Effect of Silane on Physical and Mechanical Properties of Wood Bio-Concrete Exposed to Wetting/Drying Cycles

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Abstract. Wood bio-concrete is a promising bio-based building material due to its insulating properties and its contribution to increase energy efficiency and reduce environmental impacts. However, there is still a lack of knowledge concerning its durability over time. For that reason, this study investigates the influence of accelerated aging in laboratory on the properties of wood bio-concretes through a succession of wetting/drying cycles. In order to improve the durability, silane-based water repellent was used as a surface skin protection (SSP) of the bio-concretes and as an impregnation agent of bio-aggregates (IBA). The bio-concretes were produced with a volumetric fraction of 45% of wood shavings and a cementitious matrix composed by cement, rice husk ash and fly ash. The physical and mechanical properties of the samples before and after the wetting/drying cycles were evaluated. After accelerated aging, a decrease of compressive strength was observed for the control (without silane addition) and IBA samples. It was found that SSP was more effective in improving the capillarity water absorption resistance of wood bio-concrete in the early hours only, while for IBA samples, there was a decrease of 17% comparing to control samples at the end of testing. Analysis performed by scanning electron microscopy showed cracked transition zones of the bio-aggregate and cementitious matrix of bio-concrete, due to aging process.

Keywords: Wood bio-concrete · silane · durability · physical and mechanical properties

1 Introduction

The high environmental impact caused by the building sector has led to increased research on green or alternative materials such as bio-based construction materials using local vegetable resources as lightweight aggregates in combination with inorganic binders [1]. Named as bio-concrete, this material uses plant origin particles as aggregates, and for this reason is capable of sequestering CO_2 through photosynthesis during the plant growth [2] and has interesting thermal insulation properties combined with good hygric regulation performances [3].

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Wood waste represents, nowadays, an important economic and environmental issue, and can be either used as energy recovery or reused as a building material [4]. Several authors have used wood as bio-aggregate to produce eco-friendly bio-concrete [4–6]. However, the durability of the bio-concrete is not yet itself sufficiently exploited and has been target of concern. Since the knowledge about the evolution of its long-term performance is scarce, it can limit the use of wood bio-concrete in the construction field.

Durability of a material depends on the conditions of its use and the environment of exposure [7]. Wood is a heterogeneous and anisotropic material, whose porosity is responsible for dimensional variations when exposed to outside climatic conditions, which consequently affects the physical and mechanical properties of the bio-concrete produced, despite of controlling thermal and acoustic dissipation phenomena. In addition, aging can modify the material microstructure with the hydration of the binder over time and generate dissolution/precipitation of different compounds [8]. Relative humidity, temperature, liquid water, and UV radiation cause changes in chemical and microstructural properties, modifying the functional properties of bio-based materials [7]. Water is a critical issue, since wood shavings are hydrophilic, and the presence of water leads to a swelling of the bio-aggregate which increases the stresses at the interface with the binder [9].

In this context, a hydrophobic product based on silane has been used as a protective agent for porous cement-based materials. Among commercial organic surface treatment materials, silane-based products, and especially the mixture of silane and siloxane, could be considered as favorable water repellents with good breathability [10]. Silanes belong to the pore liner category and are a group of silicones containing one silicon atom [11]. For surface impregnations, alkoxy and alkyl silanes are commonly used [12]. The alkoxy groups (CH_3O) attached to the silicate atom (Si) contain silicon-oxygen bonds that will link to silicates present in the concrete, while the organic alkylic (CH_3) group remaining will protrude from the pore structure and are responsible for the hydrophobic characteristics [13].

Silane can be used to coat exposed surface of concrete in order to protect it from attack by foreign agents. This type of treatment can suppress significantly capillary water absorption and therefore improve the durability of concrete [14]. Furthermore, silane can be used to impregnate bio-aggregates and create a hydrophobic surface capable of reducing the water absorption and improving durability issues. Alkoxy groups in silane can be hydrolyzed by reacting with water to form more reactive silanol groups, which can also attract each other and form -Si-O-Si- bonds, generating a siloxane network in the solution. Moreover, the silanol molecules attach to the surface of fibers and form hydrogen bonds with the hydroxyl groups of the cellulose in the step of adsorption, creating a monolayer of polysiloxane on the surface of the fibers and resulting in a coating characteristic [15]. However, the performance of silane in both cases depends on surface preparation, amount of silane used, time of curing or impregnation, cyclic wetting and drying and environmental conditions at the time of application.

The objective of this study was to evaluate the influence of silane-based water repellent on the durability of wood bio-concrete by exposing it to wetting and drying cycles. Two silane application methods were adopted: 1) a surface skin protection of the bio-concrete, and 2) impregnation of bio-aggregates before bio-concrete casting.

Their influence on some physical and mechanical properties of wood bio-concrete was investigated.

2 Materials and Methods

2.1 Materials

Wood Shavings

Wood waste obtained in shavings form from the city of Rio de Janeiro (Brazil) was used as bio-aggregates. The wood shavings were a mix of unknown fractions of four species: *Hymenolobium petraeum*, *Cedrela fissilis*, *Erisma uncinatum warm*, and *Manilkara salzmanni*. Wood particles were separated using a mechanical sieve and only the fraction retained in the sieve of opening 1.18 mm was used due to the high water absorption potential of the smaller particles, as suggest by Da Gloria and Toledo Filho [6]. In order to improve the chemical compatibility between the bio-aggregate and mineral binders and based on the work of Aguiar et al. [16], wood shavings were treated by immersion in calcium hydroxide solution ($Ca(OH_2)$) with concentration of 1.85 g per kg of water for 2 h. The main characteristics of the treated wood are presented in Table 1: apparent bulk density [17], water absorption, following the method proposed by Da Gloria and Toledo Filho [6], moisture content [18], and chemical composition [19].

Table 1. Physical parameters and chemical composition of wood shavings.

Physical parameters		Chemical composition (%)	
Apparent bulk density ($kg.m^{-3}$)	530	Cellulose	44.28
Water absorption (%)	70	Hemicellulose	26.43
Moisture content (%)	19	Lignin	26.84
		Extractive compounds	2.45

Supplementary Cementitious Materials

The Brazilian Portland Cement type CP II-F-40 (compound with filler) supplied by Holcim company was used as a binder. Rice husk ash provided by Silcca Nobre company and fly ash supplied by Pozofly were used as supplementary cementitious materials. The specific densities of the cement, rice husk ash and fly ash were 3050 kg/m^3, 2510 kg/m^3 and 1890 kg/m^3, respectively.

Water Repellent

The water repellent based on silane siloxane, supplied by Souza Filho Impermeabilizantes LTDA (São Paulo/Brazil), is non-flammable and has low emission of volatiles. Its density is 1.01 ± 0.02 kg/l.

2.2 Wood Bio-Concrete Manufacturing and Curing Conditions

Wood bio-concrete (WBC) was produced with a volumetric fraction of 45% of wood shavings (WS). The cementitious matrix fraction was partitioned, in mass, by 45% of Portland Cement (PC), 25% of rice husk ash (RHA) and 30% of fly ash (FA), which were defined based on previous works [20, 21] to reduce the cement consumption. The water-to-binder ratio was set at 0.30 and calcium chloride (CC) was used as setting accelerator at a content of 2% in relation to the mass of cementitious materials. Wood bio-aggregates absorb water during the mixing process and, for that reason, it must be considered a compensation water (Wc) to guarantee good workability of the bio-concretes. Thus, the total water (Wt) is the sum of the cement hydration water (Wh) and the compensation water. Table 2 shows the consumption of materials, in kg/m^3.

Table 2. Mix proportions of wood bio-concrete (kg/m^3).

WBC	WS	PC	RHA	FA	CC	Wh	Wc
WBC45	238.5	344.5	191.4	229.7	15.3	229.7	166.9

The production process started with the addition and mixing of cementitious materials and bio-aggregates in a 20-L mixer for 1 min. Afterward, the total water was added, previously mixed with CC, progressively over 1 min. Total mixing time was 4 min. Specimens were molded in three layers, mechanically vibrated (68 Hz) for 10 s each, in cylindrical molds of dimensions 50 × 100 mm (diameter × height). After 24 h, the samples were demolded and kept in a room at temperature of 22 ± 2 °C and relative humidity of 55 ± 5% until reaching 28 days of age.

2.3 Silane Treatment Procedures

Two treatment methods were evaluated using the water repellent based on silane. The first one consisted of the surface application of a water-based solution with 5% of silane on the surface of bio-concrete samples using a brush (Fig. 1a). This surface skin protection (SSP) was made in two application coats, with an interval of 10 min between applications, totaling a consumption of 13.7 mL/m^2. The second method was carried out based on the work of Al Abdallah et al. [22]. Silane emulsion was dissolved at 2 vol% in water, then wood shavings were immersed in the solution for 2 h at room temperature to couple the silane with the bio-aggregate (Fig. 1b). The ratio of wood-to-silane solution was fixed at 10 g of wood in 100 mL of solution. Drying was carried out in an oven at 60 °C for 48 h. Finally, the impregnated bio-aggregate (IBA) was used in the production of wood bio-concrete. The silane-impregnated bio-aggregate changes the characteristics of the mixture in the bio-concrete production process since it produces less bubbles and enhance density of bio-aggregate.

(a) (b)

Fig. 1. Silane treatment procedures (a) SSP, and (b) IBA.

2.4 Wetting/Drying Cycles Protocols

Assessment of the durability of materials can be done in situ, exposing them to natural weathering, or in laboratory controlled conditions by means of accelerated aging tests [23]. In this work, an accelerated aging test was chosen and it consisted of a succession of immersion cycles in water for 30 min followed by drying in a room at 40 °C for 2 h, for ten times. The ten cycles were divided into three blocks with two rest periods, in which the samples were placed in a room with T = 23 °C and RH = 70%, for 14 h. This type of aging was selected based on intense rains followed by hot weather in short periods of time, characteristic of the summer in Rio de Janeiro/Brazil, and can simulate the surrounding conditions of materials during their life cycle. At the end of the aging process, the samples were placed in a room (T = 22 ± 2 °C and RH = 55 ± 5%) for 7 days, where other control wood bio-concrete samples (non-weathered) were stored. This procedure was done in order to have the same hygric and thermal equilibrium at the moment of the tests.

2.5 Functional Properties

Compressive Strength
The compressive strength test was performed according to Brazilian standard NBR 5739 (2018) [24] using a Shimadzu - 1000 kN at a test velocity of 0.3 mm/min.

Capillarity Water Absorption
A method proposed by RILEM (protocol in progress – TC HDB 275) was followed to measure the capillary water absorption of the bio-concretes. The samples were dried in an oven at 60 °C for 72 h. To ensure one-dimensional water transport, the lateral surfaces were sealed with metallic tape. The bottom of the samples was immersed in water at a depth of 5 mm for the following times: 1, 3, 5, 10, 15, 30 min, 1, 2, 3, 4, 5, 6, 24, 48 and

72 h. The water absorbed by capillary suction was measured by weighting the samples at the indicated times.

Microstructure

To investigate the effects of the silane treatments and the accelerated aging on the microstructure of the bio-concrete, weathered samples 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. In addition, energy-dispersive X-ray spectroscopy was performed on wood particles with and without silane impregnation to verify their chemical composition.

3 Results and Discussion

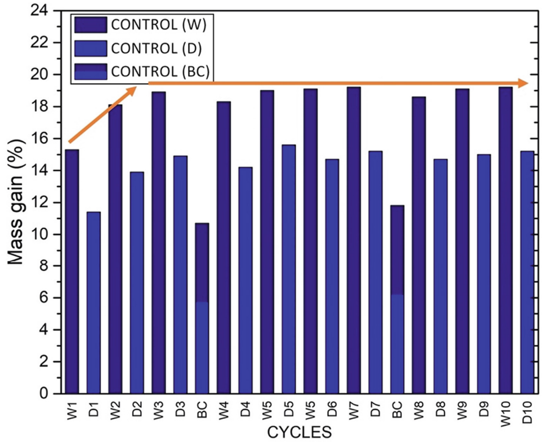
3.1 Wetting/Drying Mass Control

A control of mass (gain and loss) was performed during the wetting (W)/drying (D) cycles and in the between cycles (BC) period. Figure 2 shows the mass gain of control (CONTROL), surface skin protection (SSP) and impregnation of bio-aggregates (IBA) bio-concrete samples, as an average of 5 specimens.

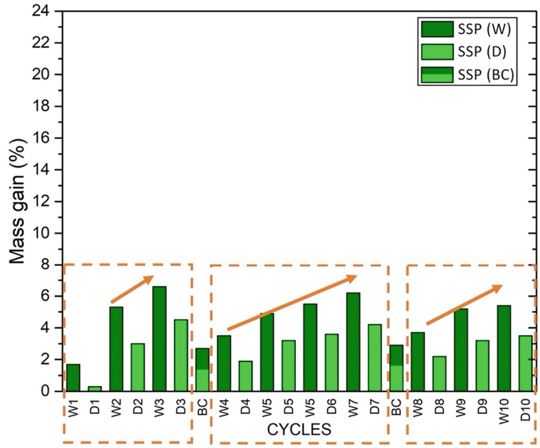
From Fig. 2a, it is noted that after the first wetting a mass gain of approximately 15% occured followed by a loss of just 4% after the drying for control samples. In the third cycle, the samples already reached their maximum absorption limit for 30 min of immersion and presented a similar behavior, with gains and losses of approximately 4%, until the 10th cycle.

For surface skin protection samples (Fig. 2b), the first wetting led to a mass gain of 1.7%, followed by a drying that caused a mass loss of 1.4%, practically returning to the initial sample mass and showing the potential of the silane to protect against water ingress in the first hours. In the second cycle, there was a mass gain of 5% and a loss of 2%. The third cycle followed the same trend. After the period between cycles (BC), a new wetting/drying block (cycles 4 to 7) behaved similarly to the two previous cycles (2 and 3), in which there is a mass loss of around 2.7%. The third block (cycles 8 to 10) showed the same trend. The use of silane as SSP was quite efficient in the first cycle, and despite the increase in water absorption in the other cycles it presented lower average mass gain (6.6%) compared to CONTROL samples (19.2%) under the study conditions. Christodoulou et al. [12] affirm that the Si-O bond that connects the hydrophobic layer and the substrate concrete can be destroyed by alkaline solution over time, UV radiation, high temperature and other factors, resulting in deteriorated protective performance.

IBA samples (Fig. 2c) showed a similar percentage of mass gain and loss from cycle to cycle (approximately 3% and 2%, respectively), which leads to a progressive mass gain over the cycles. In the 10th cycle, with absorption potential of 7.5%, it was not perceived a stabilization of an absorption plateau as for CONTROL and SSP samples. Despite the absence of a surface protection, IBA was able to reduce the ingress of water into the composite by creating a hydrophobic film on the wood shavings.



(a)



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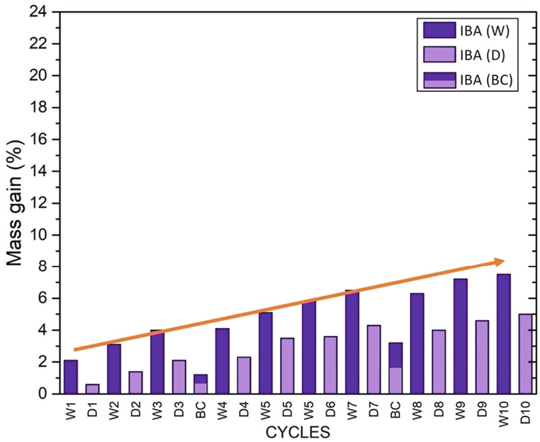

(c)

Fig. 2. Wetting/drying mass control of bio-concrete samples (a) CONT; (b) SSP and (c) IBA.

3.2 Functional Properties

Compressive Strength and Density

The compressive strength (fc_{28}) and density (ρ) values of the wood bio-concretes (weathered (W/D) and non-weathered) are shown in Fig. 3. Analyzing the results from Fig. 3a considering the non-weathered samples, an analysis of variance (ANOVA) was performed and there was no significant difference between CONTROL and SSP compressive strength, showing that the application of silane as surface treatment does not change this mechanical property of the bio-concrete. For IBA, a 23% increase in compressive strength was observed compared to CONTROL. This increase can be explained by the silane impregnation of the bio-aggregates, which changed the rheology of the mixture and made the bio-concrete denser, as can be seen in Fig. 3b.

The effect of wetting/drying cycles for CONTROL and IBA was similar, leading to a decrease of 13% and 9% of compressive strength, respectively. Behmahiddine et al. [23] explain a decrease in compressive strength after wetting/drying cycles of hemp concrete especially due to the degradation of the binder/bio-aggregate interface in addition to a possible increase in porosity, which weakens the mechanical behavior of the material. In addition, both showed greater water absorption capacity at the end of the wetting/drying cycles, compared to SSP. Despite being denser, CONTROL (W/D) has reduced mechanical strength due to the appearance of cracks in the transition zone between binder and bio-aggregate and in the bio-aggregate itself caused by the accelerated aging process. On the other hand, as the SSP samples showed a lower water absorption capacity until the last cycle, they did not present a substantial difference, according to ANOVA, in compressive strength compared to the non-weathered samples. Even after successive W/D cycles, the protective silane layer on the samples surface causes the bio-concrete to absorb less water in the wetting stage after the BC period, showing that the SSP samples were less damaged by the cycles than the others, which preserved their mechanical strength.

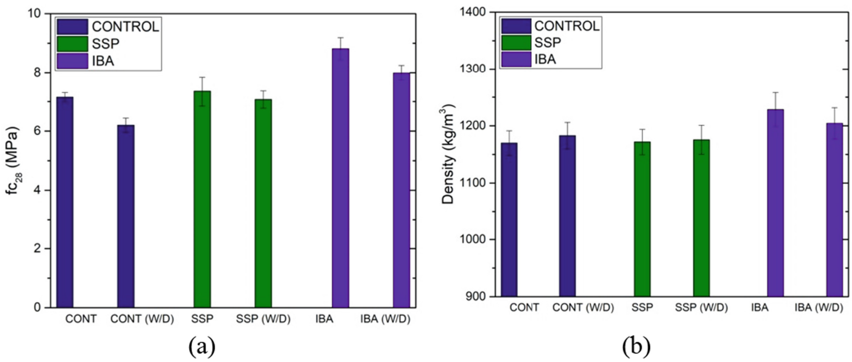

Fig. 3. Functional properties of bio-concrete samples before and after wetting/drying cycles (a) compressive strength, and (b) density.

Shen et al. [25] studied the efficiency of silane-based products as surface treatments for protecting degraded historic concrete and observed an increase in compressive strength of 16% using a water repellent based on silane/siloxane (50 wt. %) compared to untreated specimens, exhibiting a consolidation effect.

Capillarity Water Absorption

The water absorption curves of the silane treated bio-concretes are presented in Fig. 4. It can be seen that CONTROL samples presented a high potential to absorb water during the early hours, reaching about 20 kg/m^2 in 6 h of test and practically reaching saturation after 24 h of test (approximately 33 kg/m^2). Analyzing the difference of water absorption in silane application methods, SSP has a superficially hydrophobic characteristic capable of delaying the entrance of water into the sample during the first hours, with an absorption value of 9 kg/m^2 (55% lower than the CONTROL) in 6 h of test. On the other hand, this protection is temporary and the continuous contact of water with probably the sample disrupts the barrier of the silane film, meaning that after 48 h of testing SSP has similar absorption to CONTROL. IBA samples presented an intermediate behavior, because despite not having a surface protection, the water that entered into the cementitious paste did not have the same ease of penetration into the impregnated bio-aggregates. Thus, up to the 30 min of test, the absorption is low (3.5 kg/m^2), but it increased more than double after 1 h of test and the curve become closer to the CONTROL, reaching at 6 h of test a value of 17.3 kg/m^2 (13.5% less than CONTROL). The major difference is in the total water absorption capacity over time, which after 72 h of testing is 29.5 kg/m^2 (approximately 14% lower than CONTROL and SSP) and still does not show a level of stabilization. Zhu et al. [14], investigating the capillary water absorption coefficient of recycled aggregate concrete with surface silane treatment, found a decrease by 95% when compared to the control sample. This result can be related to the penetration depth of surface treated that depends on the type of hydrophobic agent applied, the porosity of the concrete, the initial moisture content, and the surface treatment of the concrete substrate [26].

By the capillarity water absorption tests, the bio-concrete (CONTROL) absorbed more water than the non-weathered ones. The wetting/drying cycles caused a tiny reduction in the absorbed water for CONTROL and IBA. For SSP, the behavior was inverse. The porous structure of bio-concrete deprived of any kind of treatment, even without the action of external agents, has a great potential for absorbing water, as expected, which consequently impairs the durability of this material. For SSP (W/D) samples, the wetting/drying cycles reduced the efficiency of the surface protection, and in the first hours. The absorption values of CONTROL after 1 and 6 h of testing were 150% and 56% superior when compared to SSP. However, in this same period, it presented a lower absorption value than control samples. After 24 h of testing, the values of SSP (W/D) are equal to SSP and show the same tendency for 48 h and 72 h. The behavior of the IBA (W/D) curve shows a greater absorption in the first minutes of the test, having a value 66% higher in 30 min of test compared to IBA. After 1 h of testing, the curves intersected and remained close until the end of the test. The rapid absorption in the first minutes may be related to the transition zone of the bio-aggregate and cementitious matrix that

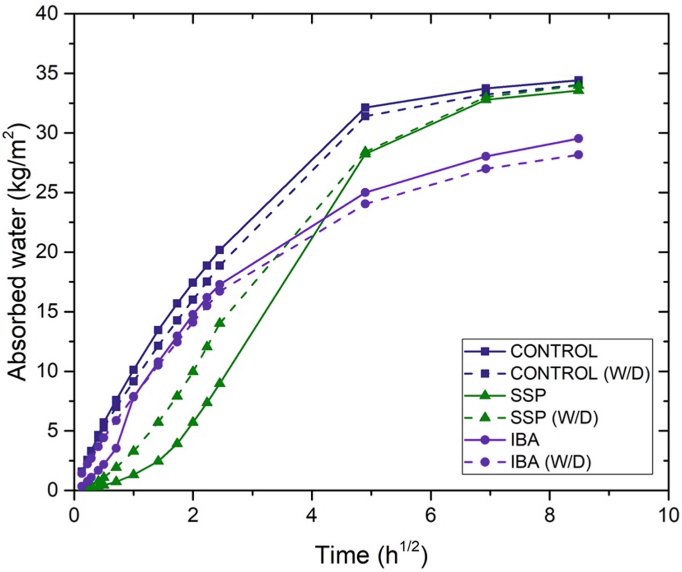

Fig. 4. Water absorption curves of the silane-modified bio-concretes non-weathered and after W/D cycles.

underwent changes during the wetting/drying cycles. But the final water absorption of IBA indicates the better performance when compared to SSP.

Microstructure

SEM images of internal bio-concretes samples before and after wetting/drying cycles can be observed in Fig. 5. For CONTROL (Fig. 5a) samples, the bio-aggregate (BA) is connected to the cementitious matrix (CM). CONTROL (Figs. 5a and 5b) and SSP (Fig. 5c) samples have a more porous structure due to a wood extractive that produces bubbles during the mixing process. These voids can further impair the durability of the material by an increase of concrete permeability, mainly under wetting/drying conditions (Figs. 5b and 5c). They create pathways for water transport and weaken the transition zone of the bio-aggregate and cementitious matrix. In addition, for CONTROL (W/D) samples, that promoted a faster entry of water, the drying process does not remove all the absorbed water, and the wood particle itself undergoes cracking. Figure 5d shows a sample of IBA, not as porous as others due to the impregnation process, but with the bio-aggregate-matrix interface cracked caused by wetting/drying cycles.

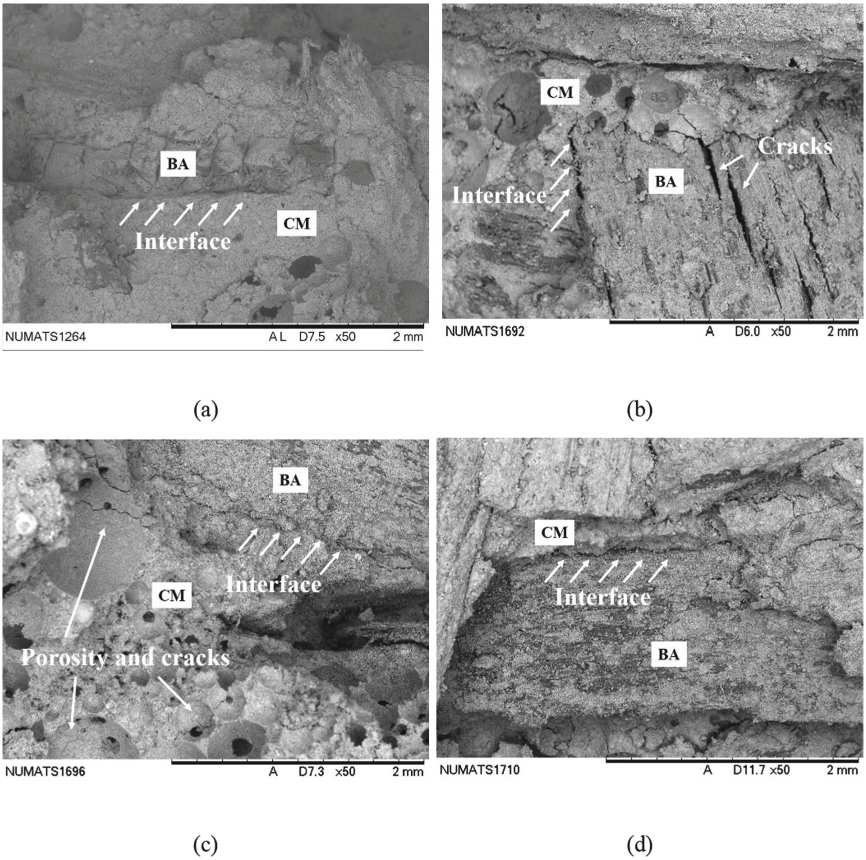

Fig. 5. SEM images of bio-concrete samples (a) CONTROL (b) CONTROL (W/D); (c) SSP (W/D) and (d) IBA (W/D).

4 Conclusions

The effect of silane application on the wood bio-concrete properties was evaluated. From the experimental results it is possible to draw the following conclusions:

- Both silane-based treatments studied proved to decrease the water absorption capacity of wood bio-concrete under immersion in the wetting stages;
- The wetting/drying cycles caused a decrease in compressive strength of 13% for CONTROL, 9% for IBA, and no change for SSP, showing that the surface treatment was more efficient for this mechanical property;
- According to capillary water absorption results, SSP samples had a temporary protection which delayed the ingress of water in the first hours, but up to 48 h of test, the water absorbed was the same with the CONTROL samples, while IBA has a 14% lower total water absorption capacity;
- Wetting/drying cycles negatively affected the performance of water absorption from the early hours for IBA and SSP samples;

- SEM images show the cracked transition zone of the bio-aggregate and cementitious matrix of bio-concrete, due to the aging process, which signals a reduction in the durability of the material.

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