

Valorization of lignin residue in mortars and concretes

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Abstract

The possibility of application for lignin, a byproduct of paper and pulp production, as a partial replacement for cement in producing mortars and concretes is presented. Initially, mortars were prepared with substitution rates ranging from 0.0% to 50.0% to determine the best rates for subsequent application in concretes. Statistical analysis showed that the rates with the highest potential for application in concretes were 2.5%, 5.0%, and 10.0%. After preparing the concretes, test specimens were molded and subjected to axial compression, splitting tensile strength, water absorption by immersion, void ratio, and capillary water absorption tests after the curing period. The results showed that lignin, in rates higher than 5.0%, led to decreased mechanical strength; however, all concretes with lignin significantly reduced capillary water absorption, which can lead to greater material durability.

Keywords: concrete, lignin, sustainability, environment, reuse of waste.

INTRODUCTION

With population growth, there is also an increase in the extraction of raw materials to produce various products consumed by humanity, which increases waste generation. These wastes can cause negative environmental impacts if not correctly disposed of. In addition to the growth in the production chain, population growth also leads to an increase in the demand for housing and infrastructure, fostering the construction industry, known for its high consumption of raw materials and generation of waste. However, several studies indicate that the construction industry can absorb waste from other sectors, such as the use of sludge from water treatment plants as fine aggregate for concrete production [1], the incorporation of waste from the processing of ornamental stones for concrete production [2], and the ability to reuse waste generated by the construction industry [3].

The pulp and paper industry uses wood to obtain its products. Wood undergoes factory transformations, and lignin is extracted as a byproduct. According to Tribot et al. [4], lignin is a natural polymer found in plants, composed of three units of phenylpropane (coniferyl alcohol, sinapyl alcohol, and small amounts of p-coumaryl alcohol). According to Bajwa et al. [5], lignin is the planet's second most abundant complex organic material. Usually, lignin has been treated as a low-value waste. Still, studies indicate that lignin can be used in high-value products such as carbon fiber, phenolic compounds, and multifunctional hydrocarbons. According to the authors, the pulp and paper industry is one of the primary sources of lignin production, producing 50 to 70 million tons per year

worldwide. Although studies point to the possibility of higher-value utilization of lignin, some authors [5, 6] explain that about 98% of all lignin produced is used as fuel for heating and energy generation within the factory. Within the construction industry, Kun and Pukánszky [6] mention that lignosulfonates are already widely used to produce concrete dispersants, which reduce the amount of water while ensuring workability and promoting faster strength development. Calado et al. [7] studied the possibility of using lignin as a partial substitute for cement in the production of self-compacting concrete and concluded that this substitution could be viable up to a limit of 5.0%. The authors said that lignin had a retarding effect on cement setting and showed good interaction with plasticizing and superplasticizing additives. Bajwa et al. [5] explain that low levels of lignin and modified lignin can be used to produce high-performance concrete, which leads to increased strength, facilitates finishing, and protects the material from moisture. They also mention that the sulfonation process of lignin allows it to act as a dispersant in the cement matrix.

This work aims to evaluate the influence of lignin waste incorporation on consistency, setting times, compressive strength, water absorption, and capillary water absorption of mortars and concrete. The use of lignin waste without treatment (as received) in mortars and concrete is shortly explored in the literature and is a novelty of this study. In this context, the present work contributes to studies that use kraft lignin waste without any treatment in the production of mortars and concrete by partially replacing cement.

EXPERIMENTAL

Initially, the influence of lignin incorporation on compressive strength in mortars after a 28-day curing period was studied with substitution levels of cement by lignin

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of 0.0%, 2.5%, 5.0%, 10.0%, 20.0%, 30.0%, 40.0%, and 50.0%, aiming to identify the limit for potential application in concrete. To produce the mortars, CP-II-Z-32 cement, Kraft lignin, quartz sand commonly found in construction materials stores, and water from the public supply system (Votuporanga/SP, Brazil) were used. The lignin waste was donated by a pulp and paper company from São Paulo/Brazil. This waste was obtained from the Kraft process and was used as received without any treatment. The quantities of materials used to produce the mortars are shown in Table I. Based on the compressive strength results in the mortars and the statistical analysis of the data, the substitution levels with the highest potential for application in concrete were defined. For concrete mix design, it was necessary to characterize the materials.

Table I - Mass (g) of materials used to prepare mortars.

Material	M0.0	M2.5	M5.0	M10	M20	M30	M40	M50
Cement	320	312	304	288	256	224	192	160
Lignin	0	8	16	32	64	96	128	160
Sand	960	960	960	960	960	960	960	960
Water	160	160	160	160	160	190*	220*	220*

* more water was necessary to ensure the molding conditions.

Characterization of materials for concrete production: to produce concrete, CP-II-F-32 cement and the same sand and water used in mortars were used. Crushed stone from construction materials stores (Votuporanta/SP) was used as coarse aggregate. The characterization tests for the materials are listed in Table II. For the characterization of lignin, optical microscopy (DM750M, Leica) and particle size analysis by dynamic light scattering (DLS) with capillary cells (Nano Zetasizer ZS, DST 1060, Malvern Instr.) were performed. The setting time test was conducted with two water quantities. The first quantity resulted in a water-to-cement (w/c) ratio of 0.268; the control mix and the mix with 2.5% lignin were tested. The second quantity had a w/c ratio of 0.373, allowing testing of all four mixtures (0.0%, 2.5%, 5.0%, and 10.0%). This procedure was necessary because the mixture with 5.0% lignin became excessively dry and lumpy, making it impossible to conduct the test with the first w/c ratio.

Table II - Material characterization tests.

Parameter	Ref.
Compacted and loose bulk density of aggregate	[8]
Specific gravity of fine aggregate	[9]
Specific gravity of coarse aggregate	[10]
Particle size distribution of aggregate	[11]
Specific gravity of cement	[12]
Initial and final setting time of cement	[13]

Concrete characterization: after characterizing the materials, the concrete was designed using the ABCP method [14] for a characteristic compressive strength of 35 MPa

and a slump of 120 mm. The mass proportion of materials used for concrete preparation was 1.000:0.916:2.271:0.393 (cement:sand:crushed stone:water) with a cement consumption of 535.0 kg/m³. The substitutions of cement with lignin were carried out relative to the cement mass, with contents of 0.0% (control), 2.5%, 5.0%, and 10.0%. After the preparation of the concrete, 15 specimens were molded according to ABNT NBR 5738 standard [15]. Then, the test specimens underwent a 28-day curing period, during which they were immersed in water saturated with lime. Next, they were evaluated according to the parameters and references presented in Table III.

Table III - Concrete characterization tests.

Parameter	No. of specimens	Ref.
Slump test	After mixing	[16]*
Compressive strength	6	[17]
Splitting tensile strength	6	[18]
Water absorption by immersion and void index	3	[19]
Water absorption by capillary	3**	[20]

* applicable at the time; ** same specimens as the water absorption by immersion test.

Statistical analysis of the results: analysis of variance (ANOVA) and Tukey’s test were conducted to aid in the discussions and conclusions, both with a confidence level of 95%.

RESULTS AND DISCUSSION

Fig. 1 presents the compressive strength and standard deviation of the studied mortar specimens. The mortar with 50% lignin destabilized during curing and was not tested. It was observed that as the substitution content of cement by lignin increased, the compressive strength decreased. To verify if the differences were significant, an ANOVA was applied, and the result is presented in Table IV. It was observed that differences were significant, so the Tukey’s test was performed, and grouping is shown in Fig. 1. The Tukey’s test confirmed that lignin content decreased the compressive strength of studied mortars. After the statistical

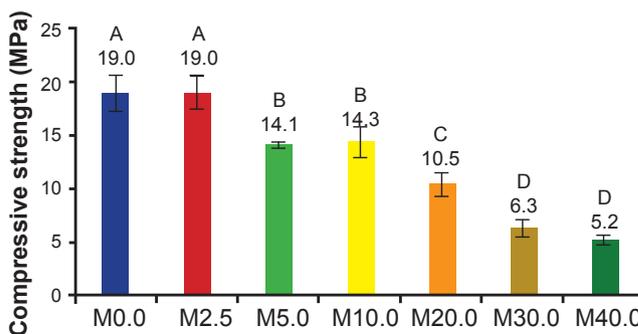


Figure 1: Compressive strength and standard deviation of mortars. Values followed by the same letter are statistically similar (p>0.05).

Table IV - Results of ANOVA for the compressive strength data of the mortars.

Source	DF	SS	MS	F	p-value
Material	6	555.612	92.602	70.959	0.000
Error	14	18.270	1.305		
Total	20	573.883			

analysis of the compressive strength of the mortars, the substitution contents identified as having the highest potential for application in concrete were 2.5%, 5.0%, and 10.0%.

Table V presents the specific masses of the aggregates and cement and important dosing parameters determined by the particle size analysis. Figs. 2a and 2b show the granulometric curves of sand and crushed stone, respectively. Analyzing Fig. 2a and comparing it with the granulometric distribution proposed in the ABNT NBR 7211 standard [21], it was concluded that the sand used was in the lower usable

Table V - Characteristics of the materials used.

Parameter	Sand	Crushed stone	Cement
Loose bulk density (kg/m ³)	1583.8	1560.9	-
Compacted bulk density (kg/m ³)	1665.1	1664.0	-
Specific gravity (kg/m ³)	2647.9	2933.1	3061.2
Fineness modulus	2.18	6.79	-
Maximum diameter (mm)	2.40	19.00	-

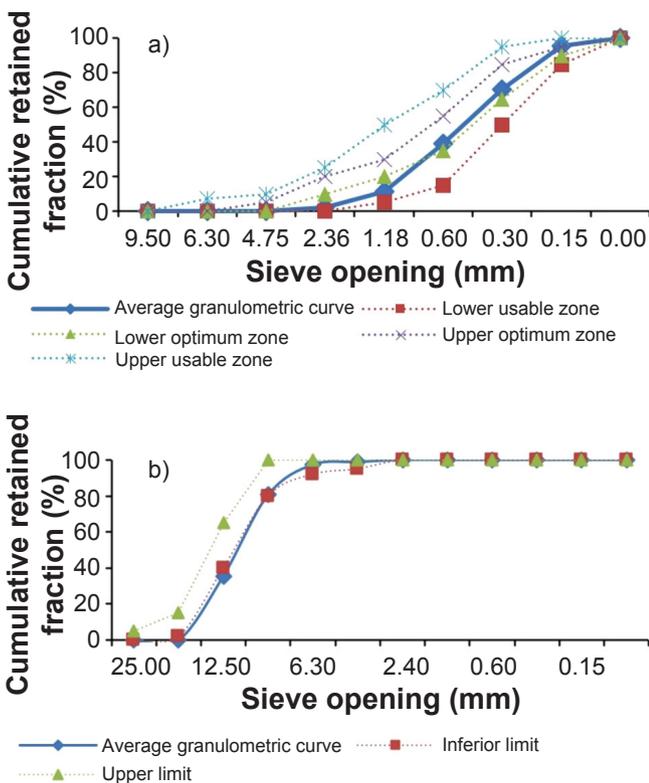


Figure 2: Granulometric curves of sand (a) and crushed stone (b).

zone. The analysis of Fig. 2b shows that the granulometric distribution of crushed stone was closer to the inferior limit, and your fineness modulus was classified in the 9.5/25 zone, according to ABNT NBR 7211 [21].

Initially, for the setting time test, two w/c ratios were determined. The w/c ratio 1 was 0.268, and the w/c ratio 2 was 0.373. Control pastes were produced using 500 g of cement. The pastes containing lignin were produced by replacing percentages of the cement mass at 2.5%, 5.0%, and 10.0%. The water quantity for w/c ratio 1 was insufficient to allow the setting time test to be conducted on the P5.0 because this mixture became too dry and lumpy. And for this reason, the mixture P10.0 was not tested. Analyzing Table VI, it is evident that the incorporation of lignin impacted the initial and final setting times, increasing them by approximately 2.5 times when w/c ratio 1 was used, and nearly 2.0 times with w/c ratio 2. These initial and final setting time increases were consistent with those obtained by Calado et al. [7], suggesting that lignin acted as a setting retarder. El-Mekkawi et al. [22] studied the use of black liquor as a plasticizer, also classified as a retardant. The authors added 30.0% of LBA (lignin-based additives), replacing the volume of water, and found that the initial and final setting times increased by 223.0% and 258.0%, respectively. They attributed this retardant effect of black liquor to the high sugar content of its composition. According to Table VI, it was noticed that the initial setting time of the P10.0 had minor alteration when compared to the P0.0. Still, the final setting time of the P10.0 became closer to those obtained by the other two mixtures containing lignin. To try to explain this phenomenon of accelerated setting time observed in the paste with 10% lignin compared to the other lignin-containing pastes, a reference was made to the work of Souza et al. [23]. In this study, the accelerating effect of high concentrations of sucrose was investigated. It was concluded that when the sucrose concentration is above a critical level, it promotes the formation and stabilization of ettringite in the early hours of cement hydration, which reduces the setting time. Kochova et al. [24] compared the effect on the setting time of various organic compounds present in lignocellulose fibers and concluded that lignin has a retarding effect. In this case, the retardation was lower than with sugars. Possibly because of this, when a 10% lignin content was used, the initial setting time approached that of the control paste. In future work, it is suggested to evaluate ettringite formation in the first hours of hydration in pastes with different contents of lignin.

Table VI - Initial and final times of setting of pastes.

Paste	w/c ₁ =0.268		w/c ₂ =0.373	
	Initial (h)	Final (h)	Initial (h)	Final (h)
P0.0	02:30	03:50	04:00	04:50
P2.5	06:05	10:15	07:38	10:33
P5.0	-	-	08:38	10:37
P10.0	-	-	04:32	10:15

Regarding the particle size of lignin, Fig. 3 illustrates the result of optical microscopy and particle size distribution by DLS. Analyzing the micrograph, it was observed that the particles had diameters smaller than 50 μm. The DLS analysis resulted in an average diameter of 2.26±0.27 μm, indicating the possibility of being a material with a high specific surface area.

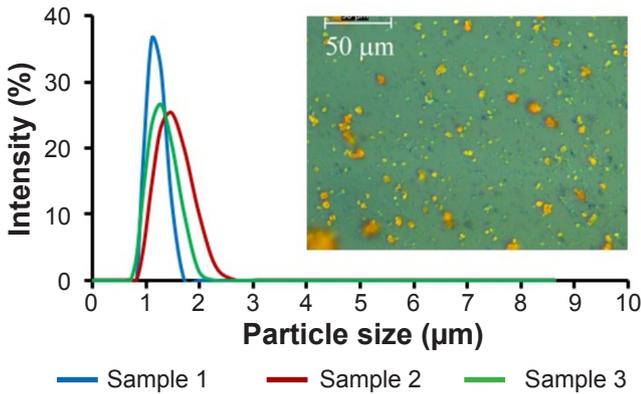


Figure 3: Particle size distribution curves by DLS and optical microscopy image of the lignin.

Table VII - Slump (mm) of the studied concrete mixes.

C0.0	C2.5	C5.0	C10.0
120.0	110.0	55.0	20.0*

* additional water was added (initial slump: 5 mm).

After the preparation of the concrete mixes, the slump test was conducted to verify the consistency, and the results are presented in Table VII. It was possible to perceive that

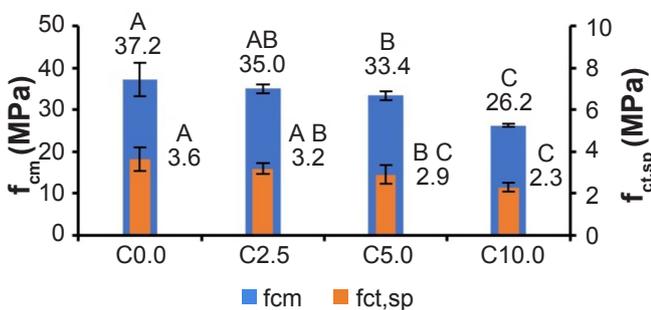


Figure 4: Compressive strength (f_{cm}), splitting tensile strength ($f_{ct,sp}$), and their respective standard deviations of the studied concretes. For each property, values followed by the same letter are statistically similar ($p > 0.05$).

lignin altered the consistency of the concrete, making it drier. This fact was also observed during the preparation of the pastes for the cement’s initial and final setting time test. Calado et al. [7] conducted spreading tests on mortars and commented that the spreading performance of mixtures with lignin was slightly inferior to those without lignin.

The results of compressive strength (f_{cm}) and splitting tensile strength ($f_{ct,sp}$), with their respective standard deviations are presented in Fig. 4. In line with the behavior of the mortars, increasing the lignin content by substituting cement led to a decrease in both compressive and splitting tensile strength. ANOVA was conducted, and the result presented in Table VIII shows that lignin influenced the mechanical strength. Tukey’s test was applied to highlight which concretes differed, and the grouping is shown in Fig. 4. It can be observed that the incorporation of lignin decreased the mechanical strength. Regarding compressive strength, it was possible to identify that the lignin content affected the compressive strength behavior, as the lignin content was increased, compressive strength decreased. The concrete C2.5 did not show a significant reduction in compressive strength compared to the concrete C0.0 (control), as seen in the grouping after applying Tukey’s test (Fig. 4). The concrete C5.0 showed a significant difference compared to concrete C0.0. However, it exhibited statistically similar behavior to concrete C2.5 (Fig. 4), indicating the feasibility of using this lignin content. For the concrete C10.0, it was necessary to use more water to allow for the molding of the specimens, resulting in an increased w/c ratio from 0.393 to 0.427. Using the Abrams’ law curve for cement available in [14] for this new w/c ratio, an expected characteristic compressive strength of 32 MPa was estimated. However, making a direct comparison between the average strength obtained by concrete C10.0 (26.2 MPa) and the expected characteristic strength after changing the water/cement ratio (32 MPa), the reduction was approximately 18%, which indicated that substitutions with 10% or higher content are not recommended unless mechanical strength is not the predominant factor for concrete utilization.

Regarding the tensile strength, the test used in this study indirectly determined the tensile strength through the diametral compression of the specimens. The relation established in ABNT NBR 6118 standard [25] was used to compare tensile strength to compressive strength. Direct tensile strength (f_{ct}) can be estimated in the function of splitting tensile strength ($f_{ct,sp}$), and mean direct tensile strength ($f_{ct,m}$) can be calculated using the characteristic compressive strength (f_{ck}); in this case, the average

Table VIII - ANOVA results of the compressive and splitting tensile strength data.

Source	Compressive strength					Splitting tensile strength				
	DF	SS	MS	F	p-value	DF	SS	MS	F	p-value
Material	3	406.035	135.345	29.418	0.000	3	5.766	1.922	12.106	0.000
Error	20	92.016	4.601			20	3.175	0.159		
Total	23	498.051				23	8.941			

compressive strength (f_{cm}) was used. The superior ($f_{ctk,sup}$) and inferior ($f_{ctk,inf}$) limits of direct tensile strength were calculated from the mean direct tensile strength, using the following equations:

$$f_{ct,m} = 0.3 \cdot f_{ct}^{2/3} \tag{A}$$

$$f_{ctk,inf} = 0.7 \cdot f_{ct,m} \tag{B}$$

$$f_{ctk,sup} = 1.3 \cdot f_{ct,m} \tag{C}$$

$$f_{ct} = 0.9 \cdot f_{ct,sp} \tag{D}$$

The results are presented in Table IX, and analyzing this table and adopting the theoretical mean tensile strength ($f_{ct,m}$) as the target value to be achieved, the concrete that came closest was the C0.0. It can be observed by the relation $f_{ct}/f_{ct,m}$ that the higher the lignin content in the mixture, the greater the difference found. However, it can also be observed, specifically within the range of tensile strength delimited by the values of $f_{ctk,inf}$ and $f_{ctk,sup}$, that all the concretes had tensile strengths (f_{ct}) within this range.

The graph in Fig. 5 presents the mean results and standard deviations obtained in water absorption tests by immersion and voids index. Differences were observed in the studied parameters. ANOVA was applied to determine if the differences were statistically significant. The analysis result is presented in Table X, and the lignin did not interfere with the material’s porosity. It should be emphasized that, according to Neville [26], good concretes have absorption rates lower than 10%. Therefore, all the concretes studied in this work can be classified as good concretes regarding water absorption.

Finally, Fig. 6 presents the results of capillary water absorption after 72 h of testing. It was observed that as the amount of lignin in the mixture increased, capillary water absorption decreased. To determine if the influence of lignin on this reduction was significant, an ANOVA followed by

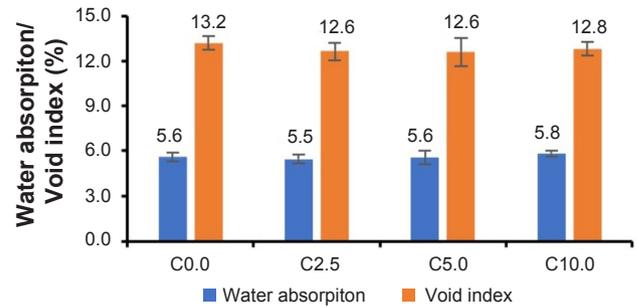


Figure 5: Water absorption by immersion, void index, and their respective standard deviations of the concretes.

Tukey’s test was applied. The results are listed in Table XI, and grouping can be found in Fig. 6. The ANOVA showed that the differences were significant, indicating that lignin had a direct influence on reducing capillary water absorption. Although lignin did not influence the voids in the concretes studied, it reduced capillary absorption. Neville [26] explains that pore volume is measured by absorption and is different from the ease with which fluids can penetrate the concrete. The author also comments that there is not necessarily a relationship between these parameters. According to Zhao et al. [27], capillary absorption is characterized by the transport of water in porous materials through the action of capillary forces present in these pores. Carvalho and Motta [28] explain that the diameter of the pores influences capillarity and note that the smaller the pores, the higher the capillary pressures, resulting in greater water rise. However, when the pore diameters are large, capillary pressures are lower, which leads to reduced capillary rise, but the volume of water absorbed is higher. The authors emphasize that the interconnectivity between pores has a significant impact on the behavior of capillary absorption. Wang et al. [29] comment that differences in the pore structure lead to variations in the capillary absorption capacity of the material. Zhao et al. [27] also explain that capillary absorption is related to the pore structure of concrete. Thus,

Table IX - Theoretical direct tensile strength based on splitting tensile and compressive strength.

Concrete	Strength (MPa)						$f_{ct}/f_{ct,m}$ (%)
	f_{cm}	$f_{ct,m}$	$f_{ctk,inf}$	$f_{ctk,sup}$	$f_{ct,sp}$	f_{ct}	
C0.0	37.23	3.34	2.34	4.35	3.64	3.28	97.94
C2.5	34.99	3.21	2.25	4.17	3.18	2.86	89.18
C5.0	33.39	3.11	2.18	4.04	2.90	2.61	83.90
C10.0	26.24	2.65	1.85	3.44	2.28	2.05	77.46

Table X - ANOVA results of the data obtained from the water absorption by immersion and void index of the studied concretes.

Source	DF	Water absorption by immersion				Void index				
		SS	MS	F	p-value	DF	SS	MS	F	p-value
Material	3	2.24E-5	7.46E-6	0.762	0.547	3	6.51E-5	2.17E-5	0.540	0.668
Error	8	7.84E-5	9.79E-6			8	3.22E-4	4.02E-5		
Total	11	1.01E-4				11	3.87E-4			

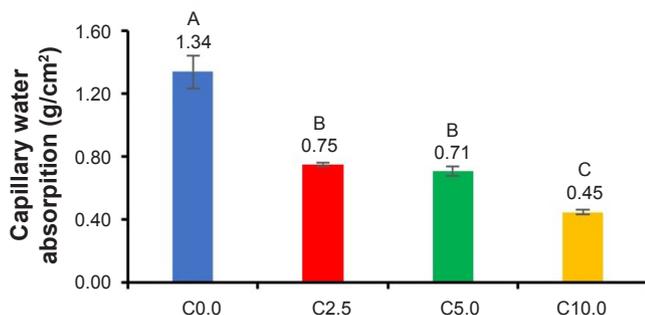


Figure 6: Capillary water absorption after 72 h of testing of the concretes. Values followed by the same letter are statistically similar ($p > 0.05$).

Table XI - ANOVA results of the data obtained from the capillary water absorption after 72 h of testing of the studied concretes.

Source	DF	SS	MS	F	p-value
Material	3	1.28	4.26E-1	135.977	0.000
Error	8	2.50E-2	3.00E-3		
Total	11	1.30			

lignin may have influenced the pore structure characteristics of concrete, reducing the capillary absorption of the studied concretes. Lower capillary absorption rates can indicate an increase in the durability of concrete. Analyzing Fig. 6, it is evident that the concretes C2.5 and C5.0 behaviors were similar, which is consistent with their behavior in the other analyzed parameters, once again indicating that substitution up to a 5% content is viable.

CONCLUSIONS

The incorporation of lignin has a negative impact on the consistency of pastes, mortars, and concretes. Therefore, it is necessary to use plasticizing and/or superplasticizing additives to ensure the fluidity of the mixture. Lignin affected the initial and final setting times, indicating a retarding action. This behavior may be advantageous in cases where the concreting operation requires a longer time or when there is a significant distance between the preparation and application. Regarding mechanical strength, it was observed that with low lignin contents (up to 2.5%), the concrete did not experience a significant decrease in strength when compared with the control concrete. With a 5% content of lignin, the behavior of this concrete was similar to concrete C2.5 in all parameters analyzed. There was no significant difference in water absorption or void index across all studied lignin contents. This suggested that lignin, being a finely ground material, did not interfere with these parameters. For the capillary water absorption, a decrease in the absorption rate was observed as the lignin content increased. In this case, lignin may have influenced the pore structure characteristics of concrete, reducing the capillary absorption of the samples. Higher lignin contents,

in the range of 10%, may be possible in situations where high mechanical strength is not required but the structure is in contact with corrosive liquid agents. Finally, based on the results obtained and the statistical analyses conducted, limiting the substitution of cement with lignin to 5.0% is feasible to maintain mechanical strength while achieving better performance in capillary water absorption.

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