


EVALUATION OF SANDWICH COMPOSITES WITH VEGETABLE FIBERS AND HONEYCOMB MADE ON A 3D PRINTER

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Gilberto Garcia del Pino¹, Abderrezak Bezazi², Antônio Claudio Kieling³, Marcos Dantas dos Santos⁴, Aristides Rivera Torres⁵, José Costa de Macedo Neto⁶, Jamile Dehaine⁷

ABSTRACT

In modern society, polymer composites are widely used due to their high strength-to-weight ratio, among other properties. In recent times, due to environmental issues and higher costs, most industrial and engineering sectors are trying to reduce the use of synthetic fibers by using natural fibers or lignocellulosic fibers. New composite materials using renewable materials and additive manufacturing enable better development of high-performance materials for various industries. The objective of the work was to determine the behavior of a fully biodegradable sandwich composite using sisal fiber sheets in a polyurethane matrix of castor oil and PLA honeycomb using a 3D printer (Fused Deposition Modeling FDM). Firstly, a study was carried out to determine the best alkaline treatment for the sisal fibers with which the sandwich composite sheets were made, with the aim of determining the best treatment that produces greater adhesion of the fiber to the resin and therefore better tensile effort. For this, several composite plates of polyurethane resin matrix with castor oil were made using the same amount of fiber in all plates (10%w), from which the specimens for the tensile tests were cut in a LASER cutting machine with dimensions according to the Standard for this test. After performing the tensile tests, the best alkaline treatment was determined to be a

¹ Dr. in Mechanical Engineering
Amazonas State University, Manaus, AM
E-mail: gpino@uea.edu.br.
ORCID ID: 0000-0003-0754-2390

² Dr. in Mechanical Engineering
Laboratoire de Mécanique Appliquée des Nouveaux Matériaux (LMANM), Université 8 May 1945
E-mail: ar_bezazi@yahoo.com.
ORCID ID: 0000-0002-4461-6689

³ Dr. in Biotechnology
Amazonas State University, Manaus, AM
E-mail: akieling@uea.edu.br.
ORCID ID: 0000-0002-0552-954X

⁴ Dr. in Mechanical Engineering
Amazonas State University, Manaus, AM
E-mail: mdsantos@uea.edu.br.
ORCID ID: 0000-0002-4356-491X

⁵ Dr. in Mechanical Engineering
Amazonas State University, Manaus, AM
E-mail: artorres@uea.edu.br.
ORCID ID: 0000-0001-5138-2932

⁶ Dr. in Chemical Engineering
Amazonas State University, Manaus, AM
E-mail: jmacedo@uea.edu.br.
ORCID ID: 0000-0003-1155-0027

⁷ Dr. in Geophysics
Amazonas State University, Manaus, AM
E-mail: sofidehaini@gmail.com.
ORCID ID: 0000-0001-9766-6308

concentration of 10% sodium hydroxide and 4 hours of immersion of the dissolving fiber. To verify the efficacy of the treatment, fibers were also observed by SEM and XRD. Subsequently, sheets of the sandwich composite of castor bean polyurethane resin and sisal fibers treated with 10% NaOH dissolution and 4 hours of fiber immersion, previously determined also with 10% of w of fibers, were also made, and the cores of the sandwich composites with PLA honeycomb structure were also made, to make a comparison, they were also made with PETG. The flexure tests demonstrated that the sandwich composites performed better with PETG. In all tests, PETG was more resistant, usually 21% to 32% more resistant than PLA. But the specific resistances are similar, with PLA compounds having a 13.8% lower core density than PETG cores. The results obtained in the study coincide with the results previously published by other authors.

Keywords: Sisal. 3D printing. Sandwich panel. Composite.

1 INTRODUCTION

Currently, a lot of research is carried out worldwide in the area of Materials Engineering to obtain new materials that are cheaper and more ecological [1, 2], using composite materials with an epoxy resin or bioresin matrix, using castor oil polyurethane and with different reinforcements of vegetable fibers [3, 4], also thinking about research for the application in the area of biomaterials such as hydroxyapatite and zirconia in the heads of coxofemoral prostheses. In recent years, science using the foundations of Mechanical, Electronic and Materials Engineering has greatly helped in the field of medicine as the work of (VAZQUEZ-SEISDEDOS 2007) [5].

Plant fibers are increasingly explored as promising reinforcements in the composition of composite materials. This awakening of this interest is attributed to its low environmental impact, affordable cost, wide availability, versatile applications (construction, packaging, furniture, transportation), low energy consumption and minimal health risks. [6-7] In addition, the biodegradability of these fibers presents a solution to mitigate environmental pollution. [8-9]. Among the wide variety of natural fibers, sisal fibers are increasingly grown around the world, minimizing their transportation needs and, ultimately, carbon dioxide gas emissions. In addition, sisal fibers are considered a viable source of natural fiber, due to their high strength and biodegradability [10]. Sisal fiber is adapted for better abrasion and fungal resistance, high strength, better thermal stability, and economic and biodegradable ratios [11, 12].

The sisal plant belongs to the *Agave sisalama family* and the fiber is extracted from the leaves, and can reach up to 2 m in length. The number of fiber bundles per leaf depends on the age and origin of the plant, with length ranging from 60 to 120 mm. A sisal fiber has a density of 1.45 g/cm³, resulting in a porosity of about 17%. This fiber is hydrophilic and can absorb up to 11% moisture in an environment where the relative humidity reaches 65%. The chemical composition mainly includes cellulose (50–78%), hemicellulose (10–20%), lignin (8–12%), and waxes (about 2%). In terms of mechanical properties, the tensile strength ranges from 300 to 430 MPa, the Young's modulus ranges from 5 to 15 GPa, and the elongation at break ranges from 5 to 14%. [7]

The problem in polymeric composites with vegetable fibers is the incompatibility between the vegetable fibers and the polymeric matrix due to the fact that the fibers are hydrophilic in nature, the polymeric matrix is hydrophobic. As direct consequences of this fact, there is a weak interfacial adhesion between the polar and hydrophilic fiber and the non-polar and hydrophobic matrix, and mixing difficulties due to the low wettability of the fiber by the matrix, which leads to a weak interface of these composites, reducing the mechanical strength of the composite [13]. Therefore, the fibers must be properly modified through

physical or chemical treatments in order to improve the adhesion between them and thus improve the mechanical resistance of the composite, one of the most used being the treatment with sodium hydroxide (NaOH). [14]

Sandwich panels are types of structural composites whose core is made of a material that is less dense than the faces. They are designed to be low weight beams with high rigidity. It consists of 2 faces joined to a core by means of an adhesive. [15] One of the most widely used cores is with Honeycomb structure. *Honeycombs* are part of the class of cellular materials, formed by "voids" separated by solid walls, which stand out for having low relative density. [16]. One of the processes that can be used for the manufacture of *honeycombs* is additive manufacturing through FDM (*Fused Deposition Modeling*) 3D printing.

In this work, the study of a sandwich structure composite was carried out using sisal fiber composite faces in castor oil polyurethane matrix and *honeycomb* structural core printed in PETG (Polyethylene Terephthalate Glycol) and PLA (Polylactic Acid) in a 3D printer, with emphasis on the mechanical behavior of the composite in relation to the dimensions of the core and the materials used. The work comprises two parts, a first to determine the best chemical treatment of the Sisal fiber to obtain the best mechanical properties based on the tension stress and a second part, the analysis of the sandwich composites, applying the best treatment obtained.

2 METHODOLOGY

First, the study with Sisal fibers sought to determine the best chemical treatment to obtain the greatest adhesion of the fiber in the matrix, and then this treatment was used in the fibers of the plates or faces of the sandwich composites. The study was carried out by varying the concentrations of sodium hydroxide and the immersion time of the fibers in dissolution, with which, subsequently, composite tensile specimens were manufactured in epoxy resin matrix to determine the highest tensile strength of all the specimens and thus the best treatment.

The materials used in this first study were: Sisal fibers supplied by the FIBRAEX Company located in Conceição do Coité, in the state of Bahia, Brazil; sodium hydroxide supplied by the Chemistry Laboratory of the School of Technology (UEA/EST), and, and castor oil polyurethane supplied by Azevedo Indústria e Comércio de Petróleo Ltda (Brazil). 0.4% of anti-bubble additive (Siladit 53) supplied by REDELEASE (17) was used in relation to the total weight of the mixture.

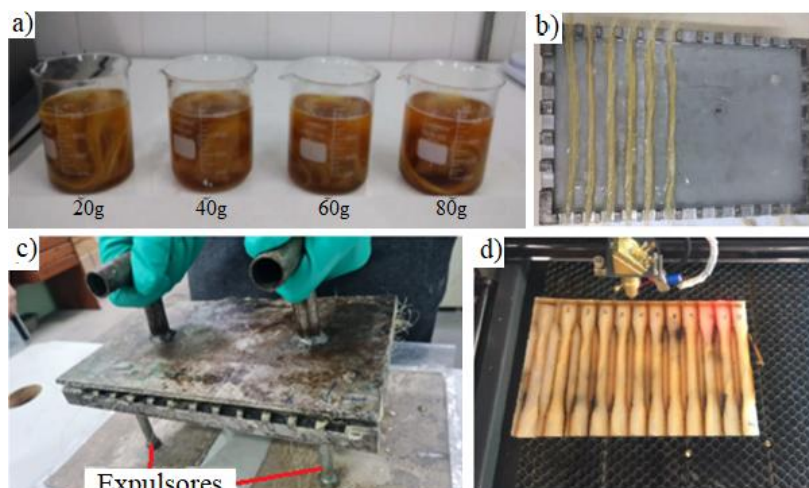
Before performing the chemical treatments with sodium hydroxide, the Sisal fibers were benefited by eliminating impurities, being washed in running water and dried at room

temperature for 24 hours. 4 different concentrations of sodium hydroxide 2.5, 5, 7.5 and 10 (wt%) were used, which corresponds to 20g, 40g, 60g and 80g of sodium hydroxide respectively in 800 ml of distilled water as can be seen in figure 1a, maintaining a ratio of 3/8 (fiber dissolution in all cases) [18]. The immersion time of the fibers at dissolution was 1, 4, 8 and 12 hours [19]. After the soaking time of each treatment was completed, the fibers were washed several times in running water and then in distilled water to stabilize the pH and placed to dry at room temperature for 24 hours and then in an oven at 60 °C for 8 hours. Finally, when preparing the composites in the mold, they were placed in the oven at a temperature of 100 °C for one hour to completely eliminate moisture. The greenhouse used was a QUIMIS model 0317M-72, which is located in the Materials Laboratory of EST/UEA.

To manufacture the specimens, composite plates were made in a metal mold with internal dimensions of 225mm x 155mm x 3.2mm. Before starting the manufacture of the specimens, a release wax was applied to the inner surface of the mold (Tec Glaze-N) supplied by the company REDELEASE itself, to facilitate the extraction of the composite from inside the mold. The specimens were manufactured by mixing the resin and the hardener in the proportion of 100/50 and 0.4% of the anti-bubbles in a Becker according to the manufacturer's recommendation by manually stirring for 15 minutes. Then a first part of the resin was placed in the mold, then the fibers as shown in figure 1b and finally the rest of the resin content. Before closing the mold with the lid, isopropyl alcohol was sprayed on the surface to help eliminate possible bubbles [19]. Finally, the mold was closed and pressure was applied to the top cover, and the method used was cold pressure.

The mold was closed under pressure for 24 hours in a first curing process, then the composite sheet was removed from the mold with the help of extractor screws (figure 1c) and placed in the oven for 8 hours at a temperature of 80 °C, to complete the curing process and increase the strength of the composite. The composite sheet after curing was taken to a LASER cutting machine (CNC Laser Router model VS6040) to cut the specimens according to the dimensions of the tensile test standard [20] (Figure 1d). All the specimens were given the same amount of fiber (20% by weight) to be able to study the effect of the chemical treatment.

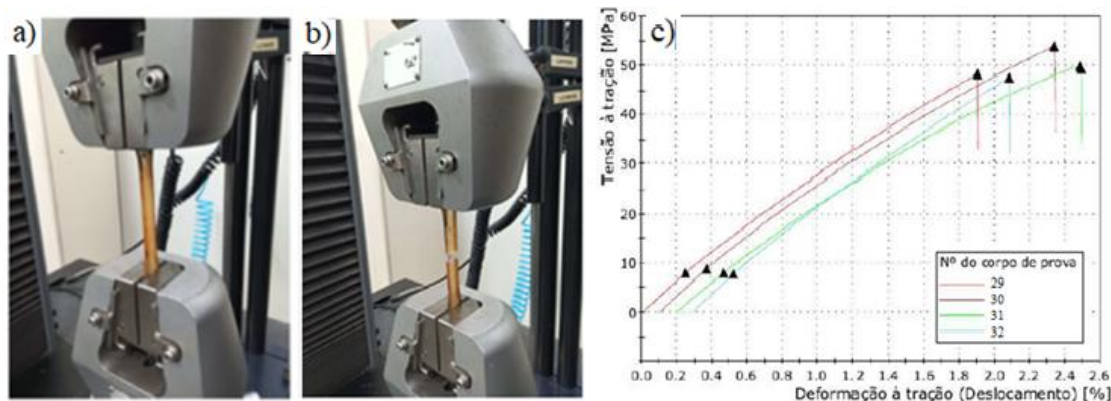
Figure 1: Manufacture of the specimens: a) - chemical treatment of the fibers, b) - placement of the fibers and resin inside the mold, c) - closing of the mold, d) - cutting of the specimens by LASER.



The specimens already cut with the dimensions of the standard for tensile tests were left at room temperature for 24 hours to relax the internal stresses and then were taken to the Mechanical Testing Laboratory of EST/UEA to perform the tensile tests in order to determine which of the specimens withstands the greatest tension stress and, therefore, it would correspond to the best treatment. All tensile tests were performed on an electromechanical universal testing machine model 5984, and a 150 KN load cell. The equipment was provided by EST/UEA, using the laboratory of the materials engineering course. These tests followed a standard speed of 5 mm/min (See figures 2a and 2b). The combination of the types of sodium hydroxide concentration (4) and the different immersion times (4) provided a total of 16 specimens. As 4 replicates of each case were carried out to take the average of the values, the total number of specimens was 64.

The best treatment in relation to the highest average value of the traction effort corresponded to the treatment of 10% by weight of sodium hydroxide concentration and 4 hours of immersion of the fibers in dissolution, which corresponds to results obtained in the literature [18, 19, 21, 22], Figure 2c presents the graph with the stress-strain curves corresponding to the chemical treatment that originated the greatest traction effort (best treatment).

Figure 2: Tensile tests: a) beginning of the application of the load on the traction machine, c) - end of the test at the time of failure, c) - deformation force graphs generated by the traction machine for the 4 replicates of the best results

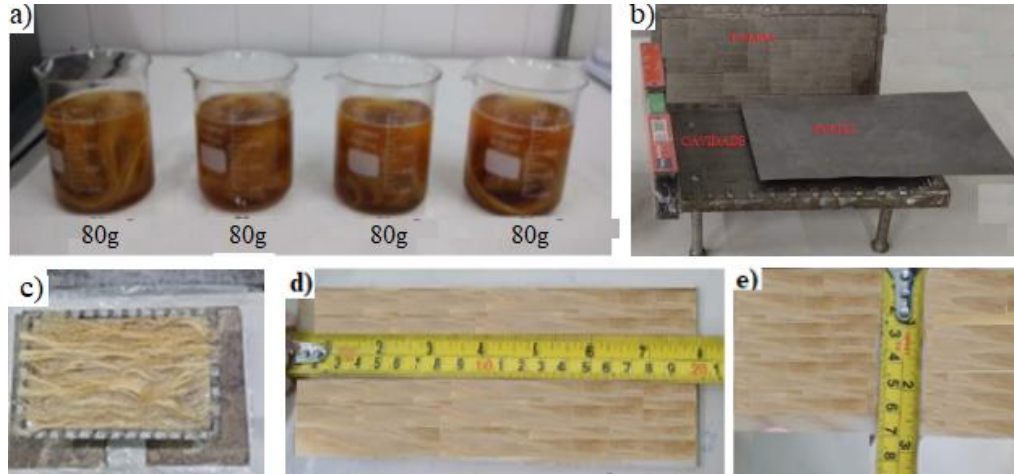


The initial work with the Sisal fibers for processing, washing and drying was the same as previously described with the exception of the chemical treatment, which in this case only the treatment determined by the previous result of 10% by weight of sodium hydroxide and 4 hours of immersion of the fiber in dissolution was used, as shown in figure 4a. The manufacturing process of sisal composite sheets in an epoxy resin matrix in the mold It was similar to the process described above, with the difference that in this case a metal sheet is placed in the mold cavity of 1 mm thick to obtain a 2 mm composite sheet, and the mold cavity is 3.2 mm, as can be seen in Figure 4B. The process of placing the fibers and the resin is similar to the process previously described, with the difference that the fibers are uniformly distributed in the area of the mold and not grouped by specimen as previously done (Figure 4c). The curing process of the composite sheets was the same as described above. After the curing process, the 2 mm thick composite sheets were cut on a LASER cutting machine with dimensions of 200mm x 70mm as can be seen in figures 4d and 4e. In total, 8 faces were cut to make 4 sandwich composites. Next, the composite cores were made in a honeycomb structure in a 3D printer. The honeycomb cores are the more conventional type of geometry used in the manufacture of sandwich panels. They are inhomogeneous nuclei, with open cells in the transverse direction of the slides, providing bidirectional support. [23]

For the 3D modeling process, the Fusion 360 software was used, a product licensed by Autodesk for free. To define the dimensions of the cores, 3 criteria were used, such as: Lowest possible layer thickness, 2 types of height in the cores, same size of the hexagons for all cores, as can be seen in figures 5a and 5b. The wall thickness was $0.3\text{mm} \pm 0.05\text{mm}$, considering the horizontal expansion of the filament (Figure 5a). The height of the cores was defined in two types 10mm and 15mm, based on the *honeycomb* cores that are currently marketed [24]. The printer used was the CREALITY ENDER 3 V2 model, in which it has a printing area of 220mm x 220mm and a maximum height of 250mm from the EST-UEA Machining laboratory. The slicing software that was Ultimaker CURA®, which is made available free of charge by the company Ultimaker, where all calibrated printing parameters

were inserted. Four cores were printed for the manufacture of the specimens. All nuclei obeyed the dimensions of the 3D models (Figure 7a).

Figure 3: Fabrication of the faces or sheet of the composite: a) alkaline treatment, b) mold to manufacture the sisal/epoxy composite sheets, c) placement of the fibers and resin inside the mold, d) length of the face, e) width of the face.



Next, the Honeycomb cores were glued to the Sisal composite faces with resin matrix. After pouring the resin over the faces, the core was placed centrally (see figure 4) and then a weight was placed on the cores. The process was repeated after 48 hours, for the bonding of the upper face [25]. Subsequently, flexure tests were performed at three points of the sandwich composites. The tests were carried out on the INSTRON 5984 model machine of EST/UEA, in the R&D Materials laboratory. All specimens have dimensions of 200mm x 70mm, with two faces of thickness of 2mm. The tests followed the ASTM standard [26, 27] of 3-point bending test, the test speed was 2mm/min. The distance between lower points was 100mm.

Figure 4: Manufacturing and gluing of honeycombs. a) Honeycomb printing, b) First face gluing

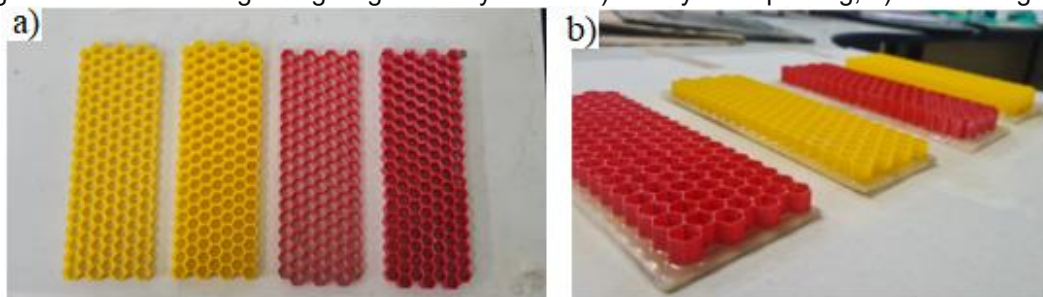


Figure 9: Flexure test at three points of specimen 1



Figure 10: Three-point flexure test of specimen 2



3 RESULTS AND DISCUSSIONS

When performing the 3-point flexure test on the sandwich panels, the results presented in Table 4 were obtained. As can be seen in Figure 13, there was no rupture in the faces, but there was detachment of the nucleus, which may have influenced the results. With the results obtained from the tests, it was possible to analyze and compare the stress and deformation of the specimens. The results obtained coincide with other previously published studies [28, 29, 30]. Analyzing the results in relation to height, we have that composites with 15mm of core supported 3.3 to 3.6 times more load than cores with 10mm.

In all tests, PETG was more resistant, generally 21% to 32% more resistant than PLA (Figure 14). Although there was no rupture in the specimens, the PETG cores deformed from 0.5% to 3.6% less than PLA. The composites with PLA were lighter, because the core density was 13.8% lower than the cores in PETG. The 15mm high cores

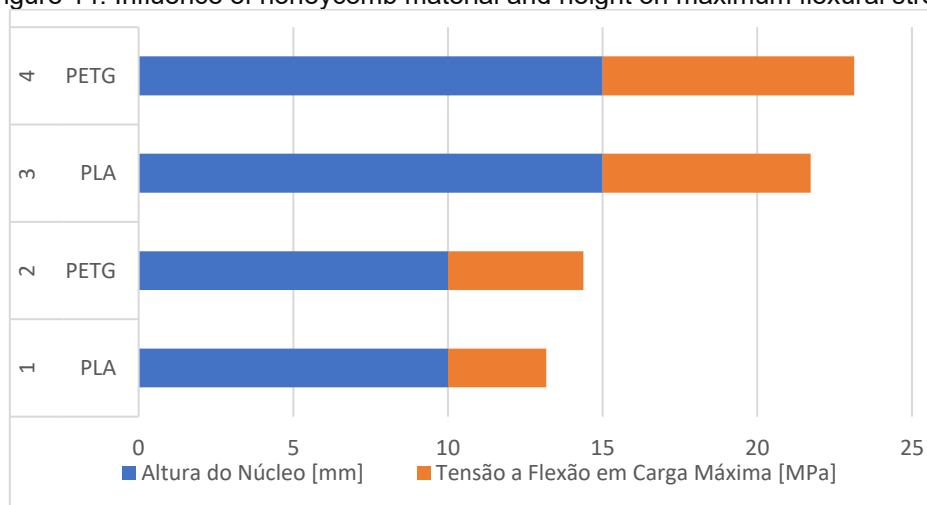
Figure 13: Specimens (1-4) After Flexion Tests



Table 1: Results of three-point flexure tests for all specimens

Specimen	Core Material	Core Height [mm]	Maximum Load [N]	Flexural Stress at Maximum Load [MPa]	Bending Strain [%]	Modulus of Elasticity [Gpa]
1	PLA	10	396,13	3,18	13,32	0,023
2	PETG	10	525,1	4,38	9,68	0,043
3	PLA	15	1443,91	6,73	2,41	0,280
4	PETG	15	1744,62	8,13	3,00	0,342

Figure 14: Influence of honeycomb material and height on maximum flexural stress



4 CONCLUSIONS

The main objective of the work was to create a sandwich composite with a *honeycomb* core made in a 3D printer, using PLA and PETG. Sisal fibers contribute to the income of small producers, research related to vegetable fibers helps the technological growth of composite materials, even enabling their use in industry. Analyzing the results in relation to height, we have that composites with 15mm of core supported 3.3 to 3.6 times more load than cores with 10mm.

The materials used in the cores were chosen for their ease of printing and for having ecological origins. PLA, as it is biodegradable and compostable, can be manufactured from corn, beets and cassava, as they are rich in starch. PETG, on the other hand, can be obtained through the recycling of PET bottles, reducing the impacts caused by pollution.

In all tests, PETG was more resistant, generally 21% to 32% more resistant than PLA. Although there was no rupture in the specimens, the PETG cores deformed from 0.5% to 3.6% less than PLA. The composites with PLA were lighter, because the core density was 13.8% lower than the cores in PETG. In general, it can be concluded that the PETG cores were better, preferably the 15mm high cores. The results were similar to other previous works in the bibliography. Honeycomb proved to be a very resistant structural arrangement with little density, occupying only 5.4% of the volume available between the faces, which allows it to be used in applications that may require the passage of other internal materials, such as pipes, cables. In addition, they can be used in applications that require a low density, such as the aeronautical and naval industries.

DECLARATION OF CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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