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PRODUCTION AND CHARACTERISATION OF BIODEGRADABLE MULTI-LAYER COMPOSITES MATERIALS FROM PELAGIC *SARGASSUM* SPP., LEAF SHEATHS AND POLYLACTIC ACID

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ABSTRACT

The massive invasion and proliferation of *Sargassum* algae on Caribbean beaches and coasts in recent years is threatening people's livelihoods, climate-sensitive areas and local ecosystems. The aim of this study was to assess the feasibility of manufacturing multi-layer composite particleboard using pelagic *Sargassum* as filler, coconut leaf sheaths as reinforcement and polylactic acid (PLA) as natural binder. The composites were subjected to three-point bending, density, porosity, morphology and biodegradability tests to assess the effect of number of layers, binder content and applied pressure during hot-pressing process on composite performance. The results show that weight loss after 15 days of biodegradation varies from 8.33 to 66.22%, with the greatest biodegradation observed on the composite consisting of a single layer of coconut leaf sheath. The stress-strain curve of the composite shows a ductile behavior of the materials, and only the composite composed of a 3-layers of coconut leaf sheaths and 10% by weight of PLA, pressed at 5 MPa, achieves the minimum flexural modulus of rupture of 18 MPa, as recommended by the Brazilian standard. Although the multi-layer composite has acceptable mechanical performance, appropriate treatment should be applied to *Sargassum* seaweed to control its water resistance.

KEYWORDS

Sargassum algae, coconut leaf sheath, PLA, multi-layer composite, flexural strength, biodegradation.

INTRODUCTION

Over the past decade, particleboard has gained popularity as a building material due to its wide range of applications and low cost [1]. In addition, particleboard is an environmentally friendly material made from wood waste such as shavings, sawdust and planing mixed with resin to form panels [2]. Due to the depletion of wood resources and the environmental impact of deforestation, the use of agricultural waste or non-wood lignocellulosic materials to replace wood in particleboard production has been increasingly considered in recent years [3], [4].

Since 2011, almost every year, massive strandings of tons of algae, mainly pelagic *Sargassum* brown algae (*Sargassum natans* and *Sargassum fluitans*) called the Great Atlantic *Sargassum* Belt (GASB), have occurred from the Caribbean to the African coast [5], [6]. As of June 2018, the estimate for this GASB was 20 million tons of biomass spread over 8,850 km [7]. Due to

the economic, social and environmental impacts associated with the large-proportion of *Sargassum* strandings along beaches and coasts, several researchers have focused in recent years on the valorization of this biomass in industry, such as cosmetics, alginates, fertilizers, bioplastics and recycled paper, and raw materials for construction [5], [6] in order to mitigate its impact. For example, the holocellulose content of *Sargassum* spp. is estimated at 46.08 wt.% of dry matter, corresponding to 20.35 wt.% cellulose and 25.73 wt.% hemicellulose [8], [9]. Therefore, the use of biomass from pelagic *Sargassum* spp. could be an alternative source of lignocellulosic fibers to meet the demand for raw materials from the wood industries. In addition, it could solve the problem of raw material shortages and provide a viable method to address the pollution problems associated with the stranding and degradation of *Sargassum* biomass along the Caribbean coast and beaches.

The particleboards production process is achieved by bonding small particles of lignocellulosic material, usually with synthetic adhesives, under pressure and heat for a period of time [2]. Compared to timber, the final structure of the panel has voids and cracks in its unity that reduce the mechanical strength [10]. Therefore, the choice of the adhesive is an important step on the design of the particleboard materials. Among the large number of adhesives, urea-formaldehyde (UF) and phenol-formaldehyde (PF) resins are mainly used [11]. This is due to their low cost compared to other types of adhesives as well as the excellent mechanical properties of the resulting bonded wood structure [12]. However, these particleboards are known to influence human safety, including potential risks to human health, such as cancer [13]. Today, the biodegradable polymers used are mainly polyesters, including poly(3-hydroxybutyrate) (PHA), caprolactone polymer (PCL) and poly(lactic acid) (PLA), whose chemical and mechanical properties are comparable to those of traditional petroleum-based polymers [14].

Due to its favorable properties, such as availability, relatively good strength, biocompatibility and biodegradability, polylactic acid (PLA) is the most frequently used biodegradable polyester in composite materials [15]. PLA is known as a versatile thermoplastic polymer derived from lactic acid through the fermentation of sugar beet, sugar cane, potatoes, corn and other starch-rich products [16]. Its degradability, compostability, favorable low melting point (160-180°C) for use with natural fibers, versatility with many applications in a variety of industries, and processability through thermoforming, extrusion, compression molding and injection molding are the main advantages of PLA polymer [17]. Several studies in the literature have addressed the development of green composites based on PLA resin reinforced with short fibers such as hemp fibers [18], waste orange peel [11], corn straw [14], oil palm stalk [19] and bamboo fibers [16].

This study attempted to assess the feasibility of manufacturing multi-layer composite particleboard using pelagic *Sargassum* as filler, coir sheaths as reinforcement and PLA as natural binder. The composites were subjected to three-point bending, density, porosity, morphology and biodegradability tests to assess the effect of the number of layers, binder content and hot-pressing pressure on the composite's performance.

MATERIALS AND METHODS

MATERIALS

Sargassum algae

The *Sargassum* used for this study was mainly pelagic *Sargassum* species, consisting of 67 wt.% *Sargassum fluitans* and 33 wt.% *Sargassum natans* algae, collected along the coastline of Grande Terre, Guadeloupe, from June to July 2022. The fresh *Sargassum* was collected in sea water about two meters from the shore to avoid contact of the *Sargassum* with the sand.

The algae were rinsed with tap water by manually removing impurities before being divided into two groups: (1) the first group was placed on the pallet for air drying while (2) the second group was subjected to two rounds of water immersion for 40 minutes at a volume ratio of 1:10 (algae:water).

Polylactic acid (PLA)

Polylactic acid (PLA) in pellets form with a density of 1.24g/cm³, a tensile modulus of 3500 MPa and an elongation at break of 5% was used as natural binder. It was supplied by NaturePlast (France). The thick PLA liquid was prepared by dissolving 14% wt./v of PLA in acetone at a temperature of 50°C under magnetic stirring [20].

Coconut leaf sheaths

Coconut fibers were collected manually from coconut palms (*Cocos nucifera L.*) in Guadeloupe (France). Coconut leaf sheaths are located at the petiole of the leaves and completely surround the trunk of the tree.

METHODS

Production of the multi-layer particleboards

The measured amount of *Sargassum* was dried at 70°C for 4 hours and transferred directly into the mixer. While mixing the *Sargassum*, the corresponding amount of PLA solution was gradually introduced into the mixer and after the complete introduction of the binder, the speed of the mixer was maintained for 5 minutes in order to obtain a homogeneous paste. Then, the paste was immediately poured into the mold (20cm x 20cm) and all was placed for three days under the fume hood for the evaporation of the acetone solvent. Two metallic plates recovered with parchment paper were used for hot pressing process of the block. A sandwich structure was made by interposing layers of coconut leaf sheaths between PLA/*Sargassum* blocks manufactured with PLA binder contents of 10 wt.%, 12.5 wt.% and 15 wt.%, then pressed in a computer-controlled hot press at a temperature of 160°C and pressures of 2.5 MPa and 5 MPa for 10 minutes (Fig. 1). Multi-layer panels were then stored in the laboratory environment for 7 days [21] before being cut to the size of 7.5 cm x 2.5 cm for physical, mechanical and biodegradation testing.

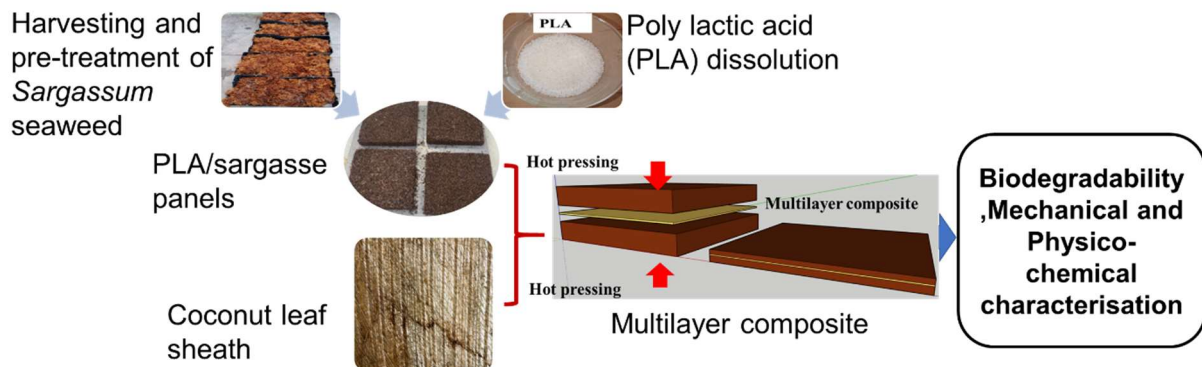


Fig. 1: Steps of the preparation of multi-layer composites particleboards

Designation of the multi-layer composites

The composite was identified by their name composed of three elements: (1) the pressure applied during the hot-pressing process (2.5 or 5 respectively for 2.5 or 5 MPA applied), (2) the content of PLA reported as PLAx for x wt.% of PLA and (3) number of coconut leaf sheaths (1FL or 3FL for respectively 1 or 3 layer(s) of coconut leaf sheath(s)).

For example, the sample 5PLA10_3FL was produced under a hot-pressing of 5 MPa, using 10% of PLA and 3 layers of coconut leaf sheaths

Bulk density, specific density and porosity of multi-layer composites

Prismatic samples of 1.5 cm x 2.5 cm x 5 cm were oven dried and bulk density was determined by dividing the mass of the samples by their volume. A helium pycnometry apparatus (Thermo Electron Corporation) was used to determine the specific density of the composites (ρ) according to the procedure described by (Mierzwa et al., 2019). The porosity (φ) of the sample was calculated from its bulk density and its specific density according to Eq. 1. The experiment was repeated three times.

$$\varphi = \left(1 - \frac{BD}{\rho}\right) \times 100 \quad (1)$$

Where φ is the porosity of the sample (%), BD is its bulk density (g/cm^3) and ρ its specific density (g/cm^3).

Three-point bending test on multi-layer composites

Five specimens of the same formulation were subjected to a three-point bending test to determine the mean bending strength and mean modulus of elasticity, for a given particleboard formulation. Samples measuring 2.5 cm x 7.5 cm were prepared from each type of panel for the assessment of mechanical properties and the test was carried out on a Shimadzu universal testing machine under a 10 kN load cell at a speed of 1 mm/min.

Morphological observation of multi-layer composites

The morphology of raw materials and composites was performed with scanning electron microscopy (SEM), FEI Quanta 250, operating under secondary electron (SE) at the acceleration voltage of 15 kV.

Biodegradation behavior of multi-layer composites

The biodegradability tests were carried out according to the methodology described by Dong et al. [22]. Five samples measuring 20mm x 20mm were cut from composites, weighed and buried in a deep layer of various composts (One commercial Compost COM and others by produced by INRAE research center Ver micron, Compost 50DV+50F, and Co 100F). Water was constantly added to the compost container each day to ensure good microbial activity. After the 15-days period, the samples were removed, washed with tap water and oven-dried for 12 hours to remove the moisture. The weight loss W_{loss} was calculated based on the following Eq. (2) [23]:

$$W_{\text{loss}}(\%) = \frac{W_o - W_t}{W_o} \times 100 \quad (2)$$

where W_o and W_t are weights of specimens respectively before and after burial in the compost soil. The mean weight loss was calculated from 5 measurements of the same formulation.

RESULTS AND DISCUSSION

Bulk density, specific density and porosity of multi-layer composite materials

According to the Brazilian standard NBR 14810-3 (2006) [24], with the exception of the panel composed of 5 layers, 15 wt.% PLA and pressed at 5 MPa with a bulk density of less than 0.75 g/cm³, the panels produced in this study can be classified as high-density panels (bulk density greater than 0.75 g/cm³) (**Fig. 2a**). Bulk density (**Fig. 2a**) and specific density (**Fig. 2b**) show the same trends. However, composites containing 10 wt.% PLA show a slight increase in bulk density with increasing applied pressure and number of layers. On the other hand, composites containing 15 wt.% PLA show a decrease in bulk density with increasing pressure. The porosity (**Fig. 2c**) of the composite decreases with increasing density.

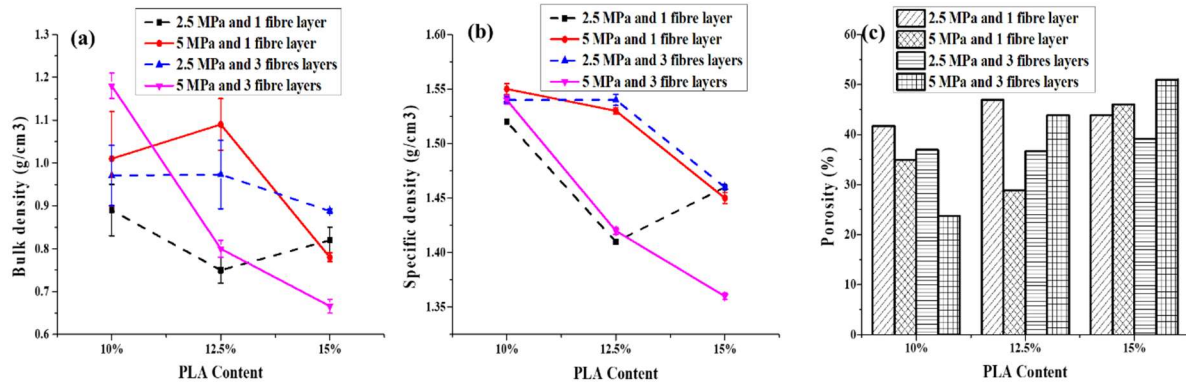


Fig. 2: Effect of PLA mass content, number of layers and applied hot pressure on the (a) bulk density, (b) specific density and (c) porosity of multi-layer composites.

Mechanical behavior of the multi-layer composite materials in three-point bending

The composites exhibit ductile behavior due to the presence of coir leaf sheath fibers, which prevent crack propagation (**Fig. 3**). In addition, increasing the applied pressure from 2.5 to 5 MPa significantly improves the flexural strength of composites made with 10 and 12.5 wt.% by weight PLA (**Figs. 3 and 4**). The opposite behavior was observed for composites containing 15 wt.% PLA. This can be attributed to the higher sensitivity to calcination of the composite made with 15 wt. % PLA, resulting from the high amount of binder. Furthermore, among all the multi-layer materials, only the 3-layer coconut leaf sheath composite with 10 wt.% PLA, pressed at 5 MPa, achieves the minimum flexural modulus of rupture of 18 MPa (**Fig. 3b**) recommended by the Brazilian standard [24].

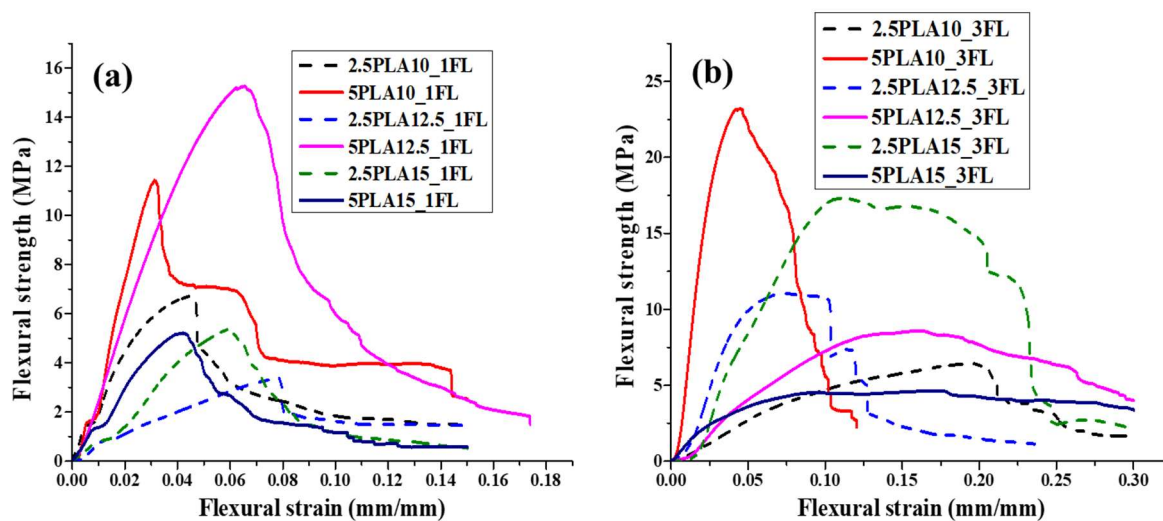


Fig. 3: Stress-strain curves for the 3-point bending test of composites with (a) 1 layer (b) 3 layers of coconut leaf sheaths.

Microstructure of raw materials and multi-layers composite materials

Micrographs of *Sargassum* seaweed (Fig. 4a) show a rough surface that can enhance the bonding and anchoring between the seaweed particles and the PLA binder. In addition, the coconut leaf sheath (Fig. 4b) shows a two-way weaving of the fibers and a rough surface of the fibers able to promote good adhesion and bonding between the coconut fiber layers and the PLA/*Sargassum* particleboards.

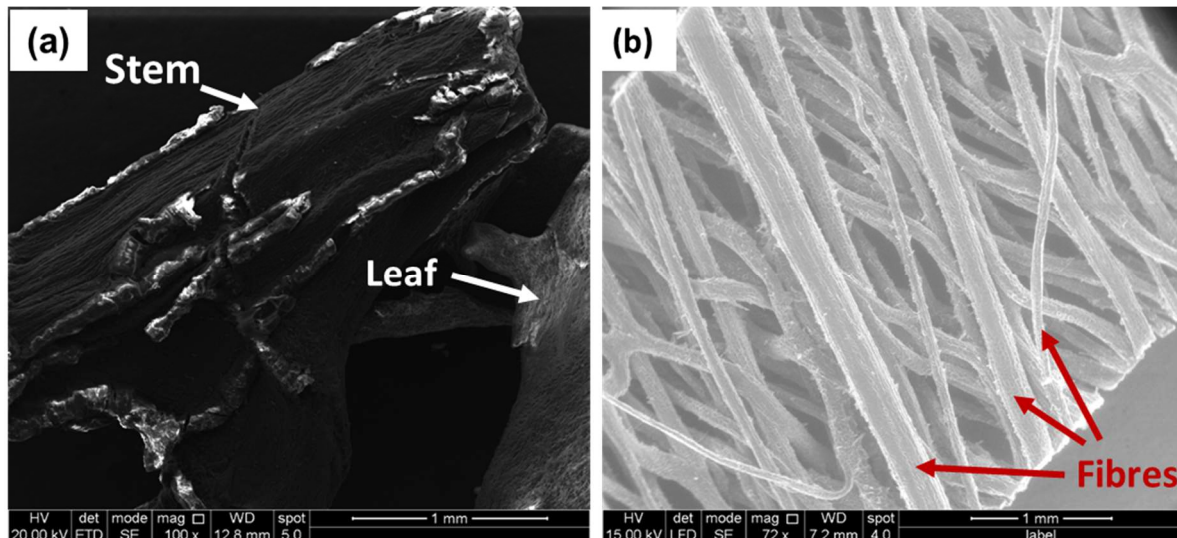


Fig. 4: Scanning electron microscopy (SEM) micrographs of (a) *Sargassum* algae and (b) coconut leaf sheaths.

Furthermore, the microstructure of the multi-layer composites made with 10 wt.% of PLA (Fig. 5) reveals the stripping of the leaf sheath fibers (Fig. 5a) prior to breakage. Figs. 5a and 5b show the presence of pores in the composite due to the *Sargassum* float and the partial bonding between the coconut leaf sheath layer and the PLA/*Sargassum* ones.

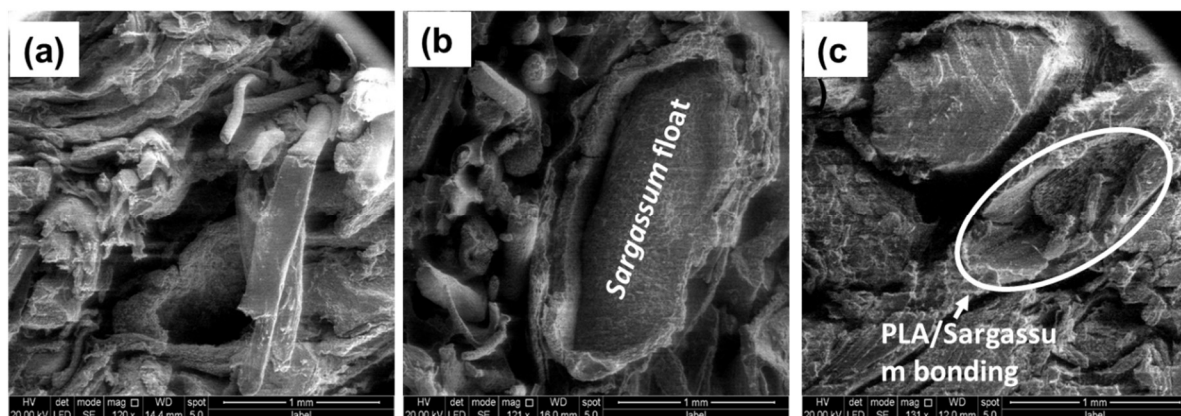


Fig. 5: SEM micrographs of composites made with 10% by weight of PLA and pressed at 2.5 MPa (a and c) and 5 MPa (b).

Biodegradability analysis

Figs. 6a and 6b show that after 15 days, the biodegradation varies from 8.33 to 66.22%, with the highest biodegradation observed for the composite consisting of a single layer of coconut leaf sheath. For all the studied composites, the impact of PLA content on the biodegradation

was more significant for the single layer coconut leaf sheath composite, which showed the highest percentage of degradation at 10 wt.% of PLA content. Furthermore, with regard to the mean values and standard deviations of the percentage degradation, the pressure applied during the hot-pressing process and the type of compost used for burial had no statistical impact on the biodegradation of the multi-layer composites.

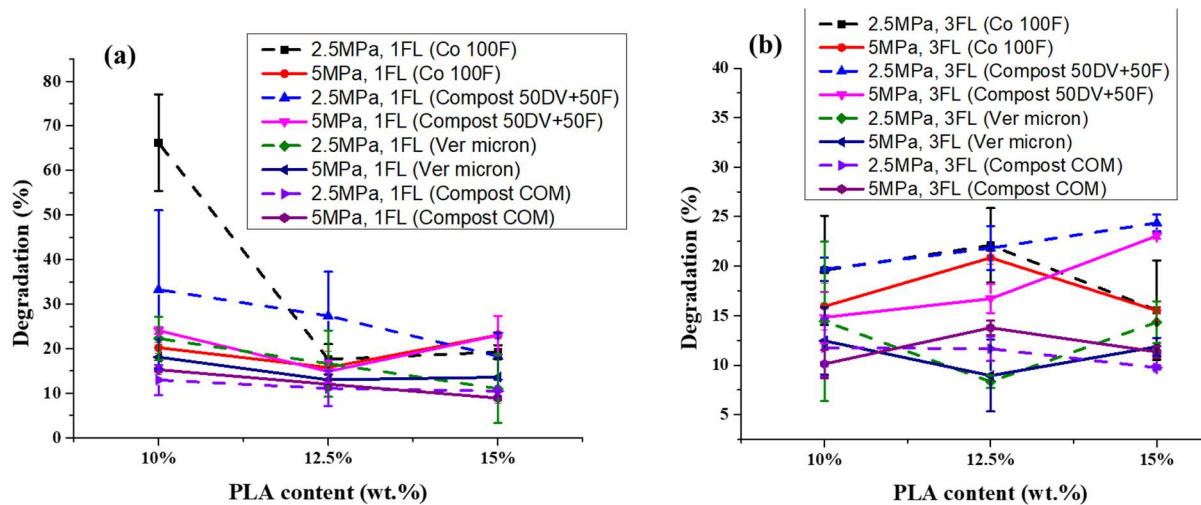


Fig. 6 Percentage of degradation of multi-layer composites made with (a) 1 layer and (b) 3 layers of coconut leaf sheath.

FINAL REMARKS (OR CONCLUSIONS)

Preliminary results on the production and physical and mechanical characterization of multi-layers composites based on PLA binder, *Sargassum* and coconut leaf sheaths show that these composite materials can be classified as high-density panels (with the exception of the composite with 15 wt.% PLA, 3 layers of coconut leaf sheaths and pressed at 5 MPa) according to the Brazilian standard NBR 14810-3 (2006). In addition, the composites exhibit ductile behavior due to the presence of vegetable coconut leaf sheath fibers, which prevent the propagation of cracks. Flexural strength grows with increasing pressure of the hot-pressing process. However, only the 3-layers coconut leaf sheath composite with 10% by weight of PLA, pressed at 5 MPa, achieves the minimum flexural modulus of rupture recommended by the Brazilian standard (18 MPa). The results show that weight loss after 15 days of biodegradation varies from 8.33 to 66.22 %, with the greatest biodegradation observed for the composite made of a single layer of coconut leaf sheath. Although the multi-layer composite has acceptable mechanical performances, appropriate treatment should be applied to *Sargassum* seaweed to control its water resistance.

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