


Development and Characterization of Polystyrene Eco-composites Reinforced with Australian Royal Palm (*Archontophoenix alexandrae*) Stem Fibers

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Abstract

Given escalating environmental concerns and stricter regulations, there's a pressing need for sustainable materials. Eco-composites, which blend natural fibers with polymer matrices for reinforcement, are gaining traction. This study stands out by employing Australian royal palm stem fibers within a polystyrene matrix for eco-composites. The research involved assessing thermal stability, quantifying lignin/carbohydrate content, and measuring moisture levels. Scanning electron microscopy was utilized to examine fiber structure. Results from evaluations of thermal and mechanical properties underscore the high-performance reinforcement potential of Australian royal palm fibers, suggesting their suitability for various eco-composite applications. Beyond meeting the demand for sustainable materials, this project introduces an innovative use of these fibers, broadening their potential applications and advocating for greener, socially responsible industry practices.

Keywords: Recycling, lignocellulosic materials, renewable resources, ecologically correct, environmental impact.

1. INTRODUCTION AND OBJECTIVES

Today, in a world driven by growing environmental and social concerns, along with dwindling oil resources and the implementation of new policies, the search for innovative solutions is more fervent than ever. It is in this context of ecological transformation that the promising era of eco-composite materials emerges (Khalid et al., 2022). These pioneering materials not only incorporate natural and recycled elements into their composition, but also play a key role in diverse applications, ranging from construction to the automotive industry (Sanjay et al., 2018).

The real innovation behind this movement lies in the research and development of composites that make use of natural fibers in combination with polymer matrices. Natural fibers, with their unique properties, such as biodegradability, abundance, adaptability to chemical modifications, ease of handling and remarkable physical and mechanical properties, are revolutionizing the market. By incorporating them as reinforcement in composites, we are reducing the consumption of synthetic fibers and, consequently, mitigating greenhouse gas emissions (Nurazzi et al., 2021; Sanjay et al., 2018).

Among the sources of natural fibers, fibers from the Australian royal palm, better known as *Archontophoenix*

alexandrae, stand out as an extremely promising reinforcement in the production of composites. It is interesting to note that Brazil, a global leader in the production and consumption of heart of palm, also generates a significant amount of waste from this resource. These residues have found several applications, but there is a growing demand for products with higher added value (Farina et al., 2022; Hafemann et al., 2019; Zanini et al., 2022; Zenni et al., 2018).

The research carried out by Mulinari et al. (2017) explores the unexplored territory of using Australian royal palm fibers in eco-composites. The main focus is on polymer matrices such as polyethylene (PE), polypropylene (PP), polystyrene (PS) and polyvinyl chloride (PVC), all processed at temperatures around 200°C (Ayre, 2018). The inclusion of plastic recycling as part of these eco-composites is a key measure to mitigate plastic emissions, especially in a scenario of increasing use of disposable packaging and non-durable products (Zhang et al., 2022).

The present study is motivated by the increasing relevance of materials from renewable and ecologically correct sources. The primary objective is innovation: to develop and characterize polystyrene eco-composites reinforced with fibers from the Australian royal palm stem, exploring their technical viability as a promising alternative to traditional plastic materials. This is a bold step towards a more sustainable future, where innovation and environmental awareness go hand in hand.

2. MATERIALS AND METHODS

2.1. Preparation of Australian royal palm fibers

Initially, the process involved collecting the stem of the Australian royal palm in Cajuri, Minas Gerais, Brazil. To prepare the fibers, they were subjected to a drying process at 110°C for a period of 8 hours. Then, the fibers were subjected to a two-stage milling step, first in a Los Angeles abrasion mill for 14 minutes and then in a Willy mill. To ensure uniformity, the fibers were sieved through a 10-mesh sieve, allowing particles up to 2 mm in size to pass through.

2.2. Kraft lignin isolation

Kraft lignin was obtained from the Kraft black liquor generated during the conventional cooking of *Eucalyptus* sp. chips. The isolation process involved adding concentrated sulfuric acid to the black liquor under constant stirring at room temperature. After lignin precipitation at a pH of 2.0, the mixture was filtered and the resulting lignin was washed with distilled water until reaching a neutral pH. Finally, the lignin was dried and ground before being used in the study.

2.3. Characterization of the stem and formation of eco-composites

The characterization of the stem aimed to identify the potential application of reinforcement of this material. Thermogravimetry (TG), total lignin content, carbohydrate content, moisture and scanning electron microscopy (SEM) were performed. Thermogravimetry was performed by simultaneous TG/DTA scanning at 10 °C min⁻¹, with an airflow of 25 mL min⁻¹ in the range of 25-800°C, using an alumina crucible, in a TG analyzer TGA-Q5000 (TA-Instruments).

The lignin and carbohydrate contents were measured in a pulverized sample, after removing the extractives. 300mg of stem were added to a test tube, followed by the addition of 3mL of sulfuric acid (72% v/v, cooled to 10-15°C). The assembly was frequently mixed for 1 hour at 30°C. After that, the mixture was quantitatively transferred to a vial of 100mL of penicillin with 84mL of water. The flask was hermetically closed and placed in an autoclave with a water bath for 1 hour at 118°C.

The carbohydrate content was performed as follows: samples from sterilized lignin content analysis bottles were filtered through a cellulose acetate membrane, and the filtrate volume was diluted to 250mL with water. Then, 50mL of the filtrate were added to a beaker and mixed with 10mL of erythritol (3000 mg L⁻¹). The pH of the mixture was adjusted to 5.3 by a Ba(OH)₂ solution and then the system was centrifuged at 5000 rpm for 2.5 minutes. The mixture was then concentrated to a final volume of 10mL. The sample was added dropwise to a sep-pak and finally injected into a high performance anion exchange chromatography with pulsed amperometric detection system (HPAEC-PAD). Chromatographic separation was performed on a DIONEX ICS-3000, equipped with a CarboPac PA1 column (250 × 4 mm). For all separations, an isocratic elution of 0.001 mol L⁻¹ NaOH and an eluent flow rate of 1 mL min⁻¹ for 45 min was applied (Wallis et al., 1996).

Soluble lignin as follows: hot autoclaved samples were filtered on a sintered glass filter, previously prepared with an asbestos layer (2-3 mm) washed with acid and weighed. The lignin retained on the filter was washed with distilled water to completely remove the acid. The set was dried in an oven at 105°C until constant weight. After that, the system was weighed to determine the lignin content.

Stem moisture was determined in three replicates, by drying in an oven (Lucadema) at 110°C for 48 hours. After that, the samples were placed in a desiccator and cooled to room temperature. Samples were weighed to constant mass. The moisture content was calculated according to equation 1 below:

$$MC, \% = [(W_0 - W_f)/W_0] \times 100\% \quad (1)$$

where: MC = moisture content, W_0 = initial weight (g), W_f = final weight (g).

Scanning electron microscopy was initially performed by preparing the sample with gold coverage, by sputtering, in a Denton Vacuum - Desk II metallizer, with an electric current of 40 mA for 10 seconds, for adequate acquisition of images. Images were obtained using a JEOL JSM - 6360LV scanning electron microscope.

The formation of eco-composites was carried out as follows: the polystyrene from plastic cups was fragmented into small pieces with the aid of scissors and an industrial beater. Then, the polystyrene pieces were dissolved in chloroform (CHCl_3) at the proportion of 7g/25mL in a beaker under constant agitation until complete dissolution. Different proportions of components were added to the eco-composite mixtures. The polymer matrix was composed of 100% polystyrene (P) or 90% polystyrene and 10% stem fiber (PC). The plasticizer glycerol was added at 10% to the PG, PCG and PL mixtures. Lignin was added at 10% to PL, PCL and PCG mixtures. Glycerin and lignin were also added to the mixtures, in order to evaluate the plasticizing properties of both substances. Finally, the different mixtures were then applied onto a 50 micrometer film-forming glass plate using a hand-held film metallic carrier. After solvent evaporation, the films were detached and cut into test pieces.

2.4. Characterization of eco-composites

The characterization of the eco-composites involved analysis by Fourier transform infrared spectrometry (FTIR) in the range of 4000 to 500 cm^{-1} after 128 scans, using a JASCO 4100 spectrometer with total attenuated reflectance (ATR) accessory. In addition, thermogravimetry (TG) was performed with a heating rate of 10°C min^{-1} in a synthetic air flow of 25 mL min^{-1} in the temperature range of 25 to 800°C using an alumina crucible and TGA-Q5000 equipment (TA-Instruments).

To evaluate the physical-mechanical properties of the eco-composites, strength tests were conducted in a Series IX Tensile Tester (Instron Corp.) on test pieces (15 mm x 165 mm) at an elongation rate of 25 mm min^{-1} , according to ASTM D 638. Analysis of variance and Tukey's tests were performed to assess means with a significance level of 5%.

Table 1. Lignin and carbohydrate content in the stem.

Sample	Soluble Lignin (TAPPI), %	Insoluble Lignin %	Total Lignin, %	Carbohydrates Contents, %				
				Arabinose	Galactose	Glucose	Xylose	Mannose
Stem	4.20	20.15	24.35	2.0	1.2	40.4	13.3	1.0

3. RESULTS

Figure 1 shows the thermogram (TG) and mass derivative (DTG) of the stem fibers.

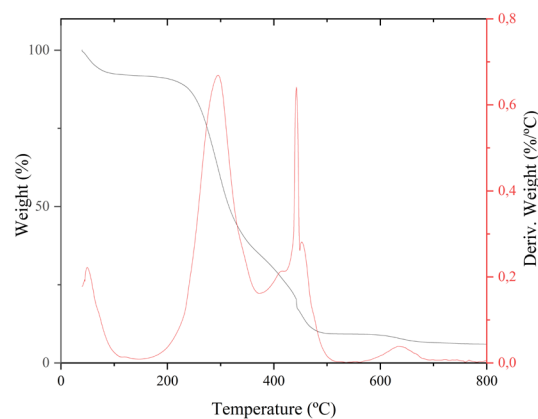


Figure 1. Stem TG/DTG thermograms.

The results of the total lignin and carbohydrate content of the Australian royal palm (*Archontophoenix alexandrae*) stem are presented in (Table 1).

Figure 2 shows images obtained by scanning electron microscopy that provide a detailed view of the fibers present in the stem of the Australian royal palm.

Fourier transform infrared spectrophotometry (FTIR) was performed for the eco-composite samples and the spectra are shown in Figure 3.

According to De Araújo and Kawano (2001), polystyrene monomers have three CH binding conditions: CH_2 , CH and aromatic CH.

The thermal behavior of the eco-composites is shown in Figure 4.

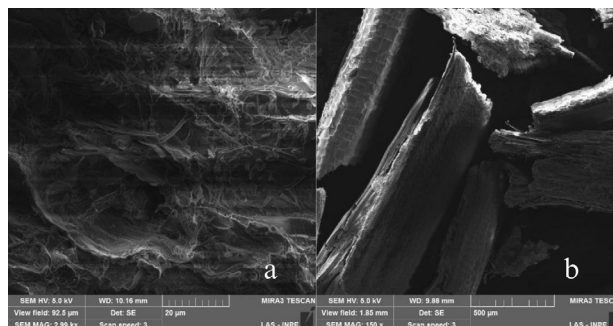


Figure 2. Scanning electron micrographs of the stem. Magnification of 150 x (a) and 2,99 kx (b).

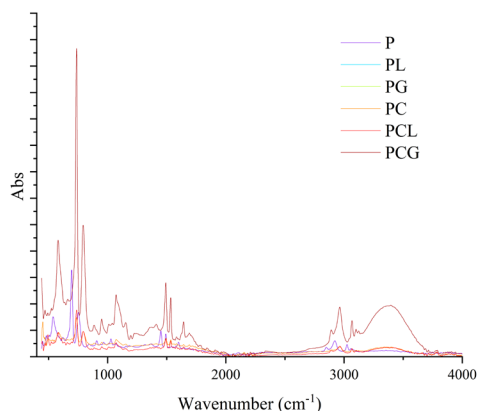


Figure 3. Fourier transform infrared (FTIR) spectra of eco-composites.

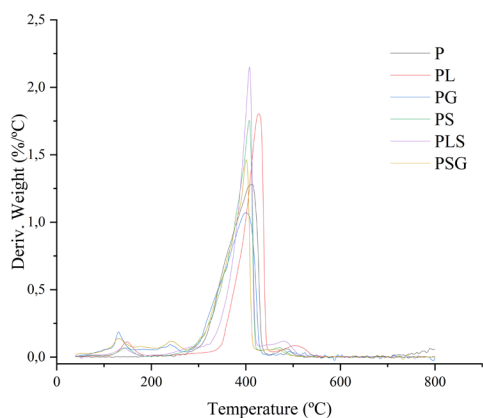


Figure 4. Thermogravimetric (TG) and mass derived (DTG) thermograms of eco-composites.

The PCG, PG and PL eco-composites showed three degradation peaks, while the PCL, PC and P eco-composites showed two degradation peaks. The first degradation peak is related to the stem fibers, the second peak is related to glycerin (in PCG and PG eco-composites) and the third peak is related to the polymer matrix. The values of the degradation peaks vary between 130.4°C and 140.8°C for the first peak, between 239.7°C and 273°C for the second peak and between 399.4°C and 413.7°C for the third peak.

Physical-mechanical tests were applied to all eco-composites. According to Joshi & Bhanot (2005), the tensile index (Nm/g) is the tensile strength (N/m) divided by the weight of the specimen (g/m²). The tensile index values of the eco-composites are shown in Figure 5.

The modulus of elasticity (Young’s modulus) indicates the stiffness of the material, so that the higher the modulus of elasticity, the stiffer the material and the less elastic deformation when a stress is applied. The values of the modulus of elasticity of the eco-composites are presented in Figure 6.

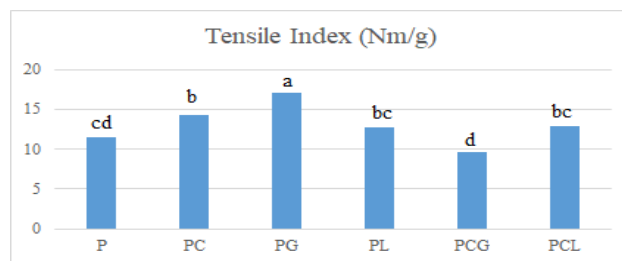


Figure 5. Tensile index of eco-composites.

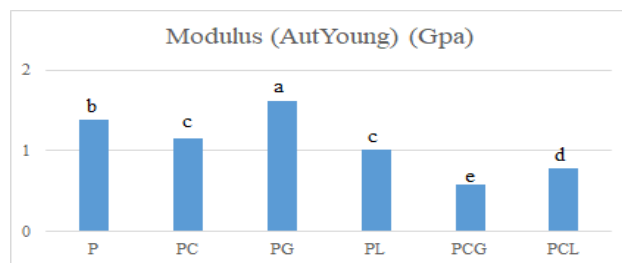


Figure 6. Modulus of elasticity of eco-composites.

The elastic limit gives the maximum stress that a material can withstand without permanent deformation. The elastic limit values of the eco-composites are shown in Figure 7.

The tensile strength limit is the stress measured at the maximum load achieved during the test. Figure 8 presents the values obtained for the tensile strength limit for the eco-composites.

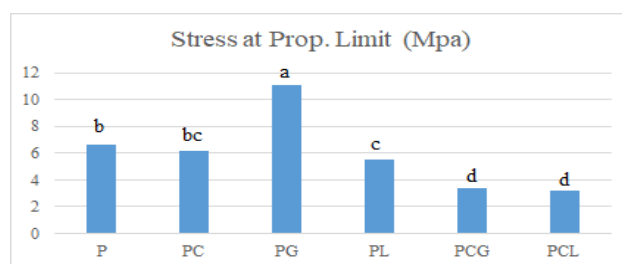


Figure 7. Elastic limit of eco-composites.

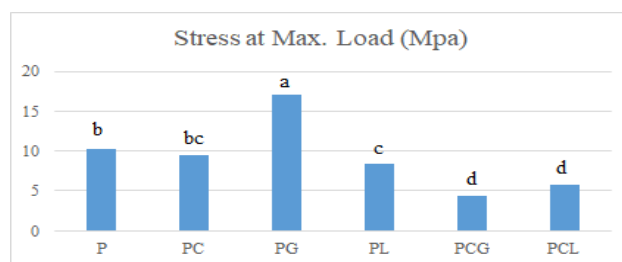


Figure 8. Tensile strength limit of eco-composites.

4. DISCUSSION

As can be seen in Figure 1, two main stages of mass loss are presented in the following ranges from 218 to 374°C and 428 to 503°C. Small mass losses below up to 100°C are due to water evaporation. The first stage of mass loss is probably related to the thermal degradation of the stem, associated with carbohydrates derived from cellulose and hemicelluloses, while the second stage may be associated with the complete decomposition of the stem, in which lignin would be the main component. This result is in agreement with Miléo et al. (2016), who described that cellulose and lignin present considerable mass loss above 300°C.

According to Thielemans and Wool (2005), lignin acts as a track, connecting the cells in the wood stems, presenting an important hole in the biomass structure, providing rigidity and resistance to compression. Table 1 shows that the total lignin content is 24%. This value is typical for this type of biomass (Rowell et al., 2005) while the total lignin content can vary from 18 to 35%. The presence of lignin can be a key factor in creating interactions between the fiber reinforcement and the polymeric matrix, due to the intensification of interactions, which can cause the transfer of charge from the matrix to the fiber during impact, providing greater resistance to the fiber. impact to the material.

Glucose and xylose present most of the carbohydrate content, which is in line with the work by de Simas et al., (2010), who found that glucose and xylose are the main carbohydrates in an analysis of the heart of palm flour.

The moisture content was 83.7% (w/w), indicating the need for prior drying of the biomass for proper application.

As observed in (Figure 2), the fibers are arranged in groups and are intertwined, as evidenced in Figure 2 (a). This means that the fibers are organized in clusters or bundles, forming a cohesive structure. Furthermore, in Figure 2 (b), it is possible to identify the presence of conducting vessels. These vessels have the function of transporting water and nutrients along the tree. In other words, they play an essential role in the plant's fluid and nutrient circulation system, allowing water and nutrients to be efficiently distributed throughout the entire structure of the palm tree (Rudall, 2007).

Biomass properties indicate that it is an interesting reinforcement material, due to its composition and structure suitable for mechanical resistance.

Thus, the production of eco-composites based on a polystyrene matrix reinforced with fibers from the stem of the Australian royal palm was proposed. Initially, polystyrene from commercial edible cups was dissolved in chloroform (CHCl₃) with subsequent addition of plasticizers (lignin or glycerol) and stem fiber as mechanical reinforcement.

The eco-composite produced showed rigidity, good aggregation and homogeneity. However, it was observed that the material is brittle and has low adherence to silicon, glass or granite.

Infrared spectrophotometry is a good technique for identifying the vibrational modes of different chemical bonds in the components of a material. This technique shows the possible changes in the conformational structure of the components and also indicates if there was formation of bonds between the components of the mixture.

As can be seen in (Figure 4), bands of aromatic vibrational modes CH and CH can be seen. In all samples, two bands are attributed to in-plane CH stretching of aromatic rings and out-of-plane CC bonding at 1500 cm⁻¹. The angular deformations of the out-of-plane CH bonds in the aromatic ring are perceived by a band at 750 cm⁻¹ and the band at 700 cm⁻¹ is attributed to the angular deformation of the out-of-plane CC bond in the aromatic ring.

When other components of the composite are added, there are no considerable changes between the spectral profiles and the spectrum of the polymer itself, due to the low content of additives in the analyzed samples. However, an increase in the intensity of the band at 3400 cm⁻¹ (attributed to OH groups in glycerol and lignin) and of the bands at 900-1000 cm⁻¹ (attributed to aliphatic CH) was observed.

The results obtained from the thermogravimetric analysis reveal important information about the thermal behavior of the materials under study. Firstly, polystyrene has a single degradation stage, indicating that its decomposition occurs continuously.

On the other hand, the PCL, PC and PL eco-composites demonstrate a different pattern, exhibiting two significant degradation steps. The first step corresponds to the degradation of the stem fibers, and it is important to note that this step results in a minimal mass loss, since the initial content of stem fibers in the material is relatively low, representing only 10% by mass. The second stage is related to the degradation of the polymeric matrix and is characterized by a more pronounced mass loss.

Mixtures containing glycerol, identified as PCG and PG, have an even more complex profile, with three distinct degradation steps. The first step involves the degradation of the stem fibers, similar to other eco-composites. The second stage, which occurs in the temperature range between 220°C and 273°C, is related to the degradation of glycerol. It is important to mention that Cordoba (2011) investigated the thermal decomposition of pure glycerol and identified that glycerol degrades between 125°C and 345°C.

It is noticeable that the addition of reinforcing materials decreases the thermal stability of eco-composites. This decrease

in thermal stability can be favorable to the application of extrusion. De Jesus et al. (2019) and Mulinari et al. (2017) found similar behavior for composites made from Australian king palm stem fiber and low-density polyethylene.

Analysis of variance (ANOVA) shows that the tensile index values showed significant differences between the eco-composites (critical $F > F$). Thus, the Tukey test reveals that the traction index of the PC sample was significantly different from the P sample ($p < 0.05$), while the stem fibers promoted a considerable increase in the traction index values when compared to the samples of P. The same behavior was observed in the PG samples in comparison with the P and PC eco-composites. In this case, glycerol plays an important role in improving the tensile strength of the polystyrene matrix. The mean tensile index of the PCL sample was significantly higher ($p < 0.05$) than that observed in the PCG samples. Tukey's test revealed a significant decrease in the tensile index of PCG samples when compared to PC, PG, PL and PCL eco-composites.

Analysis of variance (ANOVA) showed that the means of the modulus of elasticity showed significant differences ($F > F_{critical}$). Tukey's test showed a significant reduction ($p < 0.05$) in the modulus of elasticity of the PCL sample, when compared to the PG, P, PC and PL samples, and a significant increase when compared to the PCG sample. The PC elastic modulus was also significantly lower than the P value, revealing that the rod reinforcement decreases the elastic modulus, giving a more rigid material. The PG value was significantly ($p < 0.05$) higher among all samples; the PL sample was significantly ($p < 0.05$) lower than P and PG; and the PCG sample was significantly ($p < 0.05$) the lowest.

Analysis of variance (ANOVA) showed that the mean values of the elastic limit show significant differences ($F > F_{critical}$). Tukey test values showed that the PG sample had a significant increase ($p < 0.05$) in the elastic limit when compared to the other samples. The PL sample showed a significant lower elastic limit ($p < 0.05$) when compared to the P and PG samples. The PCG and PCL samples showed significantly lower elastic limits when compared to the P, PC, PG and PL samples.

Analysis of variance (ANOVA) showed that the mean values of the tensile strength limit showed significant differences ($F > F_{critical}$). Tukey's test showed a significantly higher value ($p < 0.05$) of the tensile strength limit for the PG sample when compared to the other samples. The PL sample showed a significant decrease ($p < 0.05$) in the tensile strength limit when compared to the P and PG samples. The PCG and PCL eco-composite samples showed a significant decrease in the tensile strength limit when compared to the P, PC, PG and PL samples.

In general, the physical-mechanical properties showed that the addition of 10% of stem fiber provided an increase in the traction index and a decrease in the modulus of elasticity when compared to isolated polystyrene.

5. CONCLUSIONS

This work showed that the Australian royal palm biomass is an interesting reinforcement material for polymer matrices, such as polystyrene, due to its considerable fiber and lignin contents, which play important roles in the distribution of traction and binding to the matrix, respectively. The lower thermal stability of eco-composites after the addition of stem fibers, glycerol and lignin, when compared to polystyrene, can provide good applicability to extrusion methods. This is due to the multiple degradation steps observed in the eco-composite thermograms, when compared to the single degradation step of polystyrene.


Stem fibers and addition of glycerol to polystyrene provided better tensile strength to the eco-composite, while all additives provided less rigidity to the polymeric matrix, conferring greater elastic deformation to the formed eco-composites, as shown in the results of the physical-mechanical tests.

The eco-composites developed in this work are a promising material for application in molding processes for different technologies, where renewable sources can partially replace plastics from fossil fuels.

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