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INFLUENCE OF THE USE OF SILICATE-BASED SEALER ON THE PHYSICAL-MECHANICAL PROPERTIES OF WOOD BIO-CONCRETE UNDER NATURAL AGING

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ABSTRACT

The high emissions of carbon dioxide and the concern with environmental issues have led to greater use of lignocellulosic biomass in civil construction, especially wood. The incorporation of wood bio-aggregates in cementitious composites allows the development of an energy efficient material, having its thermal insulating and carbon sequestration capacity improved. However, the durability of this product is barely known and can be harmed by the high volume of organic matter in its composition, requiring surface protection. Therefore, the objective of this work is to evaluate the influence of the use of a sealer based on lithium silicate and sodium silicate on the physical and mechanical properties of wood bio-concrete. The mixtures were produced with a volumetric fraction of wood shavings of 45%, water/binder ratios of 0.35 and 0.30, and the cement matrix was prepared with a combination of cement, metakaolin, and fly ash. Capillary water absorption, uniaxial compression tests, and scanning electron microscopy (SEM) were carried out on the bio-concretes, with and without sealer protection, under dry curing conditions, and after 3 months of natural aging. In addition, the incorporated air content of the mixture in a fresh state was determined. The results obtained showed that the use of the sealer was not efficient for the samples produced with a water/binder ratio of 0.35 and slightly efficient for the ones produced with a water/binder ratio of 0.30. For both water/binder ratios, mechanical and physical properties decreased after natural aging.

KEYWORDS:

Wood bio-concrete; sealer; physical mechanical properties; natural aging.

INTRODUCTION

High energy consumption is one of the main contributing factors to high levels of carbon dioxide emissions in the world (Al Abdallah et al., 2022). This concern has led to the development of new insulation materials in construction to reduce energy loss and conserve temperature within buildings. For reducing the consumption of natural aggregate and achieving sustainable development of concrete, various wastes are applied for the production of eco-friendly concrete, such as wood, oil palm shell, coconut shell, etc (Wu et al., 2019).

Wood waste has been used as a valuable component to produce bio-concrete with lower environmental impact (Bourzik et al., 2022; Da Gloria and Toledo Filho, 2021; Corinaldesi et al., 2016). However, the durability of this material is not well known. The deterioration of concrete is a major problem as it may threaten the stability of this material. The pore structure of concrete is a key parameter that determines the permeability and transport properties, which affect mechanical properties and chemical durability by changing the content and transmission of moisture (Xiong et al., 2022).

To extend the durability of concrete structures various kinds of surface treatment can be embraced. Some of these surface treatments can penetrate the concrete pores and react with the hydration products of concrete, reducing the surface porosity and increasing the superficial strength (Baltazar et al., 2014). These treatments are known as silicate-based impregnation.

The mechanisms through which sodium silicate-based concrete sealers improve concrete performance are still uncertain (Fronzoni et al., 2013). However, it is commonly accepted that sodium silicate-based concrete sealers permeate into concrete and the active sodium silicate reacts with portlandite in the cement matrix to produce calcium-silicate hydrates (C-S-H gels), which work blocking the concrete pores, enhancing its surface hardness and durability while reducing its structural permeability (Baltazar et al., 2014).

Lithium silicate is a kind of water glass that is often used for the surface treatment of cement-based materials (Tang et al., 2019). It can react with calcium hydroxide (CH) to form new calcium silicates hydrates (C-S-H), which results in the densification of the hydration products to improve strength and hardness (Song et al., 2021; Deng et al., 2014).

Encouraging results for the protection of concrete surfaces have been recently obtained from sodium and lithium silicate treatment (Song et al., 2021; Jiang et al., 2015; Baltazar et at., 2014). Nevertheless, no study was found impregnating silicate-based sealer in wood-cement composites. For that reason, in this work, the main purpose is to explore the action mechanism and effectiveness of silicate-based sealer (SBS) application on wood bio-concrete (WBC). On the fresh state, a flow table test and incorporated air content were performed to evaluate the workability and estimate the percentage of pores on WBC. Capillary water absorption and compressive strength were employed to analyse the changes in the pores structure, capillary channels, and surface hardness of the WBC caused by SBS. SEM was used to observe the microscopic morphology of the new external surface generated with SBS in WBC samples.

EXPERIMENTAL SECTION

Materials

The Brazilian Portland Cement type CPV-ARI (high initial strength) supplied by Holcim company was used as a binder. As supplementary cementitious materials, metakaolin provided by Metacaulim do Brasil company and fly ash supplied by Pozofly company was used. The specific density of the cement, metakaolin and fly ash were 3.01 g/cm^3, 2.64 g/cm^{3,} and 1.89 g/cm^3, respectively.

As bio-aggregate, wood waste obtained in shavings form from the state of Rio de Janeiro (Brazil) was used. The wood bio-aggregates were a mix of four different species: *Hymenolobium petraeum*, *Cedrela fissilis*, *Erisma uncinatum warm,* and *Manilkara salzmanni*. About particle size, Da Gloria and Toledo Filho (2021) suggest using only the fraction of nominal diameter higher than 1.18 mm due to the high surface area and great potential to absorb water from the smaller particles. The material was treated in calcium hydroxide solution ($Ca(OH)_2$), with concentration of 1.85 grams per kg of water for 2 hours, to improve the mechanical compatibility between the bio-aggregate and cement paste, followed by air-drying for 48 h. The bulk density, water absorption, and moisture content of the wood shavings were 530 kg/m^3, 70%, and 22%, respectively.

The concrete sealer based on lithium silicate and sodium silicate, supplied by MGPAR (São Paulo/Brazil), is composed of an aqueous dispersion of an acrylic copolymer, lithium metasilicate and colloidal silica. Its density is 1.10 g/cm^3 (at 25 °C) and pH is 9-10.

Mixture proportions

The bio-concrete mixes were prepared with a volumetric fraction of 45% of wood shavings (WS). The cement matrix was composed of 45% of Portland Cement (PC), 25% of metakaolin (MK), and 30% of fly ash (FA), which were defined based on the work of Andreola et al. (2019) to reduce cement consumption and improve the mechanical strength of the bio-concretes. Two water-to-binder ratios, 0.30 and 0.35, were used to produce bio-concrete with different densities. Calcium chloride (CC) was used as a setting accelerator at a content of 2% in relation to the mass of cementitious materials. Table 1 shows the consumption of materials, in kg/m^3. The bio-aggregates absorb water during the mixing process, which must be considered to guarantee good workability of the mixtures. Therefore, the total water (Wt) is the sum of the cement hydration water (Wh) and the compensation water (Wc), absorbed by the bio-aggregates.

Table 1 – Mix proportions of bio-concrete mixtures (kg/m³).

WBC	WS	PC	MK	FA	CC	Wh	Wc
WBC45-0.30	238.5	352.1	195.6	234.7	15.6	234.7	167.0
WBC45-0.35	238.5	328.7	182.6	219.1	14.6	255.7	167.0

Preparation of specimens

All bio-concrete blends were mixed in the laboratory using a 20-liter mixer. The production process began with a mixing of the cementitious materials and WS during 1 minute. After that, the total water, previously mixed with CC, was added progressively over 1 minute. The total mixing time was 4 minutes to obtain a homogeneous bio-concrete. The specimens were molded in three layers, mechanically vibrated (68 Hz) for 10 seconds each, in cylindrical molds of 50 x 100 mm (diameter x height). The samples were demolded after 24 hours and kept in a room at temperature of 20 ± 3 °C and relative humidity of 55 ± 5% until reaching 28 days of old.

After the curing time, the samples were impregnated with the silicate-based sealer solution. As recommended by Song et al. (2021), the samples were soaked in a 9% concentration solution for 6 h. After that they were dried to constant weight in an oven (T = 60 °C).

Natural aging

The samples were placed, for exposure to natural aging, on the roof of the laboratory located in the city of Rio de Janeiro/Brazil. The weathering period was from 18 April 2022 to 18 July 2022 and temperature and humidity data were collected by a meteorological station installed on site (Figure 1).

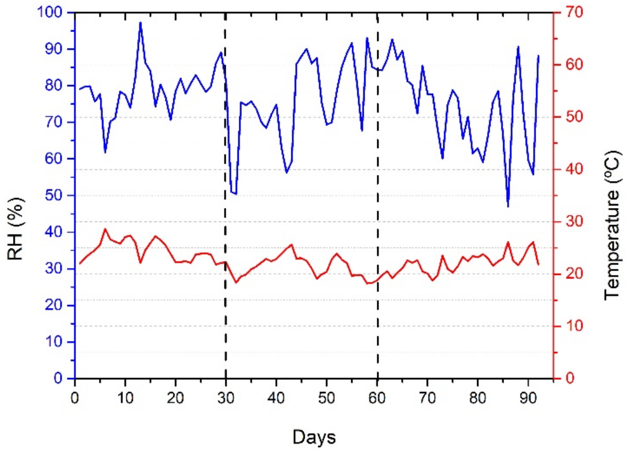


Figure 1 – Relative Humidity and Temperature data collected during the natural aging

From Figure 1 it is possible to observe how temperature and humidity behaves during the three months of study. In the first 30 days there was a temperature variation from 23˚C to 30 ˚C and RH from 61% to 97%. In the transition from the first month to the second there was a RH variation from 89% to 50% and then to 75% in 3 days. A wide variation of RH happened twice more during the second month. The third month followed a trend of decreasing RH, but at the end of the month, there were variations from 79% to 46% to 90% and 55% with temperatures varying from 23 ˚C to 26 ˚C to 21 ˚C to 26 ˚C, respectively. This behaviour may explain a possible drop in the behavior and physical properties of the material.

Flow table and incorporated air content

At the fresh state of the bio-concretes, the flow table and incorporated air content tests were performed according to Brazilian standards NBR 13276 (2016) and adaptions of the NBR 16887 (2020), respectively.

Capillary water absorption

The capillary water absorption of the bio-concrete was measured following the method proposed by RILEM, protocol in progress (TCHDB 275). The samples were dried at 60 °C for 72 h. To ensure one-dimensional water transport, except for the top and bottom surfaces, all the surfaces were sealed with metallic tape. Then, the bottom of the specimens was immersed in water at a depth of 5 mm for 1, 3, 5, 10, 15, 30 minutes, 1, 2, 3, 4, 5, 6, 24, and 48 hours. The water absorbed by capillary suction was measured by weighting the samples after different durations of immersion.

Compressive strength

The compressive strength of the bio-concretes studied were measured using a Whykehan Ferrance 250 KN at a test velocity of 0.3 mm/min. Two LVDTs (Linear Variable Differential Transformer) were used to monitor the longitudinal deformations of the samples, and the Young Modulus (E) was determined according to Brazilian standard NBR 5739 (2018).

SEM analysis

To study the effects of the silicate-based concrete sealer on the microstructure of the bio-concrete specimens, the surface microstructure of the WBC45-0.30 samples (the reference sample and the sample impregnated with silicate-based concrete sealer) were examined by a scanning electron microscope (SEM) using a Secondary Electron (SE) model *Hitachi TM 3000* from the transcoding of the energy emitted by electrons.

RESULTS AND DISCUSSION

Flow table and incorporated air content

The spreading of each mixture is shown in Figure 2. The consistency index of WBC45-0.35 is 22.5 cm and WBC45-0.30 is 19.5 cm. This decrease was expected since the amount of water in the mixture was reduced.

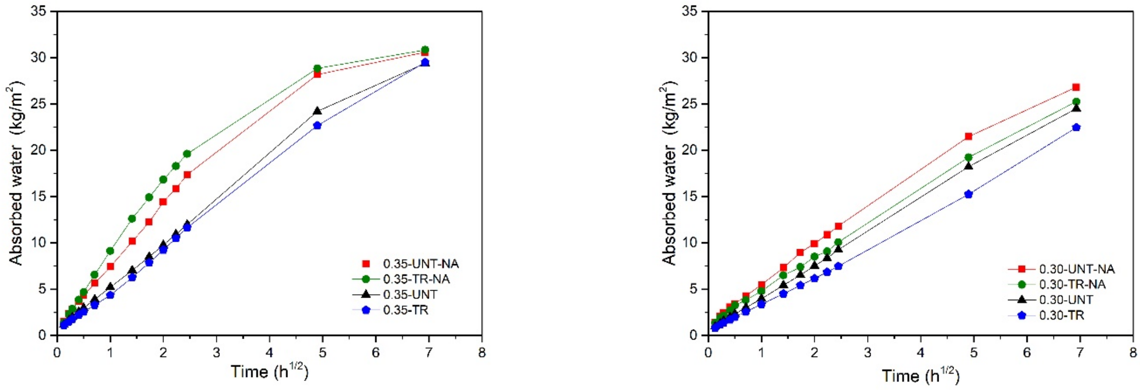
The incorporated air content results are also related to the amount of water. For the WBC45-0.35 its value was 13.8% and for the WBC45-0.30 it was 9.5%. This can be explained because the wood bio-aggregate used contain sapolic extractives, not soluble in water, that caused the release of more bubbles and changed this property of the fresh bio-concrete (Da Gloria, 2020).

(a) 22.5 cm (b) 19.5 cm

Figure 2 – Flow table test on mixtures of (a) WBC45-0.35 and (b) WBC45-0.30.

Capillary water absorption

Figure 3 shows the results of the water absorption test for the (a) WBC45-0.35 and (b) WBC45-0.30. The specimens were identified as follows: 0.35 and 0.30 for the w/b ratio, UNT and TR for untreated and treated samples, and NA for those that had been in natural aging for 3 months.



(a) (b)

Figure 3 – Water absorption test on untreated and treated samples of (a) WBC45-0.35 and (b) WBC45-0.30.

The results show a greater water absorption of WBC45-0.35 in relation to WBC45-0.30, due to its higher pores, consequence of its higher incorporated air in the fresh state. After 48 hours of test, WBC45-0.35 absorbed about 20% more water than WBC45-0.30. Analysing the effect of the natural aging on the water absorption, the aging was more harmful to WBC45-0.35, because of their higher porosity, consequence of the high amount of incorporated air.. At 6 h of the test, 0.35-UNT absorbed approximately 12 kg/m^2, while for the same time 0.35-UNT-NA absorbed 17 kg/m^2. For the WBC45-0.30 samples, at 6 h of test 0.30-UNT absorbed 9 kg/m^2 and 0.30-UNT-NA absorbed 12 kg/m^2. Once again, the highest number of pores in the WBC45-0.35 is the reason for their greater degradation with natural aging.

Evaluating the effectiveness of treatment on bio-concrete, it is not efficient for WBC45-0.35 and slightly efficient for WBC45-0.30. Sodium silicate-based concrete sealers are basically surface hydrophilic agents, yet they reduce the velocity of water ingress into the concrete structures because the expansive and insoluble C-S-H gels partially fill micro-pores, micro-voids, and micro-cracks in the concrete structure to form smaller ones and improve the compactness and water impermeability of the concrete (Song et al., 2016). For the studied bio-concretes, in addition to the micro-pores, micro-voids, and micro-cracks, there are larger pores barely filled by the C-S-H gels produced in the treatment. Figure 3.b shows that from 0.30-UNT to 0.30-TR there is a reduction of 8% in water absorption and from 0.30-UNT-NA to 0.30-TR-NA the reduction is 6%.

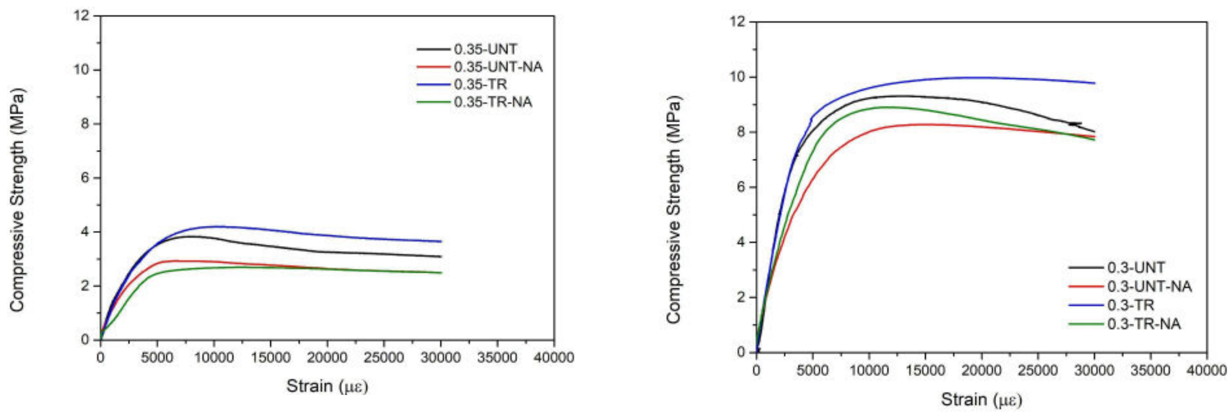
Compressive strength

Analysing the results of Table 2 and the curves of Figure 4, it is noted that the WBC45-0.35 and WBC45-0.30 present a linear behavior up to approximately 50% and 65%, respectively, of their maximum strength, followed by a non-linear section up to reach the maximum compressive strength peak. The post-peak behavior exhibits a gradual reduction in stress and an increase in strain. All samples referring to WBC45-0.30, regardless of treatment and natural aging, showed a higher compressive strength, around twice as much, as the WBC-0.35 mixtures. The WBC-0.30 has important characteristics that were highlighted in the previous items that explain this behavior, such as it absorbs less water, the incorporated air content is lower and the SBS was more efficient in this mixture.

Table 2 – Compressive strength and Young modulus (variation coefficients in % in brackets).

WBC	Compressive strength (MPa)	Young Modulus (GPa)
0.35-UNT	4.08 (10.28)	0.9 (8.33)
0.35-UNT-NA	3.32 (5.45)	0.7 (3.97)
0.35-TR	4.25 (3.29)	1.0 (7.44)
0.35-TR-NA	3.15 (7.36)	0.6 (10.87)
0.30-UNT	9.85 (12.42)	2.5 (12.30)
0.30-UNT-NA	8.59 (1.18)	1.6 (0.47)
0.30-TR	9.97 (3.62)	2.3 (11.38)
0.30-TR-NA	9.25 (4.63)	1.7 (3.11)

For 0.35-UNT-NA and 0.35-TR-NA samples, a reduction of approximately 1 MPa in strength was observed comparing to 0.35-UNT and 0.35-TR samples, indicating a degradation in samples that were exposed to natural aging. In WBC45-0.30 samples this behavior was not observed. However, the young modulus of 0.30-UNT-NA and 0.30-TR-NA samples were significantly lower than that of samples that were not exposed to natural aging.

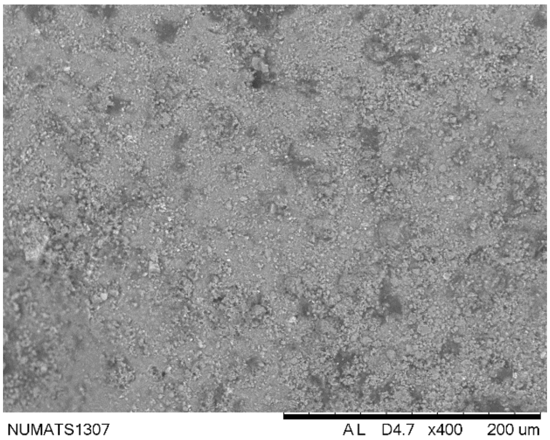


(a) (b)

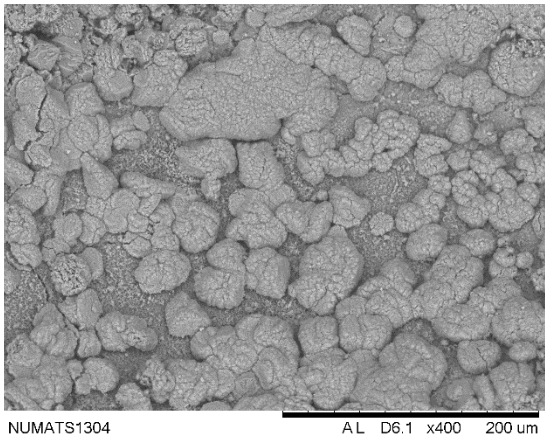
Figure 4 – Compressive strength test on untreated and treated samples of (a) WBC45-0.35 and (b) WBC45-0.30.

SEM analysis

As SBS proved to be more efficient for WBC45-0.30, SEM images of external surfaces of the untreated and treated samples can be observed in Figure 5. In Figure 5.b the spongy products formed due to the treatment can be associated with the production of C-S-H, which means that CH already reacts with SBS. It is also possible to observe that the sample's micropore wall is almost covered by these products. The results agree with the water absorption and compressive strength results.



(a)



(b)

Figure 5 – SEM images of (a) untreated and (b) treated samples of WBC45-0.30 (external surface analysis).

EDS mounted on SEM was used to perform point analysis of the chemical compositions of the untreated and treated WBC45-0.30. Table 3 indicates that oxygen, silicon, calcium, and carbon are the main elements. The content of silicon and calcium increased from 0.30-UNT to 0.30-TR, proving that the content of C-S-H in 0.30-TR is higher.

Table 3 – Chemical element analysis of the untreated and treated WBC45-0.30.

Chemical element		O	Si	Ca	C	Al
Atomic percent ratio (%)	0.30 - UNT	62.07	1.28	13.13	20.53	1.28
	0.30 - TR	61.1	1.62	16.68	20.59	0

CONCLUSION

The action mechanism and effectiveness of silicate-based concrete sealer in wood bio-concrete were systematically explored. From the experimental results it is possible to draw the following conclusions:

- For water absorption SBS it is not efficient for WBC45-0.35 and slightly efficient for WBC45-0.30 because, in addition to the micro-pores, micro-voids, and micro-cracks, there are larger pores on WBC samples;
- The application of SBS did not contribute to change WBC mechanical behavior. It was observed that the greatest difference in compressive strength is related to the water/cement content in the mixtures.
- Natural aging caused an increase of the water absorption capacity and decrease of compressive strength for all samples studied;
- SEM images show the formation of products on the external surface of samples due to the treatment that can be associated with the production of C-S-H.

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