

Influence of different methods of treating natural açai fibre for mortar in rural construction

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Abstract. Açai is a typical Amazonian fruit that has enormous potential for use in medicines and foods, whose consumption has been growing year after year. One of the major environmental impacts related to Açai is the generation of agro-industrial wastes, which are disposed of in landfills. One of the major problems related to the reuse of natural fibres in cementitious materials is related to their durability due to the alkalinity of the matrix. Thus, the objective of this work was to evaluate three different methodologies for surface treatment of Açai fibre, by immersion in NaOH, KOH and Ca(OH)₂ solution to mortar application in rural construction. After the treatments, the fibres were added in a proportion of 2.5 and 5.0% in relation to the cement mass, in addition to the reference mortar (without fibre) in the making of the prismatic specimens (40×40×160 mm) and cured for 28 days in room temperature. Right after the curing period, the specimens were evaluated according to the mechanical strength of flexion and compression, workability, water absorption by capillarity and mass density in the hardened state of each methodology. The results showed that the best treatment methodology is with NaOH solution, with the addition of 5% Açai fibre in relation to the cement mass, producing a suitable mortar for use in rural buildings.

Key words: agro-industrial wastes, açai, reuse, rural constructions, sustainability.

INTRODUCTION

The Açai is a small fruit with a rounded shape, usually dark in colour, with a small lump and its pulp (Sato et al., 2019). The fruit is found predominantly in the Amazon region, which spreads to countries like Brazil (predominantly), Peru, Colombia and Venezuela, but also in other regions of Brazil, spreading its cultivation in several places due to the constant increase of its added value in all world (Sato et al., 2020).

The most traditional açai is the fruit of the açazeiro (*Euterpe oleracea*), which is native to the amazon region, and in some cases is also called juçara, being a tree that can reach up to 30 meters in height, predominantly in the humid regions, favouring its growth in amazon region and coastal locations. The fruit's productivity is high, since in one hectare the production can reach 12 tons a year, when the appropriate management techniques are used (De Azevedo et al., 2021).

According to the Brazilian Institute of Geography and Statistics (IBGE) and the Municipal Agricultural Production (MAP) survey, Brazil produced more than 1.5 million tons of the fruit in 2018, a figure that has been growing day by day, reaching a projection of almost 2 million tons in 2021, due to the greater dissemination of this fruit internationally, which enhances exports, and research aimed at its application for medicinal purposes and uses in food (IBGE, 2019).

In Brazil, the state of Para is the main producer with 95% of the production of açai in Brazil (Cruz et al., 2019). Currently, 90% of açai production in Brazil is destined for the domestic market and 10% for export to about 40 countries, with the United States receiving the largest amount (Sato et al., 2020). Of the processed foods that contain açai and launched on the world market in the last 5 years, 22% are represented by juices, 12% energy and sports drinks, 9% snacks, 7% desserts and ice cream, 5% in the dairy category and 3% in sweets and bullets, with the United States (30%), Brazil (19%) and Canada (8%) the most representative countries in the launch of these products (Virmond et al., 2012). The açai market had a turnover of around 1.5 billion dollars in 2020 (De Azevedo et al., 2021).

All the growing production and processing of fruit for the manufacturing of its derived products generate great environmental impacts, whether in the matter of soil management, use of agricultural pesticides, increase in areas cultivated as a result of illegal deforestation in the Amazon region and the large generation of waste solids during the processing step (Silva et al., 2018). The açai fruit has a round shape and weighs about two grams. For the production of fruit-based products, a processing process must be carried out that results in the açai lump, since only 17% of the fruit is edible (pulp and peel), requiring about 2 kilos of fruit for a litter of juice (Virmond et al., 2012). The core is covered with small natural fibres, which are currently a major environmental liability since they are discarded in landfills or disposed of in open land, contaminating the environment (De Azevedo et al., 2021). Thus, in view of the constant growth of açai production and its popularization in Brazil and worldwide, the concern with sustainable practices for the generated agro-industrial solid waste is constant.

Some research has been developed around the world in relation to the use of açai lump and fibre, evaluating the potential of its reuse for medicinal purposes or even in the reuse in the development of alternative construction materials and energy generation. There are studies that assess the potential of energy generation, through açai, as a source of biomass, mainly in remote regions that use generators, such as in the Amazon region,

including reducing the costs of transporting this waste (Itai et al., 2014). Research by Oliveira et al. (2014) showed the seed of açai is a source of lignocellulosic material for the production of bioethanol, and that hydrothermal treatment can remove hemicelluloses from the seed of açai resulting in the improvement of the yield of monosaccharides released by enzymatic hydrolysis. There are also applications in composting, shown by Virmond et al. (2012), who concluded that the açai seed can participate in the process as a carbon supplier, reflecting in the improvement of soil characteristics.

A study performed by Barbosa et al. (2019) evaluated the potential of using açai lump for its application in the civil construction sector, being characterized physically, chemically, thermally and morphologically, carried out density tests by pycnometry, verification of moisture content, and the contents of lignin, cellulose and extracts. The results of this study showed that the use of açai seed potentiates the reduction of environmental impacts, providing the construction industry with the possibility of producing more ecological materials, generating sustainability indicators.

There are also applications in the making of ceramic materials, where the effect of incorporating ash from the açai stone in structural formulations was evaluated, which showed levels of incorporation of 15% ash at 1,050 °C in association with the clay mass for the manufacture of structural ceramics, improved physical and mechanical properties (Martins et al., 2014). Another study, by Valença et al. (2011) evaluated the mechanical behaviour of sand-asphalt mixtures with the insertion of açai fibre, added to construction and demolition residues and asphalt binders, showing that as insertion of açai fibre, provided greater aggregate-ligand interaction for the thick covering of surfaces. However, in this study the mechanical results have not demonstrated adequate performance, showing the need for further studies.

More recently, the application of only the natural fibre of açai in cementitious materials has been investigated, two recent published articles show the feasibility of using and applying additions of 3% of the fibre, treated with NaOH solution, in relation to the cement mass in the development of mortar reinforcement, this percentage was also favourable in terms of durability, as long as there is treatment in NaOH solution, called mercerization, due to the high alkalinity of the cementitious matrix (Marvila et al., 2020; De Azevedo et al., 2021). It is known in several works in the literature that the use of natural fibres in cementitious matrices must be preceded by the superficial treatment of the fibres, due to the impossibility of their use, however there are not many discussions about alternative methods to NaOH for the superficial treatment of fibres, which can result in performance improvements or financial costs (Marvila et al., 2020).

Although the treatment by immersion in NaOH solution is the most used and researched, alternative solutions have been evaluated, such as the immersion of fibres in acid solutions, as in the processes of acetylation, propionylation and silanization. These methodologies were used according to the chemical characteristics of the fibers, such as their hemicellulose and cellulose content (Azevedo et al., 2021). A major problem with the use of these treatment methods is their potential environmental damage that can be generated with remnants of the treatment solution and questions of durability of cementitious materials over the long period of exposure (Albinante et al., 2013). The chemical composition of the natural fibre, together with the elements present in its surface treatment process, react with the cementitious matrix, which throughout its

hydration process can promote a delay in the curing time, influencing the mechanical properties of the mortar (de Azevedo et al., 2021).

The acetylation and propionylation processes of natural fibres are related to their modification in order to make them more hydrophobic, arising from the reaction of esterification of the hydroxyl group of the fibre constituents (hemicellulose, lignin and amorphous cellulose) with the acetyl group (de Mendonça et al., 2020). The silanization method, on the other hand, is a chemical treatment with silicon compounds, whose molecules have, at one end, a hydrophobic terminal group and, at the other end, a hydrophilic group, forming bridges (Albinante et al., 2013).

Thus, the objective of this work was to evaluate three different methodologies for surface treatment of açai fiber, by immersion in NaOH, KOH and Ca(OH)₂ solution to mortar application in rural construction.

MATERIALS AND METHODS

Materials

The fibres and lump the açai were collected from the municipality of Rio Novo do Sul - ES - Brazil, in an agroindustry of açai production for ice cream. Fig. 1 shows the disposal site for this agro-industrial waste, where it is possible to observe the large amount of waste generated and its environmental liabilities.

The fibre was manually separated from the lump, resulting in the material that will be incorporated into the mortar. The other materials used for making the mortar will be natural sand, hydrated lime and Portland cement.

The sand used for the research was collected on the Paraíba do Sul riverbed, in the city of Campos dos Goytacazes - RJ, acquired from local companies that do the exploration. The sand was standardized in a 50 mesh sieve (fine sand), meeting the standards for use in cementitious materials. Hydrated lime type CH-III was used as a hydraulic binder in the mortar, providing greater strength and improving properties in the fresh state of the mortar. Finally, Portland cement type CP-II-E-32 was used, a type well used in the study region.



Figure 1. View of the açai waste deposit place.

Methods

The fibre was collected and cleaned with distilled water. After that, it was greenhouse dried at a temperature of 60 °C for 24 h (De Azevedo et al., 2021). After drying the fibres, they were submerged in the respective solutions for 30 min, immediately after the fibres are washed in natural running water and subsequently by immersion in HCl. Immediately after cleaning the fibers, they must be stored in an oven at 60 °C for 24 h. Açai fibre has a shorter length than other natural fibres, so it is expected to have a length of about 25 mm, and a diameter of approximately 0.30 mm (Castro et

al., 2010). The alkaline treatment by NaOH is one of the most used chemical treatments in several studies that study plant fibbers (Castro et al., 2010; Marvila et al., 2020). In this work, in addition to NaOH, the fibre was subjected to treatment with KOH and Ca(OH)₂. The use of more than one type of hydroxide, aimed to observe the behaviour of fibrous reinforcement for more than one type of alkaline medium. The solutions were subjected to the application of the three types of hydroxide, as shown in Table 1.

The açai fibre was immersed in the respective solutions for a period of 30 min, soon after, the fibre was washed with distilled water and acetic acid, which neutralize the fibre, interrupting the reaction by removing the residual hydroxyl (De Azevedo et al., 2021). Subsequently, the fibres were washed with running water, allowing removing the excess hydroxide still contained in the fibre. Afterwards, the fibres were again dried in greenhouses.

Mortars were made according to the Brazilian technical standard, with a material ratio of 1:1:6 (cement: lime: sand), in mass, where the fibres after each treatment were added in the proportions of 2.5 and 5.0% in relation to cement mass, in addition to the reference mixture, without any addition of fibre (NBR 13276, 2016). Prismatic specimens of 40×40×160 mm were used for each mixture and exposed to an ambient cure (approximately 23 °C) in the period of 28 days.

The consistency index test follows the standard of the standard (NBR 13276, 2016), on mortar for laying and covering walls and ceilings - determination of the consistency index. The test uses a circular thickening table, and consists of measuring the horizontal spread of the mortar molded in a standardized cone, after the application impacts on the mortar, providing an adequate workability to the mixture.

The three-point flexural strength test was carried out on a universal testing machine with an S 30-capacity ballot through the application of a $50 \pm 10 \text{ N s}^{-1}$ load. After the flexural rupture, the remaining parts were used for the compression test, with a load application of $500 \pm 10 \text{ N s}^{-1}$ (Ahmad & Fan, 2018). The mass density test of the material was carried out by measuring three measurements of the prismatic specimens with a calliper to obtain the volume of the material, and the weighing of the mass with the aid of a scale. Then the density is calculated by dividing the mass by the volume (NBR 13278, 2005a).

Capillarity occurs when there is contact with liquid with the substrate. The liquid in contact with a solid surface or capillary walls of a porous material generates forces of interaction and repulsion between the liquid and the solid, causing part of the liquid to be absorbed by the porous material (Lertwattanakruk & Suntijitto, 2015). This is detrimental to cementitious material. Since water affects the bond between the aggregate and the slurry, it influences the strength of the concrete. The capillary water absorption test induces the absorption of water by the cementitious material. The specimen was weighed at 0, 10 and 90 min, and placed in contact with a water layer of approximately 5 mm (NBR 15259, 2005b). After that, the specimens were weighed again, the difference in mass indicates how much water was absorbed.

Table 1. Concentration of solutions used in alkaline treatment

Hydroxide type	Concentration
NaOH	21 g to 100 mL of water
Ca(OH) ₂	21 g to 100 mL of water
KOH	15 g to 100 mL of water

RESULTS AND DISCUSSION

The Table 2 shows the results related to the mixing of the materials, such as the proportion, in mass, of the constituents for making the mortar. It is observed that for each type of treatment methodology, the fibres were added proportionally to the cement mass, without changing the quantity of the other materials, such as cement, lime and sand.

Table 2. Workability properties of mortars and mixtures

Treatment Type	Cement (g)	Lime (g)	Sand (g)	Water (g)	Fibre (g)	water/cement	Horizontal spreading (mm)
NaOH	100	100	600	45	2.5	0.45	259 ± 14
	100	100	600	45	5.0	0.45	257 ± 12
Ca(OH) ₂	100	100	600	45	2.5	0.45	277 ± 10
	100	100	600	45	5.0	0.45	264 ± 6
KOH	100	100	600	45	2.5	0.45	278 ± 11
	100	100	600	45	5.0	0.45	262 ± 18

It can be seen in Table 2 that the water/cement ratio was kept constant, at 0.45, which is a value widely used in the literature on cementitious composites for reinforcement with natural fibres (Candamano et al., 2020; Morón Barrios et al., 2021), another point is that when choosing to keep this parameter fixed, the comparison ratio between mixtures and the evaluated treatment methods is better.

When we evaluate the horizontal spreading, which is a property directly related to the workability of the mixture, we have that the literature speaks of values around 260 ± 5 mm, which is the same as that adopted in the Brazilian technical standard for applications in coating mortars (NBR 13276, 2016; De Azevedo et al., 2021). Therefore, it was decided to use this reference value, and the treatment with NaOH (mercerization), was be showed with the most stable values and within the acceptable range, keeping a mortar with good workability.

In the Ca(OH)₂ and KOH treatments for the 2.5% fibre content, the nominal average spreading exceeded the maximum limit, even within the standard deviation values, this can be attributed to the lower adhesion that the fibre had, with these treatment methodologies, along with the cementitious matrix, resulting in more fluid mortars (Marvila et al., 2020). With higher levels, such as 5%, both treatments obtained nominal values within the acceptable range (260 ± 5 mm), but still very close to maximum limit, which may denote not being a significant and acceptable result for the application in rural construction (García-Esparza et al., 2018).

Thus, the treatment with NaOH was the one that showed the greatest stability regarding the horizontal spreading of the mortar, showing an acceptable workability according to the literature values (Marvila et al., 2020). Another important point is that for applications in rural constructions, the workforce want at a mortar with workability, since the buildings present a facades greater irregularity to application surface and greater exposure to heat, which can influence the final durability of the coating in the environment rural (García-Esparza et al., 2018; Marvila et al., 2020).

Fig. 2 shows the results of the mass density of the specimens in the hardened state, that is, after the curing period.

Fig. 2 shows the result of the mass density in the hardened state, where comparatively the reference mixture (without the addition of any fibre), the treatment with NaOH was the one that showed the best behaviour, with reduced density in all two levels of incorporation, which is beneficial for mortars in general, as it reduces the load on masonry in rural constructions (De Azevedo et al., 2021). This reduction with the treatment of NaOH can be justified due to the greater impermeability that the surface treatment caused in the fibres, reducing the effect of the alkaline medium in the matrix in the reinforcement (Asim et al., 2020). The treatments with KOH and $\text{Ca}(\text{OH})_2$ were less efficient since there was a dissipation of part of the formed film, which in the case of $\text{Ca}(\text{OH})_2$ was influenced by the hydration of the cement (Morón Barrios et al., 2021).

The results of the literature itself indicate that mortars for use as external cladding of facades should have a mass density around 1.7 to 1.9 g cm^{-2} , which shows that the treatments of the fibres with KOH and $\text{Ca}(\text{OH})_2$ do not are recommended for this property (Marvila et al., 2020; De Azevedo et al., 2021). Obviously, the amount of fibre addition also influenced, since greater proportions indicated a possible increase in density, which corroborates with other studies in the literature on the use of natural fibres in mortars for coating (Asim et al., 2020).

Fig. 3 shows the result of water absorption by capillarity for the different fiber treatment methodologies.

The absorption of water by capillarity is a phenomenon that evaluates the interconnection potential of the internal pores, which culminates in the transfer of fluids, such as water or external gases, into the cementitious matrix, affecting its behaviour and durability (Nadelman & Kurtis, 2019).

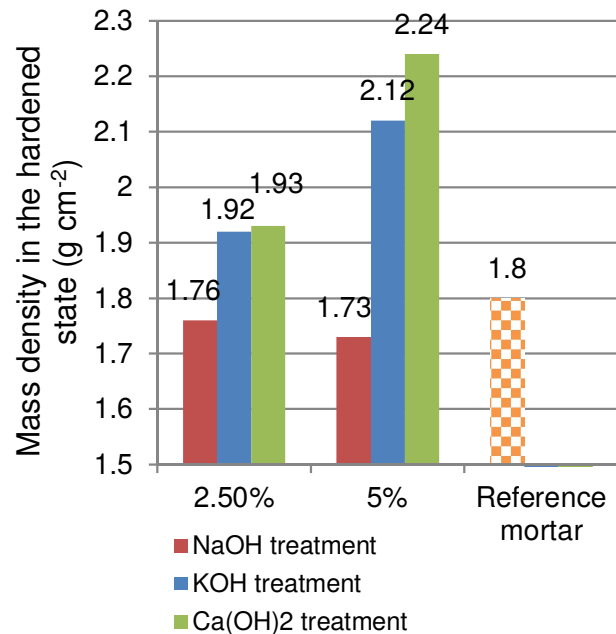


Figure 2. Results of mass density in the hardened state of each methodology of treatment.

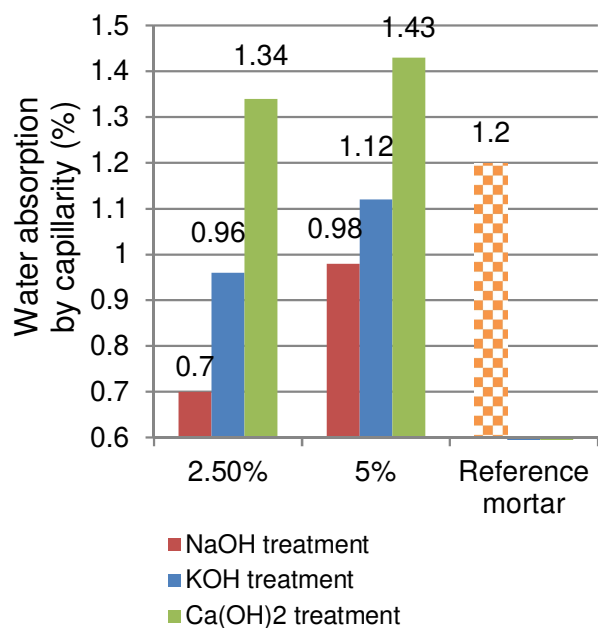


Figure 3. Results of water absorption by capillarity of each methodology of treatment.

It was observed in Fig. 3 that the treatment with $\text{Ca}(\text{OH})_2$ induced an increase in this water absorption by capillarity due to the fact that this treatment, which is based on hydrated lime dissolved in the aqueous solution, participates in the hydration process of cement matrix, together with existing cement, reducing the protection efficiency, which helps in the hydrophilic power of natural fibres, in this case more exposed (Marvila et al., 2020).

In the case of treatment with NaOH and KOH what was observed was a reduction, mainly with NaOH, which is justified by the fact of the actual superficial impregnation of the treatment in the fibre, which reduces its effect of formation of capillary veins. In addition, there is a greater internal compactness of matrices with Açai type fibres, as they are shorter and less thick, which corroborates their internal filling effect, in addition to the reinforcement, thus reducing the capillary absorption effect, as observed in results of Fig. 3 (Nadelman & Kurtis, 2019).

Rural buildings are highly susceptible to exposure to the effects of heavy rains, and depending on the region with high temperature rates, a positive point of these environments is the lower exposure to gases, such as atmospheric CO_2 , which reduces the incidence of pathologies in this sense in these areas buildings (García-Esparza et al., 2018).

Figs 4 and 5 show the results of mechanical strength flexion and compression, respectively, in the different treatment methodologies analysed. All results of mechanical strength are preceded by the respective standard deviations, as a result of the samples of the specimens used in this study. It can be seen visually that the specimens before the execution of the tests, in all treatment conditions, presented a good consolidation of the mortar, without occurrence of voids due to failure of the density of the dough in the moulds and the occurrence some small parts of these fibres for the external side. After the execution of the tests of mechanical strength to compression and flexion, it was observed that the test pieces with NaOH treatment presented, in the rupture region, greater shortening of the fibres that were left out in the test pieces, due to the greater resistance that they imposed on these samples. In the other treatments, the fibres were severely broken, showing the greatest fragility of these samples.

The results observed in Fig, 4 indicate that the reference mortar (without adding natural fibers in its mixture), presented a mechanical resistance to flexion of 3.64 MPa, this value is consistent with the main applications that relate the coatings, which according to the study from the literature, it must present at least 3 MPa for medium

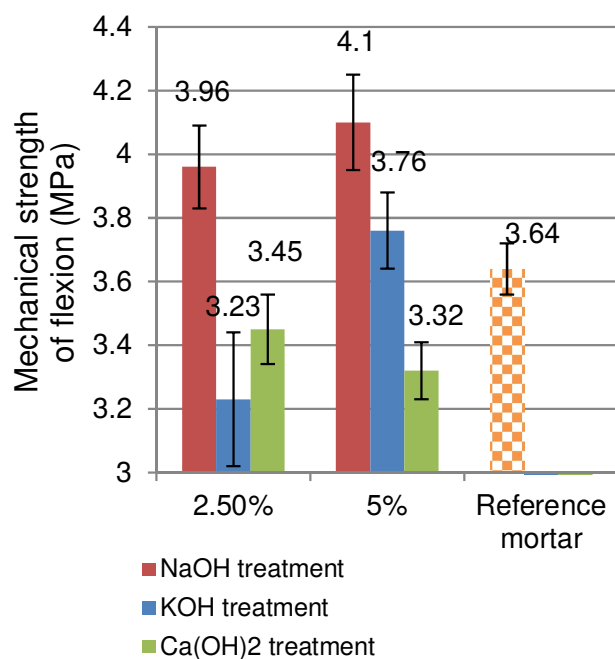


Figure 4. Results of mechanical strength of flexion of each methodology of treatment.

aggressive environments (Hong et al., 2020). Thus, if we analyze all other treatment methodologies, we can conclude that in all of them this minimum value is reached, with the exception of the incorporation of 2.5% with KOH treatment, which in its standard deviation (tests performed in triplicate), was found close to the limit.

When analysing the treatment with NaOH, it presented the greatest increase in this resistance, about 8.79% (2.5% of fibre) and 12.63% (5% of fibre), which can be justified by the influence of the increase in the internal stiffness of the matrix caused by addition of fibre as a reinforcement material.

In other treatments, such as KOH and mainly with Ca(OH)₂, the effect of adding the fibre was more like filling the matrix, reducing performance in most cases of strength, this can be justified by the lower or almost no efficiency of this surface treatment systems, which were deteriorated by the aggressiveness of the alkaline medium of the cementitious matrix (Ahmad & Fan, 2018).

In Fig. 5, the compressive strength underwent a similar trend to that observed in flexion, with treatment with NaOH providing a significant increase in the strength values of 14.51% (2.5% fibre) and 31.45% (5% fibre), thus being able to effectively conclude that the fibre acted as a reinforcement mechanism in the mortar, which for applications in external coatings of rural buildings is positive (Candamano et al., 2020). The reduction in the compressive strength values is just as significant, highlighting for mortars treated with Ca(OH)₂ that in some specimens they deteriorate to such an extent during the curing period that the rupture was impaired, proving inefficiency application of this treatment process for Açai fibres (Mathavan et al., 2020).

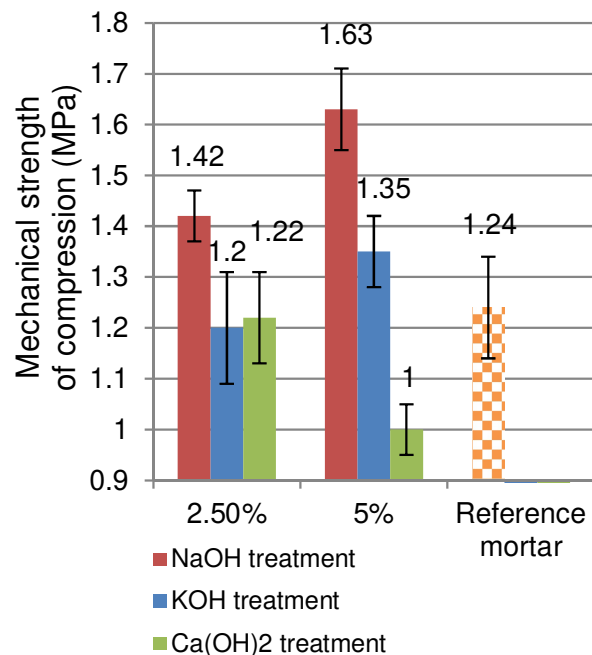


Figure 5. Results of mechanical strength of compression of each methodology of treatment.

CONCLUSIONS

It can be concluded with this research that there is a viability of using the natural fibre of Açai in mortars for applications in rural buildings, as long as the fibre undergoes a treatment process. The immersion of natural fibre in NaOH solution, a process known as mercerization, was the one that presented the best properties of mortars in the fresh and hardened state, as a protective film of the fibres was developed in the interfacial region that prevented their direct contact with the internal pores of the highly alkaline cementitious matrix.

Other methodologies for the treatment of the natural fibre of Açai, such as immersion in KOH and Ca(OH)₂ solution, were not as efficient, in these cases the fibre behaved as a filler in the matrix, while in the treatment with NaOH it provided that the Açai fibre behaved as a reinforcement of the matrix, which was verified in the results of mechanical strength.

As for the percentages of addition of natural fibre, it was observed that values of 5% in relation to the cement mass have adequate behaviour and within the normative standards and the literature of the area. Although higher percentages are not evaluated, other studies related to the use of natural fibres in cementitious matrices have shown that these higher values are harmful. Thus, treatment by immersion in NaOH solution with the addition of 5% Açai fibre is possible for making mortars for use in cladding and stabbing in rural buildings.

As a suggestion for other works, it is possible to verify the influence of the orientation and arrangement of the fibres in the molding process of the specimens, in addition to other intermediate additions to those already evaluated.

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