting by 10% and hot water increased its by 7% when compared to the composite with untreated bamboo particles. For all of the treatments, compressive strength was enhanced when compared to the untreated bamboo particles: sodium silicate increased it by 83%, hot water by 74%, cold water by 108% and silane by 87%. Even the treatment with sodium hydroxide which did not achieve satisfactory results regarding compatibility index increased compressive strength by 44%.

It is noticeable the influence of the silica present in the bamboo chemical composition, since it reacts with the calcium hydroxide present in the cement matrix, forming an extra amount of hydrated calcium silicate, which is the main one responsible for the increasing the composite's compressive strength.

For all of the treatments applied to the bamboo particles, crystallinity index increased. Pre-treatments applied to the bamboo particles were efficient for the removal its amorphous extractives, increasing its crystallinity. The pre-treatment with silane was the most efficient in 68.13.

5. Conclusions

The mosso bamboo particles can be employed in cement-wood production, as the cold water treatment used increased the axial compression strength by 108% when compared to the reference ones.

The treatment with 1% of sodium silicate and hot water were satisfactory regarding to the compatibility index between bamboo particles and cement, increasing this index by 69% and 63%, respectively.

The addition of 4% of calcium chloride in the mixture allowed a significant improvement in cement and bamboo particles compatibility, increasing compatibility index by 82%, and raising the composite hydration temperature by 180% when compared to the reference. We also verified that the increase of compatibility positively influenced on the mechanical properties of this composite, which is a positive and desirable feature for construction uses.

The treatments applied to mosso bamboo particles increased the compressive strength of the composites. The hydration temperature of the CPV-ARI paste and the fiber crystallinity index were also enhanced compared to the reference composite made with untreated bamboo.

Thus, it can be concluded that mosso bamboo particles presents suitable characteristics for its use in cement-wood composites. All of the pre-treatments applied to the bamboo particles its enhancing the compatibility, compressive strength and crystallinity of composites.

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