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ANALYSIS OF THE MECHANICAL PROPERTIES: POLYESTER COMPOSITES VERSUS POLYESTER WITH AQUATIC MACROPHYTE ASHES

Fernanda Beatriz Aires de Freitas
LMEst - Structural Mechanics Laboratory, Federal University of Uberlandia, School of Mechanical Engineering, Av. João Naves de Ávila, 2121, Uberlândia, MG, 38408-196, Brazil.
fernanda.beatriz@ufu.br

Nilson Francisco da Silva
Ana Claudia de Melo Caldas Batista
Daniel Freitas Freire Martins
Federal Rural University of the Semi-Arid, Sítio Esperança II, Zona Rural, Caraúbas, RN, 59780-000, Brazil.
nylsonsilva23@gmail.com, ana.batista@ufersa.edu.br, dffm@ufersa.edu.br

Abstract. *This study evaluates the use of ash from two aquatic macrophytes characterized by the X-ray fluorescence technique and used as a filler in the preparation of a polymer composite. A glass plate mold was used to obtain the proposed materials. After the composites were fabricated, the specimens were cut using a laser cutting technique, and their mechanical and physical performance were evaluated for different percentages of particulate reinforcement. Our results obtained so far have shown that the inclusion of ashes on composites has not significantly altered their density. The addition of Pistia stratiotes ashes (5 %) has positively influenced its elastic modulus, with a 10 % increase when compared to composites manufactured from Resin. We also observe that composites manufactured from Pistia stratiotes have better properties than those made from Eichhornia crassipes. This might be associated with the greater metal percentages in the macrophytes composition. This proves that using Pistia stratiotes can be a viable alternative for the application.*

Keywords: *Aquatic macrophytes, polymer composites, mechanical and physical properties.*

1. INTRODUCTION

On account of the current environmental impacts, present-day engineering seeks solutions that can minimize its degradation, and standing-out phytoremediation, a method that makes use of plants to reduce concentrations of toxic composites in polluted environments (Urbaniak *et al.*, 2020), is considered to be an economical and not so invasive alternative (Ashraf *et al.*, 2019), furthermore, within aquatic environments, macrophytes are frequently used for that purpose (Queiroz *et al.*, 2020; Eid *et al.*, 2020).

Still, the biomass produced at the end of this process might represent an obstacle to its implementation (Patra *et al.*, 2020), and strategies for this technique's sustainable disposal are necessary. A possible answer for the matter is using this material in the production of polymer particulate composites. Different particles can be used in the production of composite materials, which are divided into two groups: large-particle and dispersion-reinforced composites.

Large particles, which can be millimeter-sized or larger, tend to hold back the deformation of the matrix around their common surfaces. Dispersion-reinforced composites, on the other hand, use nanosized particles, and the matrix holds the larger volume of load when subjected to mechanical stress. The reinforcement mechanism in this case takes place at the atomic level and consists in hindering the propagation of displacement paths along the matrix through the dispersed particles (Egbo, 2021).

Particles are generally used to improve some properties of these materials, such as stiffness, high-temperature resistance, thermal and electrical conductivity or insulation, friction reduction, surface wear resistance, machinability, and increased surface hardness (Berthelot, 2010). Therefore, the choice of particles and matrix depends on the desired properties and applications. For example, stone waste powder particles used as an epoxy resin matrix for the manufactured composite generate composites with low moisture absorption, high dielectric constant, and flexural strength (Sahu *et al.*, 2020).

In the literature, numerous authors have explored various materials in composite fabrication. For instance, Mamman and Ramalan (2020), utilized orange peel particulate, Reddy *et al.* (2020) used sugarcane powder and fiberglass as reinforcement in epoxy resin. Obasi *et al.* (2021) used coconut shell particles as reinforcement to manufacture polypropylene composites. Ighalo *et al.* (2021); Alweendo *et al.* (2020) focused on composite production using rice husk. Chlob and Fenjan (2022) made composites with wood powder, cow bone, date palm fiber, and sheep wool as reinforcement in an epoxy matrix. Souza *et al.* (2020) investigated cement powder, while Faria *et al.* (2020) of wood shavings waste. Pujar and Mani (2022) studied the development of composites using pigeon pea stalk. Lintz *et al.* (2022) developed a self-sensing cement composite with graphite, and Figueiredo *et al.* (2022) of quasicrystalline metal powder.

For this reason, our research suggests the reuse of phytoremediation residues (biomass), through the development of a composite material with polymer matrix, reinforced with macrophyte ashes from *Pistia stratiotes* and *Eichhornia crassipes* species. So, the pollutants may be attached to this new material and be removed from the environmental medium.

2. EXPERIMENTAL TESTS

In the present study, we analyzed the influence of the percentage rate of the ashes acquired from *Pistia stratiotes* and *Eichhornia crassipes* (as reinforcement) plants on the mechanical properties of the particulate composite, applying uniaxial tensile tests and physical characterization, and also through density test and X-ray fluorescence analysis, aiming to characterize them in terms of its constituents. For so, the ashes of *Pistia stratiotes* and *Eichhornia crassipes* were produced and analyzed, obtained after the calcination process on those plants. It was used as a matrix a polyester resin, manufactured by MANUCHAR, potted by HIDROGLASS EQUIP. AND CHEMICAL PRODUCTS LLC, having a gelation time of 40 minutes in adjustment to the process, and since it maintains a direct relation with ambient temperature, we have opted for a 0.4 % concentration of the catalytic regarding the amount of resin used in the procedure.

2.1 Reinforcement acquisition

During the acquisition process of the aquatic macrophytes, firstly the plants were collected, cleaned, dried with paper towels, dehydrated at 70 °C in a greenhouse with forced air circulation (SOLAB/SL-102) for approximately 48 hours, and then milled, trying to obtain a reduced size of the particles.

Secondly, they were put in porcelain capsules previously treated and calcined in a furnace oven (NOVUS/N1200) at 500 °C for 3 hours. After the calcination process was finished, samples were placed into a desiccator until they could achieve ambient temperature, then homogenized and milled using a mortar with a pistil, obtaining the required ash (Fig. 1). Finally, the ashes were analyzed through X-ray fluorescence analysis.

(a) *Pistia stratiotes*. (b) *Eichhornia crassipes*.

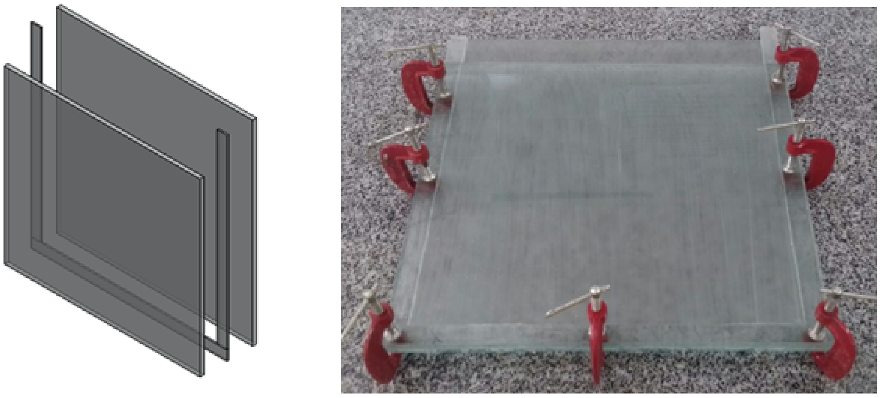
Figure 1. Ashes of aquatic macrophytes.

2.1.1 X-Ray fluorescence analysis

This analysis consists of a non-destructive method that identifies and quantifies the presence of elements contained in the studied material, through the acquisition of radiation emitted by these elements, due to their exposure to X-rays (Donais and George, 2018). For this reason, it was used to identify the chemical elements and their respective concentration proportions on ashes of *Pistia stratiotes* and *Eichhornia crassipes*. The samples were placed on extractable support composed of transparent polymer, to enable the passage and incidence of X-rays. Eventually, elements emitted characteristic radiations when submitted to proper stimulation, allowing their identification.

2.2 Manufacturing composite material plates

Our composite material plates were developed using a mold with glass plates. Figure 2 represents its montage scheme, in which it was used glass plates of 3 mm thick in a letter U shape to obtain an opening where the mixture of resin, ashes, and catalytic could be poured, guaranteeing a thickness of approximately 3 mm for each manufactured composite, and maintaining a thickness of 3.2 ± 0.4 mm, established by norms ASTM D638 (ASTM, 2014). Plates were attached using type C clips, see Fig. 2(b).



(a) Scheme. (b) Constructed.

Figure 2. Mold montage.

Initially, the resin and macrophyte ashes were weighed in required proportions (2.5 % and 5 %, regarding the sample's total mass) in a plastic container, then the mixture was homogenized and taken into a vacuum pump for 10 minutes, to eliminate the bubbles. Afterward, it was added 0.4 % of the catalytic, mixing manually for around 2 minutes, then it was once again put into the vacuum pump for another 10 minutes. Subsequently, the material was poured inside the mold, there remaining for 24 hours to achieve the curing process. Concluding the curing phase, the material was finally removed from the mold, resulting in the required plates (Figure 3). In the manufacturing of the pure resin plate used for comparison purposes, the material was purely polymeric and followed the same procedure described earlier, without the addition of ashes. Table 1 describes the used nomenclature for all fabricated plates.

Table 1. Nomenclature used for plates and test specimens.

Nomenclature	**Definition**
PR	Pure Resin
PS2.5	Composite with 2.5 % of charge from the *Pistia stratiotes* ashes.
PS5	Composite with 5 % of charge from the *Pistia stratiotes* ashes.
EC2.5	Composite with 2.5 % of charge from the *Eichhornia crassipes* ashes.
EC5	Composite with 5 % of charge from the *Eichhornia crassipes* ashes.

(a) **PR**. (b) **PS2.5**. (c) **PS5**. (d) **EC2.5**. (e) **EC5**.

Figure 3. Manufactured plates.

2.2.1 Density and uniaxial tensile

We determined the volumetric density according to the parameters of norm ASTM D792 (ASTM, 2008). It was manufactured five samples of each one of the plates, weighed on an analytical balance (SHIMADZU/AUY2020) with a maximum capacity of 210 g and resolution of 0.1 mg. For this test, we performed measurements of the composite's mass, dried and immersed in water, and with the achieved values it was possible to calculate the material's density.

The uniaxial tensile testing were conducted based on norm ASTM D63822 (ASTM, 2014), using a universal testing machine (EMIC/DL-10000) with a maximum capacity of 100 kN and speed of 5 mm per minute. Eight test specimens (Fig. 4) for each analyzed condition were manufactured and also analyzed while dried and immersed in water, obtaining five valid tests after the testing phase. Moreover, the universal testing machine provided values for the strength and strain of all test specimens. With the acquired data it was possible to calculate the maximum tension for the tensile (with the strength provided by the machine and test specimen's transversal section area), the strain (strain also provided by the machine, and test specimen's gage value, which represents 50 mm, as established by the norm) and at last, the elastic modulus (with 50 % of stress failure and calculated strain).

Figure 4. Test specimens.

3. RESULTS

3.1 Chemical categorization of macrophyte ashes per X-ray fluorescence

Our results achieved per X-ray fluorescence, which determines the composition of ashes, are presented in Fig. 5, where 22 elements were identified for *Pistia stratiotes* and 15 elements for *Eichhornia crassipes*. The elements contained within the plants are named: essential elements; benefit elements (Na, Si, Co, I, V); and toxic elements, also being named by heavy metals (Marschner, 1996).

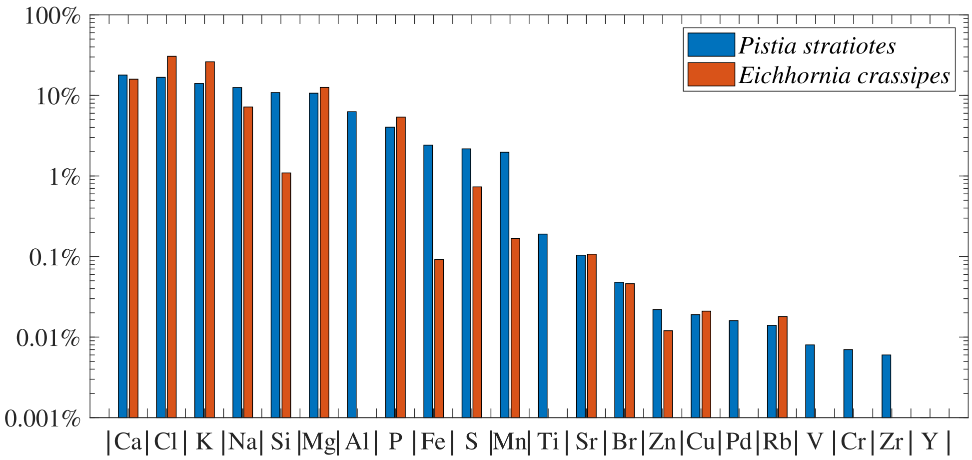


Figure 5. Composition of *Pistia stratiotes* and *Eichhornia crassipes* ashes.

Essential elements are divided into macronutrients (N, P, S, K, Mg, Ca), which are presented in greater quantity; and micronutrients (Fe, Mn, Zn, Cu, B, Mo, Cl, Ni), being presented in lesser amounts (Marschner, 1996; Jones Jr, 2012).

Heavy metals, such as Cu, Ni, Zn, Cd, Pb, Cr, Fe, Mn, are considered to be toxic metal pollutants in the environment and, even in low concentrations, they might cause severe toxic actions on plants (Ashraf *et al.*, 2019). Besides, some heavy metals, such as Cu, Fe, Mo, and Zn are micronutrients, required by plants in minimum quantities. However, they can cause toxicity when their levels exceed permitted limits (Saleem *et al.*, 2020).

Observing the results for *Pistia stratiotes*, it is possible to perceive that the elements with greater percentage detected through X-ray fluorescence analysis, are Calcium (Ca), Chlorine (Cl), Potassium (K), Sodium (Na), Silicon (Si), and Magnesium (Mg). While the elements in greater quantity found for *Eichhornia crassipes* are chlorine, potassium, calcium, and magnesium. These elements might be separated into two groups: macronutrients (Ca, K, Mg); benefit elements (Na and Si), as expected, taking into consideration that these elements are required for plants to grow; and micronutrients (Cl).

The greater presence of chlorine is inconsistent since it is a micronutrient (which is essential in lesser quantities). Although, it can be found on the plant's tissue because of its bioaccumulation capacity, and for being an element that plants can easily absorb (Pinto *et al.*, 2015; Câmara *et al.*, 2016).

The metal percentages, for example, Iron (Fe), Manganese (Mn), and Zinc (Zn), found in *Pistia stratiotes* ashes, were superior to the ones found in *Eichhornia crassipes*, giving more emphasis to iron, with a percentage rate of 2.417 % for *Pistia stratiotes* and 0.092 % for *Eichhornia crassipes*, due to the great difference on the values observed. Strontium (Sr), Copper (Cu), and Rubidium (Rb) presented similar values on both macrophytes. It was also observed that some metals contained in *Pistia stratiotes* ashes, like Aluminum (Al), Titanium (Ti), Palladium (Pd), Vanadium (V), Chromium (Cr), Zirconium (Zr), and Yttrium (Y), were not found in *Eichhornia crassipes* ashes.

In studies performed by Pinto *et al.* (2015) and Câmara *et al.* (2016), it is shown that part of these elements found on both *Pistia stratiotes* and *Eichhornia crassipes* ashes are found in the water of the river located in the same environmental area studied here. It is proving its potential for phytoremediation.

3.2 Volumetric density test

The results achieved by the volumetric density test for all specimens are described in Tab. 2, where it is possible to notice that volumetric density – when compared to the value acquired from samples of Resin – has shown a slight increase after adding the ashes, and low standard deviation in all cases, indicating consistency of the test specimens.

Table 2. Volumetric density samples.

Volumetric density [g/cm^3]					
Samples	**PR**	**EC2.5**	**EC5**	**PS2.5**	**PS5**
1	1.2267	1.2364	1.2613	1.2378	1.2578
2	1.2299	1.2354	1.2624	1.2374	1.2550
3	1.2274	1.2347	1.2641	1.2393	1.2564
4	1.2232	1.2358	1.2648	1.2391	1.2551
5	1.2280	1.2348	1.2662	1.2400	1.2597
Average	1.2270	1.2354	1.2637	1.2387	1.2568
Standard Deviation	0.0025	0.0007	0.0020	0.0011	0.0020

It stands out that the higher density increase was obtained with the addition of 5 % of *Eichhornia crassipes* (**EC5**), causing the increase of only 3 % in comparison to Resin (**PR**). This emphasizes that the addition of ashes will not result in significant changes to the density of the material. Still, the value of 1.2270 g/m^3 found for the polyester resin is similar to the ones shown in the research literature. For example, Baloyi *et al.* (2021) reached a value of 1.10 g/m^3, and Martínez-López *et al.* (2021) found values from 1.09 to 1.11 g/m^3.

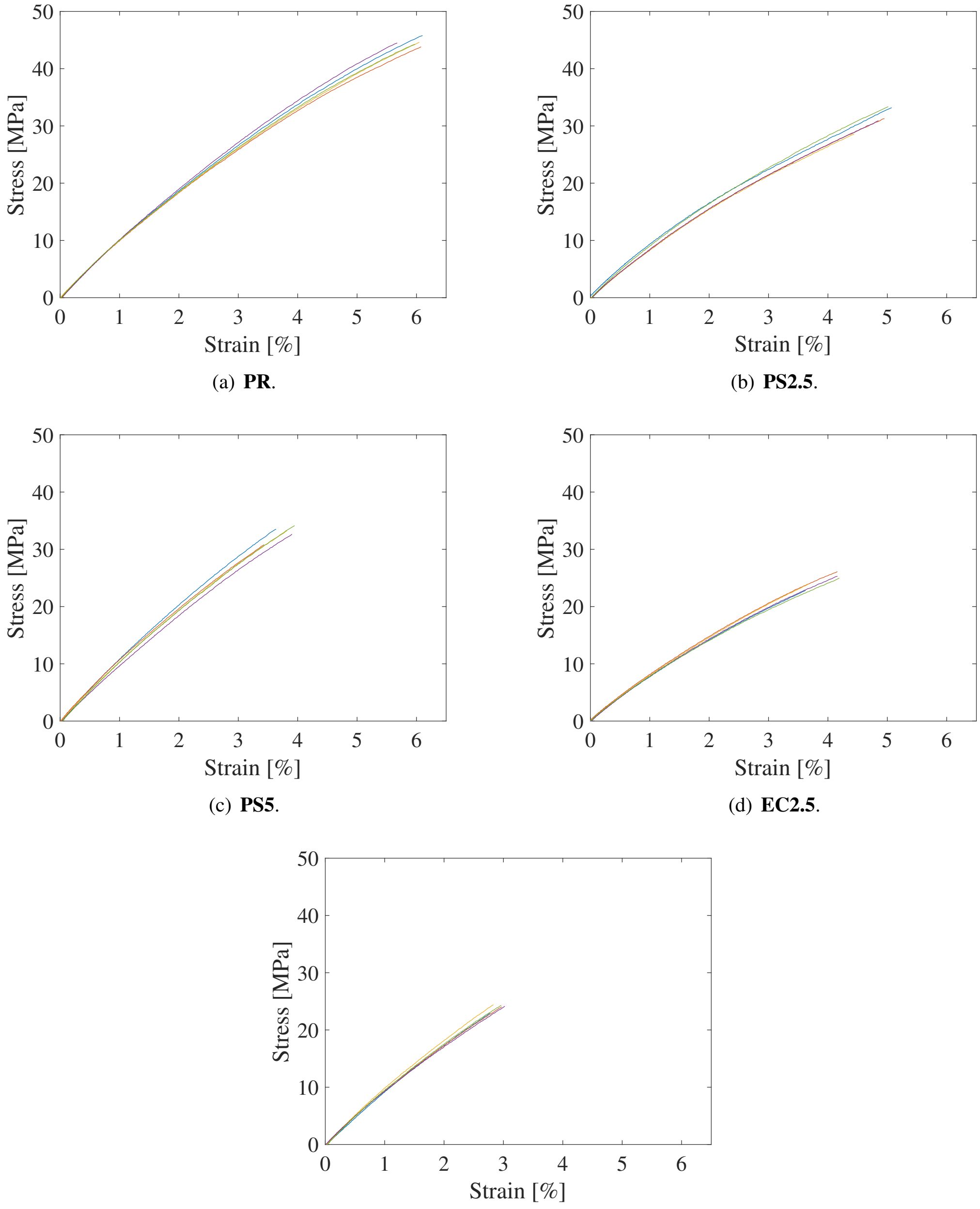
3.3 Uniaxial tensile test

Figure 6 represents the five valid stress-strain curves of the composites produced with Resin, and ashes of *Pistia stratiotes* and *Eichhornia crassipes*, in proportions of 2.5 % and 5 %. The behavior of all materials until reaching failure is similar, revealing that the addition of ashes has not altered their typical answer when submitted to this type of test. However, test specimens with *Eichhornia crassipes* ashes presented a significant reduction in values of stress, yet as the addition of 5 % of *Pistia stratiotes* (**PS5**), we obtained improvement on the elastic modulus, which provided more stiffness for the material.

The average values and their respective standard deviations, obtained for stress, strain, and elastic modulus of the five valid test specimens for each studied condition, are shown in Tab. 3. This one informs that, regarding the samples with 2.5 % of ashes in their composition, *Pistia stratiotes* revealed an increase of 30.14 % for strength; 25.78 % for strain, and 9.46 % for elastic modulus, when compared to **EC2.5**; and reduction of 25.97 % of maximum stress, 12.89 % of normal strain and 10 % of elastic modulus when compared with **PR**.

Table 3. Mechanical properties of test specimens.

Mechanical properties	**PR**	**PS2.5**	**PS5**	**EC2.5**	**EC5**
Maximum stress [MPa]	44.97±0.81	33.29±4.42	33.59±1.97	25.58±2.31	24.18±0.84
Normal strain [%]	6.05±0.26	5.27±0.76	3.86±0.35	4.19±0.52	2.95±0.16
Elastic modulus [GPa]	0.90±0.02	0.81±0.02	0.99±0.04	0.74±0.02	0.91±0.02



(a) **PR**.

(b) **PS2.5**.

(c) **PS5**.

(d) **EC2.5**.

(e) **EC5**.

Figure 6. Stress x Strain.

After comparing the samples with 5 % of ashes in their composition, we observed an increase for *Pistia stratiotes* concerning their strength (38.92 %), strain (30.85 %), and elastic modulus (8.79 %) in comparison to **EC5**; and reduction of 25.31 % for maximum stress, 36.20 % for strain and increase of 10 % for elastic modulus, when compared to **PR**. This means that the addition of *Pistia stratiotes* had a positive effect on the stiffness of the plastic when in comparison to *Eichhornia crassipes*. Although the test specimen with **PR** obtained greater strain and mechanical resistance, the modulus of elasticity, on the other hand, showed better results in **PS5**. This confirms what was presented by Jose *et al.* (2021), who state that biological waste reinforcements can be used to increase the modulus of elasticity of the composite fabricating when compared to the polymeric composite only, i.e., without the addition of particles in its composition.

The provided results explain that even with the ashes' percentage increase, composites with *Pistia stratiotes* have better properties in relation to composites with *Eichhornia crassipes*. It might be related to the composition of the ashes studied, through X-ray fluorescence analysis (Fig. 5), in which ashes of *Pistia stratiotes* revealed a greater quantity of metals within its composition.

Many researchers studied the influence of nanoparticles of metals or metallic oxides on composite materials. We can mention the works of Hezma *et al.* (2019), who used nanoparticles of zinc oxide on composites and managed to improve the thermal stability and mechanical properties (resistance to traction, elastic modulus, tenacity, and stretching until failure). Also, Sahmani *et al.* (2019), who added magnesium oxide and aimed for a significant increase in resistance to compression). Abu-Oqail *et al.* (2019) used a double matrix of Cu-Fe reinforced with graphene nanoplatelets (GNP) and achieved greater resistance to compression and lower deterioration rate for the samples up to 0.6 % of GNP.

Therefore, it is possible to notice that implementing metallic nanoparticles in composites is efficient to improve the mechanical properties of the manufactured material, which lead us to believe that better properties of composites with *Pistia stratiotes* ashes really account for higher metal percentages in their composition.

4. CONCLUSION

According to the results found for the tensile test, it was verified that test specimens (**PS2.5**, **PS5**, **EC2.5**, and **EC5**) when compared to the ones manufactured with **PR**, showed a reduction for maximum stress and normal strain. And that the test specimen **PS5** has presented a 10 % increase in its elastic modulus about the ones produced with **PR**. This is a positive result of the addition of 5 % PS in the manufactured composite. We can still observe that composites manufactured from *Pistia stratiotes* have shown better properties, opposing to the composites made from *Eichhornia crassipes*. This might be associated with the greater metal percentages in the macrophytes composition. This proves that using **PS5** is a viable alternative for application, as long as it is not required high mechanical strength, since all test specimens manufactured with the ashes have shown the worst mechanical resistance than the ones produced only with Resin.

5. ACKNOWLEDGEMENTS

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