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Influence of Mercerization on the Physical and Mechanical Properties of Polymeric Composites Reinforced with Amazonian Fiber

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Abstract: Natural fiber has been an attractive alternative as reinforcement in polymeric composites. However, their physical properties can be impaired due to the hydrophilic behavior of the vegetal fibers: the weak bonds at the interface fiber/matrix emerge due to the hydrophobic behavior of some polymeric matrices. This paper aims to evaluate the influence of mercerization process in *Leopoldinia piassaba* fibers on the physical, mechanical, thermal, morphological, and accelerated aging properties of polyurethane (PU)-based composites produced by the thermoforming process. The experimental design (2^{2+1}) was applied considering the parameters NaOH Treatment and %(PU)Resin. The NaOH Treatment consisted of 5 % and 10 % NaOH solution for fibers mercerization, and %(PU)Resin consisted of different amounts of PU (15 %, 17.5 % and 20 %). The mercerization process chemically modified the interface fiber/matrix and improved the resistance of the composite to screw withdraw strength, ranging from 573 N to 1019 N, and from 574 N to 1648 N considering Surface and Top, respectively. According to Scanning Electron Microscopy (SEM) analysis, the mercerization process also improved the fiber/matrix adherence and reduced pores, resulting in swelling decreasing and thermal conductivity increasing. Furthermore, the mean thickness swelling (TS) value ranged from 6 to 15 % and was not statistically different after accelerated aging. The developed composites containing untreated fibers showed thermal conductivity values similar to the commercial medium density fiberboard (MDF).

Keywords: *Leopoldinia piassaba* fibers, Castor oil, Mercerization, Polymeric composites, Screw withdraw strength

Introduction

Natural fibers have been an interesting alternative as reinforcement in polymeric composites due to their abundance, renewability and low cost [1-3]. Their use represents a great potential to develop sustainable composites based on natural fibers and bio-resins [4], contributing to reduce the environmental impacts from synthetic materials [5]. Natural fiber-reinforced composites present low density, higher deformability, and biodegradability [6], contributing to reduce the use of synthetic fibers and the exploitation of raw materials and/or energy sources from non-renewable resources [4,7,8]. Furthermore, natural fibers have been used as reinforcement in polymeric composites based on a range of polymeric matrices such as polyethylene, epoxy, and polyester [9-14].

Brazil is rich in renewable resources and has a wide variety of natural fibers such as the Amazonian piassava (*Leopoldinia piassaba*). This fiber is mainly found in Barcelos, municipality of Alto Rio Negro, in the Amazonas State. The *L. piassaba* fibers are soft, flexible, elastic, and commonly used in the manufacture of brushes, brooms, handicrafts, as well as rustic roof [8,15].

The hydrophilic behavior of lignocellulosic fibers compromises the matrix/reinforcement adherence, resulting in weak bonds at the interface, as well as in poor mechanical properties. The matrix/reinforcement interface plays a significant role in these properties due to the load transfer from the polymer to the fiber [16,17]. In this context, the chemical modification of the fiber's surface is important to improve matrix/reinforcement bonds. For this reason, the mercerization process proposed here was based on 5 % and 10 % NaOH solutions due to its low cost and effectiveness in the interaction polymer/fiber. Moreover, in our previous work [29], this mercerization process modified the morphology and dimensions of the *L. piassaba* fibers, reducing the cell wall thickness from 3 μm to 1 μm. The percentage of crystallinity was also increased and attributed to the removal of organic materials from fibers [13,21,29]. Some reports also revealed that the mercerization process can increase the roughness of the fiber surface and reduce the porosity of the composites [8,16,18-20]. However, the

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defibrillation of fibers can be observed depending on the methodology employed in this chemical treatment, affecting mechanical properties [22-24].

The goal of this work was to evaluate the effect of the mercerization process in *L. piassaba* fibers on the physical, thermal, and mechanical properties of polymeric composites. The castor oil-based PU (*Ricinus communis*) was used here as composite matrix due to its partially renewable resources. To the best of our knowledge, no reports have been found in scientific literature considering the mercerization process applied to this Amazonian fiber, as well as its application as reinforcement in PU-based composites. Accelerated aging testing was performed to simulate the indoor exposure conditions of high levels of temperature and humidity of the samples. Then, the influence of the mercerization process on the swelling properties of the developed composites was evaluated. Finally, thermal properties were analyzed by the conductivity test in order to verify the insulating properties of the developed composites.

Experimental

Materials

L. piassaba fibers were supplied by O.A. Nunes Neta-ME, Manaus-AM. NaOH (ACS reagent, ≥ 97.0 %, pellets) was purchased from Nuclear™. Polyurethane resin based on castor oil (Polyol: LECOPOL F 0911 and Isocyanate: LECOPOL E 0921) was donated by Plural Indústria e Comércio de Produtos Químicos Ltda., São Carlos-SP, Brazil.

Methods

Mercerization

L. piassaba fibers (~1 cm length) were added to 5 % and 10 % NaOH solutions for 60 min [29]. The excess of NaOH content was removed using distilled water until the pH of washings reached neutrality. Then, fibers were dried in a conventional oven at 60 °C for 6 h to reach approximately 10 % humidity [24].

Composites Production

The developed composites were produced by the thermoforming process. PU resin was manually added to the *L. piassaba* fibers for 20 min for mixing. Table 1 describes the parameters and levels used for two-level factorial design experiment considering 5 % or 10 % NaOH treatments and the percentages of PU for each developed composite. Then, the resulting material was inserted into a wooden mold (40× 40×1) cm^3 and subjected to 15 MPa on a hydraulic press (Model PHH 11007, Hidral-Mac®) at 100 °C for 10 min [30]. Figure 1 shows the developed composite based on *L. piassaba* fibers and PU resin.

Table 1. Parameters NaOH treatment and %(PU)resin considered in the two-level factorial design experiment

Composite	NaOH treatment	%(PU)resin	Level
15ST	-	15	(-1 -1)
20ST	-	20	(-1 +1)
17.5 CT*	5 % NaOH	17.5	(0 + 0)
15CT	10 % NaOH	15	(+1 -1)
20CT	10 % NaOH	20	(+1 +1)

ST (untreated fibers), CT (treated fibers); composites labeled using the numbers 15, 20 and 17.5 represent 15 %, 20 % and 17.5 % in mass, respectively. *Central bottom (triplicate).

Figure 1. Composite based on *L. piassaba* fibers and PU resin.

Five types of medium density panels were produced based on the experimental design (2^{2+1}) [25]. The parameters NaOH Treatment (5 % and 10 % NaOH) and "%(PU)Resin" (15 %, 17.5 % and 20 %) were considered in the applied two-level factorial design experiment, as shown in Table 1.

Accelerated Aging Testing (AAT)

AAT was performed to simulate the sample indoor exposure conditions (high levels of temperature and humidity) based on the methodology adapted from the APA Standard - Standard Accelerating Test [27]. The developed composites were subjected to the ultraviolet (UV) radiation on an EQUV aging chamber for 8 cycles, each cycle is represented by 8 h of UV radiation (0.49 radiance at 60 °C) and 4 h condensation at 50 °C [31]. Then, the composites were tested by thickness swelling.

Density and Thickness Swelling (TS)

The measurements for density and thickness swelling were performed according to the Brazilian Standard NBR 14810-2:2018 [26]. All samples were prepared with dimensions of (5.0×5.0×1.0) cm^3, following the recommendations of 14810-2:2018 Brazilian standard [26]. The composite density was calculated using the following equation (equation (1)):

$$\text{Density } (\rho) = \frac{Mass(m)}{Volume\ (V)} \quad (1)$$

The TS test was applied to measure the swelling of the developed composites. Before the composites were soaked into the distilled water at 23 °C, the thickness of every sample was measured, and each sample was labeled. After

24 h, the composite was taken out and dried before it was measured. The thickness value of the samples was taken using a Mitutoyo digital micrometer. The TS test was continued for several days until constant thickness values were obtained. The TS calculation is shown in equation (2):

$$\text{Thickness swelling (\%)} = \frac{T1 - T0}{T0} \quad (2)$$

where $T1$ and $T0$ represent the thickness after and before soaking, respectively.

Thermal Conductivity (TC)

Composites were cut into two identical (5.0×5.0×1.0) cm^3 pieces for use in the guarded hot plate TC test on a TCi C-Therm equipment. The TC values were obtained according to the ASTMC177 [28] standard set method ranging from 0.0 to 500 W/mK. Testing temperatures were considered between -50 °C and 200 °C.

Scanning Electron Microscopy (SEM)

SEM experiments were performed on a Hitachi TM3000 equipment, using 15 kV at 25 °C. Samples were placed on a carbon tape and recovered with a thin gold layer prior to analysis aiming to improve the contrast and maximum resolution [29].

Mechanical Properties

To evaluate the influence of the mercerization process on the mechanical properties of the developed composites, samples were tested to screw withdraw strength (Surface and Top) with speed of 15 mm/min^{-1} on the equipment MTM - AMSLER according to the NBR 14810-2:2018 [26]. In addition, three-point bending mechanical test was performed on a mechanical testing machine AME-5kN, according to the NBR 14810-2:2018 [26].

Statistical Analysis

Results were evaluated by ANOVA using the software Statistica, version 7. The LSD (least significance difference) intervals (p-value<0.05) were estimated, aided by response surface graphs (Surface response).

Results and Discussion

Density Evaluation

Table 2 shows the average apparent densities of the developed composites, as well as their standard deviation. According to the NBR 14810-2:2018 [26], the composites were classified as median density panels, presenting values ranging from 601 kg/m³ to 821 kg/m³. The composite 15 ST (containing untreated fibers) and 15CT (containing treated fibers using 10 % NaOH solution) presented the lowest and highest apparent density values, respectively. Moreover, the composite 20CT presented the highest standard deviation, 13 %.

Considering the density of composites, the statistical results indicated that the parameters NaOH Treatment and %(PU)Resin were not significant (p-value=0.179). However, the composite presenting higher density (15CT) was obtained using a higher concentration of NaOH (10 %) and lower %(PU)Resin (15 %). These results suggested that the increased roughness after mercerization process, as previously reported [29], may have resulted in the enhancement of composite compaction due to the increase of the matrix/fiber interfacial interaction. Furthermore, uniaxial thermal compression was applied during the production of the composites by the thermoforming process, resulting in gradients of heat and pressure (from the surface to the middle of the composite) [34]. For this reason, some regions could be differently compressed, and a density profile probably was created, increasing the standard deviation.

Table 2. Average density of the developed composites

Composite	Density (kg/m^3)
15 ST	601±11
20 ST	710±6
17.5 CT	793±8
15 CT	821±9
20 CT	693±13

Thickness Swelling (TS) Evaluation

Table 3 shows the TS(%) evaluation performed for 24 h before and after the aging test. The average TS values ranged from 6 % to 15 %. Accordingly, the NBR 14810-2:2018 [26] stated the maximum value of 22 % for medium density particleboards (P2).

Statistical analysis of TS data did not indicate an interaction between the analyzed parameters NaOH Treatment and %(PU)Resin, as well as they were not significant in the applied conditions. However, Figure 2 shows the response surface design, revealing considerable water sorption when both lower resin percentage and 5 % NaOH solution were used.

The mercerization process induces hydrophobicity in fibers, as well as increases the fiber/matrix adhesion and decreases the water sorption [35]. The lower TS value was observed in the composite 20ST (6 %), suggesting a tendency of stabilization when the percentage of PU was

Table 3. Thickness swelling performed during 24 h before and after the aging test

Composite	Thickness swelling (%) 24 h	Thickness swelling after aging test (%) 24 h
15 ST	15±5	13±1
20 ST	6±2	11±2
17.5 CT	8±3	7±2
15 CT	8±2	9±2
20 CT	10±2	9±3

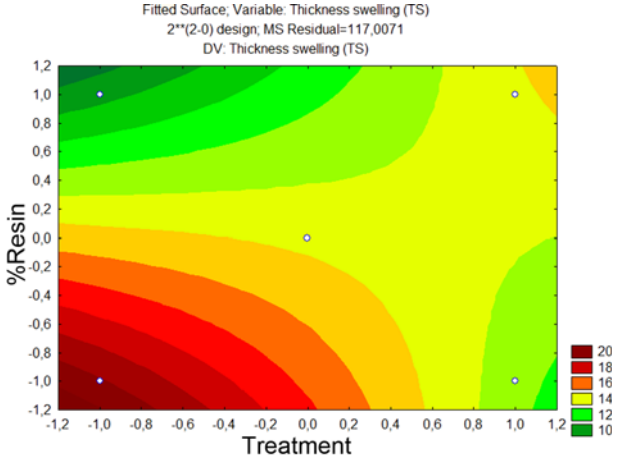

Figure 2. Surface response of the TS effect as a function of the parameters NaOH treatment and %(PU)Resin.

increased. This behavior can be attributed to the composite's matrix since PU has long hydrophobic chains inducing hydrolysis resistance [32,33]. The TS values obtained after accelerated aging were not statistically significant (p-value > 0.05).

Reports considering the effects of aging in composites usually evaluated mechanical [41,44] and optic [35] properties. In the present work, the color of the composite surface turned yellowish. This fact can be explained by the PU oxidation reactions [39].

Table 4. Thermal conductivity of the developed composites

Composite	Thermal conductivity (W/mK)
15 ST	0.24
20 ST	0.24
17.5 CT	0.34
15 CT	0.36
20 CT	0.30
Commercial MDF	0.24

Thermal Conductivity (TC) Evaluation

The TC results are shown in Table 4. Composites containing untreated fibers presented TC values similar to the commercial composite MDF (0.24 W/mK). On the other hand, the treated-fiber composites 20CT and 17.5CT presented higher TC values of 0.30 and 0.34 W/mK, respectively.

The TC results were not statistically significant (p-value > 0.05). However, the developed composites presenting a lower percentage of PU and treated fibers showed higher TC values. These results may be related to parameters such as density, homogeneity, and morphology, as well as to the influence of the PU resin as insulating material [40].

SEM Analysis

Figure 3, 4 and 5 show the SEM images of the composite's surface under different processing conditions. Filling failures were observed in the composite containing untreated fibers (Figure 3a and 3b). These failures can be generated by naturally occurring waxes and extractives accumulated on the fiber surfaces, and/or through the process of extraction, processing, and storage. As a result, the presence of waxes and extractives can decrease the matrix/fiber adhesion [17, 42,43]. Furthermore, the moisture of the fibers was another factor to be considered since untreated fibers present greater water absorption capacity. These fibers can absorb water from the environment or during the homogenization process due to poor matrix/fiber interaction at the interface [16,44].

Figure 4 and 5 show the SEM images of composites presenting treated fibers using 5 % and 10 % NaOH solutions, respectively. It was observed voids and microvoids in composites prepared using untreated fibers and treated fibers (Figure 3 and Figure 4), respectively. On the other hand, was observed the improvement of interfacial matrix/fiber

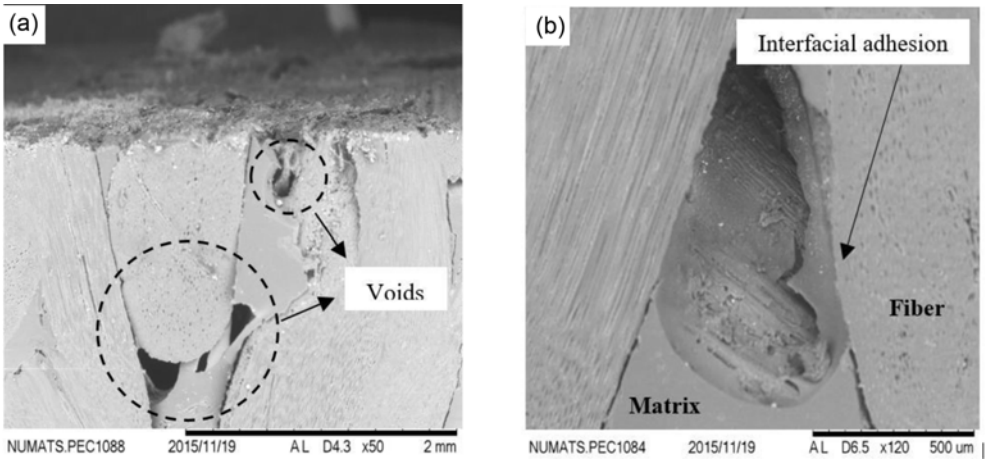

Figure 3. SEM images of the composite 15ST; (a) cross-section and (b) filling failures.

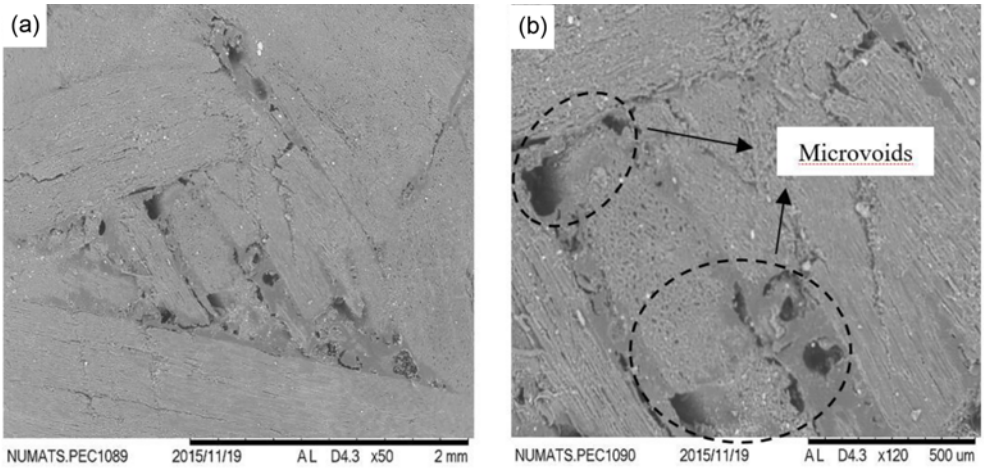

Figure 4. SEM images of the matrix/fiber interface of the composite 15 CT (5 % NaOH); (a) ×50 and (b) ×120.

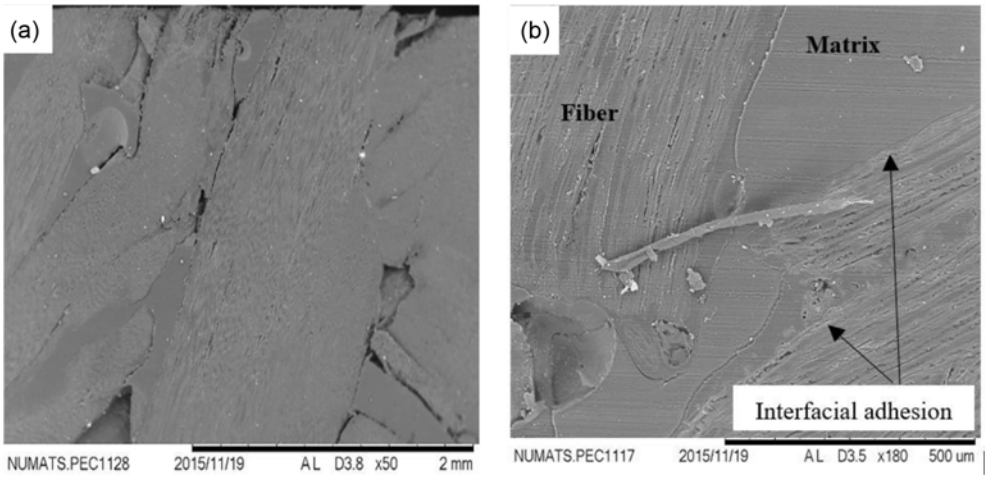

Figure 5. SEM images of the matrix/fiber interface of the composite 15 CT (10 % NaOH); (a) ×50 and (b) ×180.

adhesion in composites with treated fiber and some reports observed the same improvement after the mercerization process, resulting in composites with enhanced mechanical properties [13,20].

Mechanical Properties Evaluation

Table 5 shows the evaluated mechanical properties of the composites. The experimental conditions presented the minimum specifications required of 800 N for screw withdraw strength (top), with the exception of the composite 15ST (573 N). For screw withdraw strength (surface), only the composite 15CT was in accordance with the NBR 14810-2:2018 [26].

The results presented statistical difference considering the interaction between parameters (surface and top), resulting in *p*-value=0.014 and R2~94 % for surface, and R2~97.73 for top. Figure 6 shows the surface response for the screw withdrawal strength (top). It is possible observed that the composite presenting treated fibers (15 CT and 20 CT or level (-1+1) and (+1+1), respectively) showed higher screw withdraw strength, probably due to the increase the fiber/matrix adhesion.

Table 5. Mechanical properties of the developed composites

Composite	Screw withdrawal strength (surface) (N)	Screw withdrawal strength (top) (N)	Three-point bending MOR (N/mm²)	Three-point bending MOE (N/mm²)
15 ST	573	574	20	2956
20 ST	931	1049	16	2280
17.5CT	882±163	1262±207	18±2	2260±87
15CT	1019	1648	19	2582
20CT	848	1402	21	2260

The results of modulus of rupture (MOR) are shown in Table 5 and are in accordance with NBR 14810-2:2018 [26],

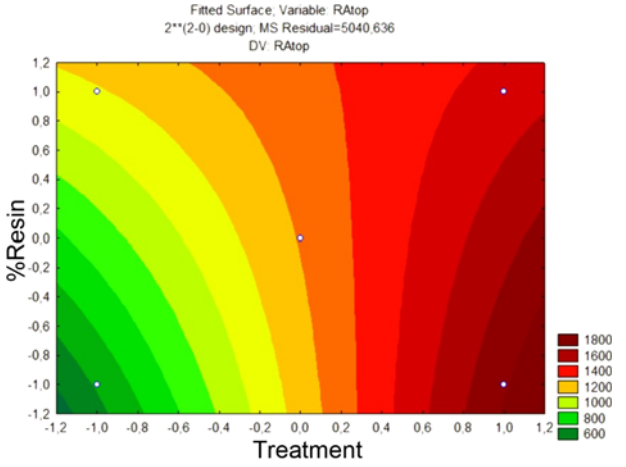

Figure 6. Surface response of screw withdrawal strength (top).

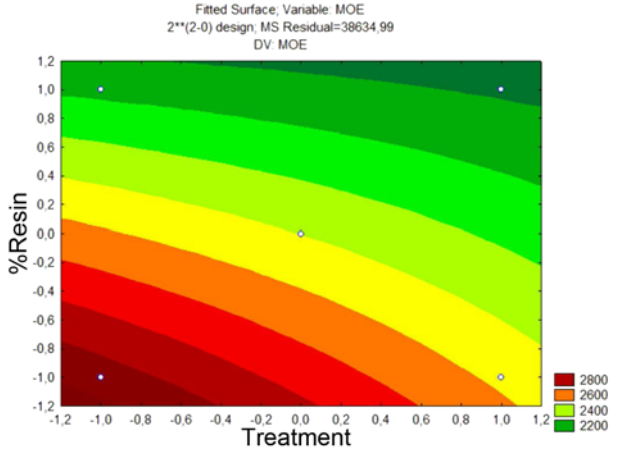

Figure 7. Surface response of the MOE effect as a function of the parameters NaOH treatment and %(PU)resin.

which recommends a minimum value of 11 N/mm^2. On the other hand, the results were not statistically different (p-value > 0.05). Figure 7 shows the response surface of the MOR effect as a function of the parameters NaOH treatment and %(PU)resin. Considering the higher concentration of PU, the MOR values increased when the concentration of treatment was increased, indicating that the mercerization process improved the mechanical properties.

The results of modulus of elasticity (MOE) are in accordance with NBR 14810-2:2018 [26] for all employed conditions. In addition, Figure 7 also shows that there was a tendency to smaller MOE values in the composites presenting treated fibers for the lowest PU concentration. This tendency was not observed for higher concentrations of PU. Furthermore, considering the same NaOH concentration (5 % or 10 %), the increase of the PU concentration decreased the MOE values probably due to the intrinsic characteristics of this polymeric resin.

Conclusion

Based on the results reported here, the PU resin concentrations and the alkaline treatment contributed to reduce the thickness dimensional variations of the composites. However, after the accelerated aging, there was no statistical variation of the thickness swelling. Composites presenting untreated fibers showed thermal conductivity similar to the commercial MDF. SEM analysis showed the porosity reduction in composites presenting treated fibers, besides the enhancement of the matrix/fiber adhesion at the interface. The porosity reduction was coherent with the increased density and decreased swelling. The mercerization process was statistically significant for the screw withdrawal strength (surface and top). the results of modulus of elasticity (MOE) and modulus of rupture (MOR) are in accordance with the NBR 14810-2:2018 standard for all employed conditions.

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