Influence of chemical treatment of rice husk on the performance of sustainable composites

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Abstract: Nowadays, the concept of sustainability and eco-efficiency are included in several strategic documents of national and European policies. According to this conceptual vision, the practice of incorporating undervalued and underused agricultural waste and by-products has emerged with the purpose of generating a positive environmental impact. Natural residues, such as rice husk, have several positive characteristics, such as their lightness, thermal and acoustic behavior, and negative effect that can influence the performance of composites. Through chemical treatments, there is the possibility of improving the characteristics of the rice husk, improving the interaction between the rice husk and the cement paste and, consequently, increasing the mechanical strength of the composites. The present investigation has as main objective to analyze cementitious composites of rice husk through a chemical treatment procedure with sodium silicate, comparing the results obtained with the conclusions reached when using the sodium hydroxide pretreatment. This dissertation also aimed to study the effects that both pre-treatments (sodium silicate and sodium hydroxide) produce on the rice husk. In this way, several procedures were carried out to evaluate the characteristics of the husk, before and after treatment. The characteristics of cementitious pastes and composites produced with this waste were also analyzed, evaluating their properties in the fresh and hardened state. It is concluded that the treatment with sodium silicate changes the hydrophilic characteristics of the rice husk, resulting in an increase in the characteristics of the husks and the mechanical properties of the composites, obtaining the best increment for the concentration of 6%.

Keywords: Rice husk, chemical treatment, sodium silicate, cementitious composites, sodium hydroxide.

1. INTRODUCTION

Over time, there has been an increase in environmental concerns, both in the political sector and by society. Environmental concerns about CO_2 emissions and the management of natural and non-renewable resources are very important. Another major environmental problem is reducing the environmental impact caused by landfilling agricultural waste and by-products. The reuse of agricultural by-products, such as rice husk (RH), in cement composites contributes to the sustainability of the concrete industries (Carmona et al. 2013).

The incorporation of fibers into composites is a theme that has been studied for many years. However, in recent years, there has been an increase in interest in vegetables natural, leading to a continuous growth of their use in composites. RH is not yet one of the most used vegetables materials in composites (Soltani et al., 2015). One of the reasons is that there is not yet much research on the proper procedure for treating these materials in order to optimize the performance of cementitious materials produced with RH.

In the present investigation, it was intended to analyze cementitious composites with rice husk, evaluating the influence that pretreatment with sodium silicate produces in RH. In addition, it is intended to compare the results obtained with the research carried out on RH subject to pretreatment of sodium hydroxide.

2. LITERATURE REVIEW

2.1 RICE HUSK CHARACTERIZATION

Rice husk (RH) is made up of vegetables fibers and is characterized by being a natural material corresponding to the group of vegetable fibers. As can be seen Figure 2.1 in order to obtain the rice husk, it is necessary to milling the rice in husk, resulting in the husk and rice with bran (brown rice).

The husk is characterized by having a high volume, a high porosity and a low density, consisting of approximately 50% cellulose, 30% lignin and 20% silica (Fernandes et al. 2016).

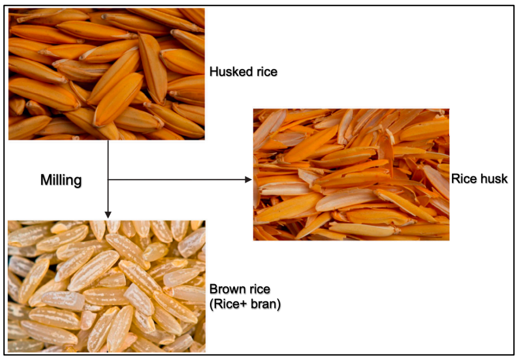

Figure 2.1 – Illustration of the process and constituents of rice. Adapted from Buggenhout et al. (2013).

Regarding its morphology, RH is composed of four structural layers: external epidermis, sclenochy or hiderm fiber, spongy parenchyma cell and internal epidermis (Houston 1972).

2.2 NATURAL FIBERS

Fibers can be of two types, natural or synthetic fibers. Within natural fibers there are three groups, vegetables, animals or minerals, which are classified according to their origin (Chandramohan & Marimuthu 1996). Natural vegetable fibers are complex polyemic compounds, with a three-dimensional structure consisting mainly of cellulose, hemicellulose, pectins and lignin (Clemons & Caulfield 2005).

2.3 CEMENTITIOUS COMPOSITES WITH RESIDUES SUBJECT TO PRETREATMENT

Composite performance depends on the physical properties of the composites and matrix, but it also depends heavily on the interface that exists between these two elements. Many phenomena of relevant importance occur at the interface, and such phenomena are dependent on their structure and the tensions generated there (Pilato & Michno 1994).

The use of RH can lead to the appearance of some more fragile areas in the composites, as a result of the weak connection between RH and cement paste and the high absorption of moisture/ water from the husk. To overcome these problems, it is intended to treat these fibers with chemical methods in order to improve their properties. Within chemical methods, there are numerous treatments, such as alkaline, through sodium hydroxide (NH) and mineralization, through sodium silicate (NS).

2.3.1 Influence of pretreatment with sodium hydroxide

The chemical treatment of alkalinization with NH aims to remove the moisture content of the fibers, increase their bending resistance and remove all impurities adjacent to this residue (Chandramohan & Marimuthu 2011). This solution removes waxes and oils from the surface of the fibers, which contributes to an increase in adhesion between the fibers and the matrix (Li et al. 2007).

According to Esteves (2021), there is a morphological change in the fiber through the pre-treatment of alkalinization of rice husk.

2.3.2 Influence of pretreatment with sodium silicate

As there are no investigations on the use of pretreatment of sodium silicate in RH, an analysis of the use of this pretreatment in other plant fibers, such as wood, was performed (Table 2.1).

Table 2.1 – Treatments with sodium silicate applied to vegetables fibers.

Material	Sodium silicate concentrations	Reference
Chinese fir	20%	Li et al. (2020)
Southern Pine	5%, 15%, 25%	Peng e Gardner (2010)
Pine	5%, 15%, 25%	Peng, Han e Gardner (2012)
Wild pine	15%	Pfeffer, Mai e Militz (2012**)**

According to Li et al. (2020), the use of pretreatment of sodium silicate, through the impregnation method, in wooden specimens causes a filling in the lumen or even in the cell walls of wood. This treatment may also result in the formation of chemical bonds between sodium silicate and cell wall substances.

This pretreatment causes improvements in wood properties, such as weight gains, density growth rate, resistance to biological agents, bending resistance, compressive strength and dimensional stability (Ghosh et al. 2012).

3. MATERIALS AND TESTING METHODOLOGIES

3.1 MATERIALS

In the present experimental campaign, four materials were used to produce the pastes and composites: cement (CEM II/A-L 42,5R), water, rice husk (RH) and sodium silicate powder (NS).

3.2 COMPOSITION OF PASTES AND COMPOSITES

In the present investigation, four composites were produced, the reference and three with a pretreatment in rice hush with different concentrations of NS (3%, 6% and 9%). Table 3.1 shows the compositions of the composites produced.

Table 3.1 –Compositions of composites.

Mixtures	Pretreatment	Cement (kg/m^3)	Effective water (kg/m^3)	RH (kg/m^3)
C_Ref	T0	660,5	381,8	130,9
C_S3%	NS3	641,6	506,6	121,7
C_S6%	NS6	630,2	529,9	132,0
C_S9%	NS9	658,1	412,0	126,4

Legend: C_Ref – composite of reference RH; C_S3% – composite of RH pretreated with NS3; C_S6% – composite of RH pretreated with NS6; C_S9% – composite of RH pretreated with NS9; T0 – pretreatment with 0% sodium silicate; NS3 – pre-treatment with 3% sodium silicate; NS6 – pretreatment with 6% sodium silicate; NS9 – pretreatment with 9% sodium silicate.

In order to analyze the characteristics of the cementitious matrix present in the composites mentioned, four pastes were also produced, the reference and three with water resulting from the immersion of RH with pretreatment (with 3%, 6% and 9% of NS). Table 3.2 shows the compositions of the produced pastes.

Table 3.2 – Mixtures of pastes produced in the experimental campaign.

Mixtures	**Pretreatment**	**Cement (kg/m^3)**	**Water (kg/m^3)**
P_Ref	T0	1580,2	489,8
P_S3%	NS3		
P_S6%	NS6		
P_S9%	NS9		

Legend: P_Ref – paste of reference; P_S3% – paste produced with the water of RH pretreated with NS3; P_S6% – paste produced with the water of RH pretreated with NS6; P_S9% – paste produced with the water of RH pretreated with NS9; T0 – pre-treatment with 0% sodium silicate; NS3 – pre-treatment with 3% sodium silicate; NS6 – pretreatment with 6% sodium silicate; NS9 – pre-treatment with 9% sodium silicate.

3.3 DESCRIPTION OF PRE-TREATMENTS

In Figure 3.1, it is possible to observe the process of preparation and production of pastes and cementitious composites. For each of these cementitious materials, the procedures used in the reference mixtures and the others are presented separately.

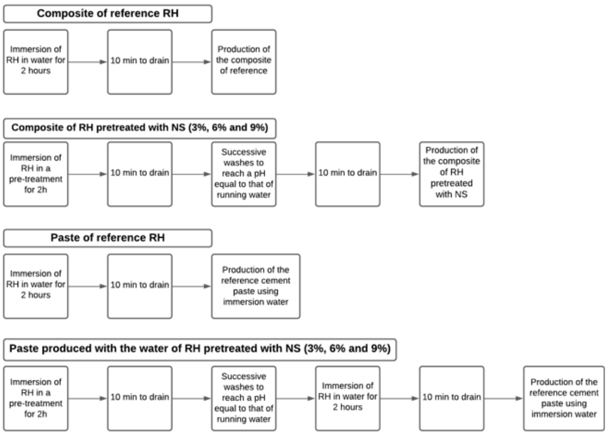

Figure 3.1 – Illustrative procedure for the preparation and production of cementitious pastes and composites.

4. RESULTS AND DISCUSSION

4.1 CHARACTERIZATION OF RH

The use of RH in composites has some disadvantages, related to its chemical components. Thus, the use of this residue can lead to the appearance of some more fragile areas in the composites, as a result of the weak connection between RH and cement paste and the high absorption of moisture/water from the husk. To overcome these problems, it is intended to treat these fibers with chemical methods in order to improve their properties.

Through the present study, it was possible to conclude that the use of a pretreatment with NS in RH causes some changes in the characteristics of this residue. Table 4.1 shows a summary of the main properties analyzed in RH. It can also be verified the variation that the pre-treatment with NS caused in the various characteristics on the husk, compared with the values of RH without pretreatment.

As can be seen in Table 4.1, it was possible to verify that the dimensional variation of RH, when subjected to a change from wet to dry state, is much higher in RH subject to sodium silicate treatment. As will be examined later, this aspect has negative consequences on the properties of composites, in terms of durability.

Through the values of bulk density and volume mass of dry and saturated RH with dry surface, with and without pre-treatment, it was found that there is no significant variation of these properties with the use of pretreatment. However, regarding the bulk density of RH in the saturated state with wet surface, an increase was observed with the use of 3% and 6% of pretreatment. This increase indicates that the treatment causes increased water retention by RH, with an impact on compaction efficiency. As can be seen later, this increased water retention by RH benefits the characteristics of RH and, consequently, the properties of composites produced with these husk.

Table 4.1 – Tests performed in the RH without and with pretreatment of NS.

RH testing	RH_Ref	RH_S3%	RH_S6%	RH_S9%
Variation of dimensional RH between saturated and dry states [%]	-1,82	-7,41	-8,56	-10,00
	-	57%	370%	449%
Bulk density of RH$_s$ [kg/m^3]	72,36	72,01	71,65	73,06
	-	0%	-1%	1%
Bulk density of RH$_{ssh}$ [kg/m^3]	165,09	181,95	229,01	159,47
	-	10%	39%	-3%
ρ_{rd} according to NP EN 1097-6 [kg/m^3]	649,9	622,1	687,1	630
	-	-4%	6%	-3%
ρ_{ssd} according to NP EN 1097-6 [kg/m^3]	1180,2	1133,8	1145,1	1145,8
	-	-4%	-3%	-3%
WA$_{24}$ according to NP EN 1097-6 [%]	81,6	82,3	66,7	82
	-	1%	-18%	0%
TOC content of immersion water [mgC/l]	38	10	11	10
	-	-74%	-71%	-74%

Legend: RH$_s$ – dried rice husk; RH$_{ssh}$ – saturated rice husk with moist surface; ρ_{rd} – volume mass of kiln-dried particles; ρ_{ssd} – volume mass of particles saturated with dry surface; WA$_{24}$ – water absorption.

Though the TOC values presented in Table 4.1, it is shown that, at the time of the production of composites, there is a smaller amount of organic compounds released by the subject to pretreatment. This conclusion was found that pretreatment with NS allows to increase the amount of organic compounds released over the pretreatment itself.

4.2 CHARACTERIZATION OF PASTES

To evaluate the evolution of the characteristics of the various pastes, 4 prismatic specimens of 160×40×40mm were produced.

Table 4.2 shows a summary of the properties analyzed in the pastes produced, with and without pre-treatment, as well as the variations obtained for the paste of reference. Thus, it can be concluded that there is a maintenance of the properties in the fresh state (volume mass and consistence by flow table) with the use of immersion water of RH resulting from pretreatment. The same was verified in the dynamic modulus of elasticity (MED), ultrasound velocity (USV) and open porosity tests. In turn, as can be seen in Table 4.2, it was possible to conclude that the mechanical strength according to EN 1015-11 (compression and flexion) improve with the use of pretreatment. These results are due to the lower content of organic compounds released by the pretreated bark, as previously observed.

Table 4.2 – Results obtained in the tests performed on cementitious pastes.

Tests the pastes	P_Ref	P_S3%	P_S6%	P_S9%
Volume mass in fresh state according to EN 1015-6 [g/cm^3]	2,07	2,08	2,08	2,07
	-	0%	0%	0%
Consistence by flow table according to EN 1015-3 [cm]	20,8	20,9	20,5	20,5
	-	0%	-1%	-1%
Compressive strength at 28 days according to EN 1015-11 [MPa]	61,6	63,4	68,2	66,8
	-	3%	11%	8%
Bending resistance at 28 days according to EN 1015-11 [MPa]	1,8	2,3	2,5	2,2
	-	28%	39%	22%
MED at 28 days according to ASTM E 1876-01 [GPa]	25,9	25,7	26	25,5
	-	-1%	0%	-2%
USV at 28 days [m/s]	4060	4058	4084	4068
	-	0%	1%	0%
Open porosity at 28 days according to EN 1936 and RILEM I.1 [%]	28	28	28	28
	-	-1%	-1%	0%

Legend: MED – dynamic modulus of elasticity; USV – ultrasound velocity; Green – positive variation greater than 2%; Red – negative variation greater than 2%.

4.3 CHARACTERIZATION OF COMPOSITES

To evaluate the evolution of the characteristics of the various composites, 4 prismatic specimens of 160×40 40mm were produced.

4.3.1 Characterization in mechanical terms

In Tables 4.3 and 4.4 can be observed a summary of the main properties studied in composites with RH, with and without pretreatment, as well as the variations obtained for the reference composite.

Table 4.3 – Results achieved in mechanical tests performed on cementitious composites.

Composite testing	C_Ref	C_S3%	C_S6%	C_S9%
Volume mass in fresh state according to EN 1015-6 [g/cm^3]	0,88	0,96	0,95	0,93
	-	9%	8%	6%
Compressive strength at 28 days according to EN 1015-11 [MPa]	3,0	4,2	4,3	4,0
	-	40%	43%	33%
Bending resistance at 28 days according to EN 1015-11 [MPa]	8,3	8,8	9,7	8,7
	-	6%	17%	5%
MED at 28 days according to ASTM E 1876-01 [GPa]	3,4	3,8	4,0	3,7
	-	12%	18%	9%
USV at 28 days [m/s]	2023	2237	2545	2219
	-	11%	26%	10%

Legend: MED – dynamic modulus of elasticity; USV – ultrasound velocity; Green – positive variation greater than 2%; Red – negative variation greater than 2%.

Regarding mechanical properties, it was concluded that the use of RH subject to pretreatment caused a generalized and, in some cases, significant increase of these properties. Thus, an increase in mechanical properties was observed with the increase of the concentration of pretreatment up to 6%. On the other hand, it was found that the use of pretreatment concentrations of NS higher than

6%, did not cause a greater increase in these properties. The hydrophilic characteristic by the husk seems to play a very important role in improving the mechanical properties of the composites with husk, since it contributes to the highest water retention. As a result, it is possible to remove more air volume during compaction, resulting in increased volume mass. It is also expected a greater adhesion between the paste and the surface of the husk, with positive impact on the mechanical strength of the composite.

Figure 4.1 shows photographs obtained in the binocular magnifying glass of each composite. Through this technique, it was possible to observe the places where the various composites were ruptured.

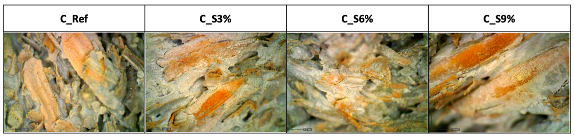

Figure 4.1 – Images of the different cementitious composites captured using the binocular magnifying glass.

The composite C_S6% is the one with the highest bending resistance, and it was found that the ITZ between the husk and the paste is stronger than in the other composites. That is, after an analysis with the binocular magnifying glass, it was found that in this composite there is a greater tendency for the rupture to occur by the fibers and not by the ITZ. On the contrary, this analysis allowed to verify that the lower resistance obtained in the C_Ref results from the lower force existing in the ITZ between the husk and the paste, since it was observed that the rupture in this composite occurred mainly by the ITZ.

These results show that the use of pre-treatment with NS in RH causes an improvement in the characteristics of these fibers and in the ITZ between them and the paste. It should be refound that, in the analysis of leachates, it had been verified that the RH without pretreatment contain a lower TOC value, which shows that the use only of running water in RH does not allow a reduction of the organic elements (waxes, oils and organic material) existing in this residue. Thus, this expulsion of organic compounds, through the use of pre-treatment with NS, may be the reason for the improvements observed in the ITZ between the peels and the paste and, consequently, the increase in the resistance of cementitious composites.

4.3.2 Characterization in terms of durability

Regarding properties in terms of durability, it was concluded that the use of pretreated RH does not seem to significantly influence cementitious composites produced with RH. To understand the effect caused by drying/ wetting cycles, it was necessary to compare the resistance values obtained in this assay with the results of the compressive and bending strength tests at 90 days. Through this comparison, it was possible to conclude that the compressive and bending strengths of the composites, after drying and wetting cycles, decrease more markedly in composites with RH subject to pretreatment. This result is due to the fact that RH presents strong dimensional variations when it passes from the dry state to the saturated state, and vice versa (as previously analyzed) and this dimensional variation increases with the addition of the pretreatment concentration. Thus, it was concluded that in these composites there are more tensions between the paste and the pretreated RH, forming more microcracks, which lead to a reduction in the properties of the composites (Table 4.4). It should be noted that, in terms of absolute results of mechanical resistance after drying/ wetting cycles, composites with pretreatment RH still have higher compressive and bending strength values than the reference composite.

Table 4.4 – Results achieved in durability tests performed on cementitious composites.

Composite testing	C_Ref	C_S3%	C_S6%	C_S9%
Open porosity at 28 days according to EN 1936 and RILEM I.1 [%]	46,9	46,1	45,7	46,3
	-	-2%	-3%	-1%
Variations in compressive strength in composites due to drying/weting cycle [%]	-53,9	-55,5	-58,0	-58,5
	-	3%	8%	9%
Variations in bending strength in composites due to drying/wetting cycle [%]	-54,3	-55,7	-59,9	-60,1
	-	3%	10%	11%

Legend: MED – dynamic modulus of elasticity; US – ultrasound velocity; Green – positive variation greater than 2%; Red – negative variation greater than 2%.

4.4 PRETREATMENT OF SODIUM SILICATE VS SODIUM HYDROXIDE

4.4.1 Influence of pretreatment on RH

Table 4.5 shows the average dimensions of RH treated with dry and saturated NH and their average ratios between length (L_1) and width (L_2). This table shows that pretreatment with NH produces morphological variations in RH higher than those observed in the treatment with NS. Thus, it can be predicted that the losses of mechanical resistances, after drying and wetting cycles, will be much higher in composites with RH subject to pretreatment with NH, since the state variations, in principle, will lead to more microcracks in these composites.

Table 4.5 – Dry and saturated RH ratios after pretreatment with NH and NS.

Pretreatments	Pretreatment of NH	Pretreatment of NS
	Variation between RH states (%)	Variation between RH states (%)
RH_Ref	-6,55	-1,82
RH_3%	-46,36	-7,41
RH_6%	-52,96	-8,56
RH_9%	-35,88	-10,00

Legend: RH_Ref – Rice husk of reference; RH_3% - pretreated rice husk with 3%; RH_6% - pretreated rice husk with 6%; RH_9% - pretreated rice husk with 9%.

4.4.2 Influence of pretreatment on pastes

Through the analysis of Figures 4.2 and 4.3, it is possible to verify that the NH and NS produce increases in the compressive strength of very similar pastes (maximum increases of 12% and 11%, respectively). On the contrary, the increases in bending resistance, when using these different treatments, are quite different.

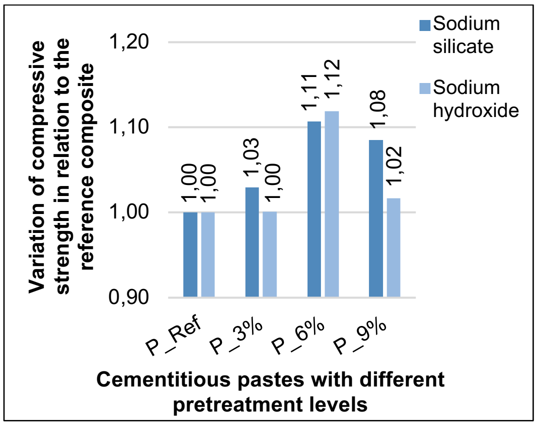

Figure 4.2 – Variation of compressive strengths in the pastes at 28 days with pretreatments of NS and NH.

Pretreatment of sodium hydroxide (NH) leads to an increase in the bending strength of the pastes between 17% and 70%, while sodium silicate (NS) only produces an increase between 4% and 16% (Figure 4.3).

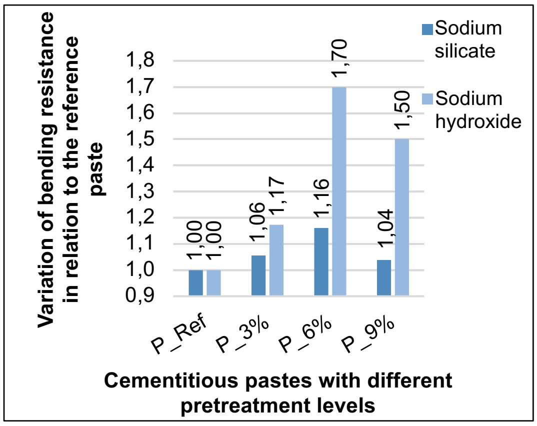

Figure 4.3 – Variation of bending resistance in the pastes at 28 days with pretreatments of NS and NH.

4.4.3 Influence of pretreatment on composites

To conclude the present comparative analysis between the pre-treatments of NS and NH, a comparison is made of the composites produced with RH subject to these pre-treatments, evaluating the characteristics in the fresh state (volume mass) and in the hardened state (compressive and bending resistance).

As mentioned earlier, pretreatment with NH produces changes in the dimensions of RH much higher than those caused by NS. Therefore, the changes in the original ratios used in the composition of the pastes will also be much higher than those observed with the use of NS. Table 4.6 shows the different ratios before and after pretreatment, highlighting that the ratios after pretreatment are the true ratios of composites produced.

Table 4.6 – Paste/ RH ratios of cementitious composites with RH with pretreatment of NH and NS.

Mixtures	**Paste/ RH ratios**			
	NS		**NH**	
	Theoretical	**Real**	**Theoretical**	**Real**
C_Ref	8,07	7,96	8,07	8,22
C_3%		9,97		15,32
C_6%		8,79		13,88
C_9%		9,14		15,21

Legend: C_Ref – composite of reference RH; C_3% – composite of RH pretreated with 3%; C_6% – composite of RH pretreated with 6%; C_9% - composite of RH pretreated with 9%; NS – sodium silicate; NH – sodium hydroxide.

Figure 4.4 allows a real perception of the different cementitious composites produced with 6% pretreatment. The figure proves that the composite with pre-treatment of NH presents a much higher amount of cementitious paste than the composite with pre-treatment of NS.

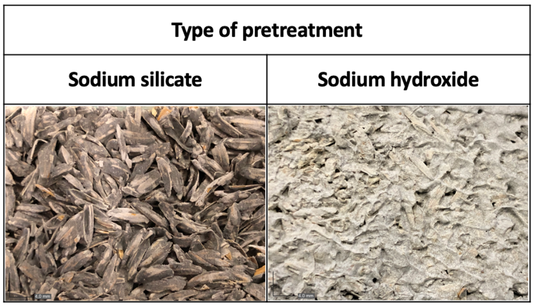

Figure 4.4 – Composites with 6% pretreatment of NS and NH.

To compare the effects of the different pretreatments on the mechanical behavior of the composites, the compressive and bending resistances were analyzed. Figures 4.5 and 4.6 represent the variation that exists between the resistances of the reference composite and the resistances of composites with RH subject to different pretreatments. Thus, it is possible to observe that the pretreatment with NH causes increases in resistances much higher than those caused with the pretreatment of NS.

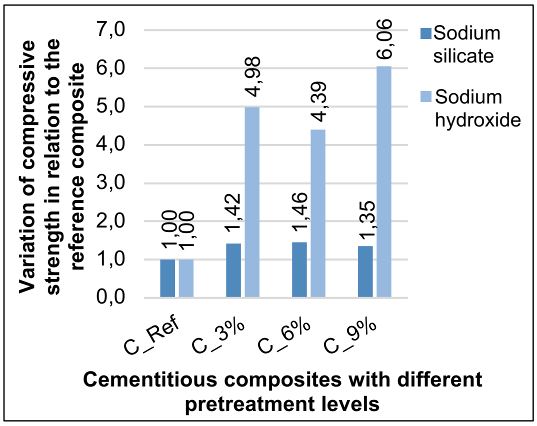

Figure 4.5 – Variation of compressive strength in composites with different RH pretreatments at 28 days.

The results obtained can be explained by the actual paste/RH ratios existing in the various composites. As mentioned, the actual paste/RH ratios of composites with pretreatment of NH are much higher than those of composites with NS pretreatment.

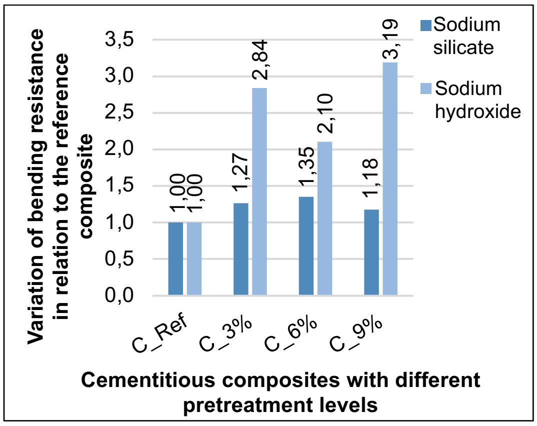

Figure 4.6 – Variation of bending resistance in composites with different RH pretreatments at 28 days.

5. CONCLUSIONS

This master's thesis aimed to analyze cementitious composites with rice husk (RH) previously subject to a pretreatment of sodium silicate (NS). Therefore, it was intended to evaluate the effect caused by different concentrations of this pre-treatment in RH and, consequently, on cementitious composites produced with this residue. This study followed a previous investigation, which consisted of the analysis of cementitious composites with RH pretreated with sodium hydroxide (NH). Thus, in this dissertation, a comparison was also made between the effects caused by the two pre-treatments on the use of RH in cementitious composites.

Finally, it was concluded that the use of pretreatment with NH seems to provide the production of composites with RH with better mechanical behaviors, both in comparison with the reference composites, as in comparison with composites with RH pretreated with NS. On the contrary, although no evaluation tests have been carried out on drying/ wetting cycles under the same conditions, everything indicates that pre-treatment with NH causes greater mechanical reductions after drying/wetting cycles, since the pretreated with NH presents much greater dimensional variations when it passes from the dry to the saturated state, and vice versa. Thus, it was concluded that the use of both pre-treatments can be very beneficial for the performance of cementitious composites with RH and that the choice of preferential pretreatment should be made according to the type of composite desired for a given application.

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