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A STUDY ON THE MECHANICAL PROPERTIES OF SOME GREEN-COMPOSITES

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Abstract

In this paper, we determined some mechanical properties of a new category of green composites, based on tensile tests. More precisely, we selected a set of samples from a new bio-resin based on Dammar and two sets of samples from green-composites with matrix by bio-resin and reinforced with almost compact layers of *Typha latifolia* or *Schoenoplectus lacustris*. For these built materials, we obtained the characteristic curve, tensile strength, percentage elongation after fracture and elasticity modulus. In addition, for each sample, we observed the breakage area with an electronic microscope. The roughness profile parameters for a bio-resin specimen were determined. Based on the EDS analysis we obtained the chemical structure of the bio-resin. Finally, based on the properties of these green-composites, we proposed a few applications in civil engineering and medical fields.

Key words: chemical structure, green composites, mechanical properties, roughness

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1. Introduction

Some natural resins (vegetable or animal) reinforced with natural fibers (proteinic or cellulosic) generates certain green-composites (biodegradable). The "green" term is given in composites name due to their "friendly" properties with environment, since after collecting they can be recycled, or can be left to degrade very quickly without affecting the environment. These type of materials will represent a new generation of composite materials in the near future, more durable, lighter and more resistant than the existing ones. However, in order to achieve these performances, some shortcomings in exploitation of these composites must be removed. For example: natural fibers have a tendency to absorb external moisture and are less homogeneous than glass, carbon, or kevlar fibers; adhesion between natural fibers and bio-resin is weaker than in case of synthetic

components. For a part of these inconveniences are being made attempts to be eliminated by processing and treating both natural fibers and bio-resins.

Among the advantages of using green composites we can mention: low cost of manufacturing (compared to synthetic composites) because both fibers and bio-resins are abundant in nature, even in the form of waste; are totally biodegradable; have relatively good mechanical properties; processing equipment cost will be smaller because they will suffer less damage during operation and because of this it will be changed less often.

Up to now, the bio-composite materials have just natural reinforcement and the matrix is a synthetic resin (the most commonly used are polyester or epoxy resins). In many papers, for this type of bio-composites (in particular those reinforced with *Typha latifolia* and *Schoenoplectus lacustris*) were studied various properties (mechanical and optical; thermal

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and acoustic isolation; biodegradability and recyclability; low cost due to abundance in nature). Also, in these papers there are presented more ways for such materials practice usage (such as in aeronautical and automotive industry, or especially in civil and industrial constructions). In this regard, we can mention for example the following papers (Amuthan et al., 2017; Balciunas et al., 2016; Dedeepya et al., 2012; Dieye et al., 2017; Dittenber and Gangarao, 2012; El Omari et al., 2017; Hoi-Yan et al., 2009; Ibrahim et al., 2014; Joedodibroto et al., 1983; Kabir et al., 2012; Koronis et al., 2013; Ku et al., 2011; Kurzawska et al., 2014; Luamkanchanaphan et al., 2012; Malkapuram et al., 2008; Mei-Po et al., 2012; Mohanty et al., 2002; Mohanty et al., 2000; Oksman et al., 2003; Oksman, 2000; Ponnukrishnan et al., 2014; Ramanaiah et al., 2011; Rowell et al., 1996; Sana et al., 2015; Singh et al., 2015; Thakur et al., 2014; Wille Dick et al., 2017; Witztum and Wayne, 2014; Witztum and Wayne, 2016; Wuzella et al., 2011).

Although there are many studies on composite materials reinforced by natural fibers, few of them take into account the composites with a matrix of natural fiber and, particularly, Dammar. In (Zakaria et al., 2012) a new silicon and Dammar binder is put forward and the way in which the Dammar addition contributed to improving rigidity, modulus of elasticity and hardness is examined. In (Hamdani et al., 2018) the use of Dammar as PCM (Phase Change Materials) in concrete buildings is studied and (Fauzi et al., 2016) look into the possibility of using Dammar as additional material to improve thermal conductivity when preparing the material of changing the phase to composites. Pethe et al. (2013) have studied the mechanical characteristics, as well as the tensile strength, the elongation at break, the modulus of elasticity, and the moisture absorption characteristics of Dammar films which contain and which do not contain softening agent.

There is few papers which study the different properties of green composites both with the natural matrix and with reinforcement. Stanescu (2015) and Ciuca et al., (2017) studied the mechanical properties of green-composites based on Dammar bio-resin (with different percentages of epoxy resin for polymerization), reinforced with cotton, flax, silk and hemp fabric. Other studies pay attention to high-performance bio-composites based on nanotubes. In (Cavallaro et al., 2014) composite films, based on cellulose, chitosan and halloysite clay nanotubes, were prepared, using a solution casting method, which allowed for a uniform distribution of nanotubes within the material and provided control over the morphology of the composite. Pectin bio-nanocomposite films filled with various concentrations of two different types of halloysite nanotubes were prepared and characterized in (Makaremi et al., 2017) as potential films for food packaging applications. The paper of (Bertolino et al., 2018) is a contribution to the design of multilayer bio-composites based on halloysite nanotubes (HNTs) and chitosan.

2. Material and methods

2.1. Sample Preparation

Bio-matrix used to achieve green-composites was obtained by combining a ratio of 85 % Dammar natural resin with 15 % Resoltech 1050/1058 S (epoxy resin / hardener). The percentage of synthetic resin and hardener was necessary to initiate a rapid process of Dammar resin polymerization. A similar resin (but with a higher percentage of epoxy resin) was used in the paper (Ciuca et al., 2017).

In the first step of obtaining this bio-resin, we molded a plate from which we cut a samples set with 200 mm length, 25 mm width and 4.3 mm thickness, with densities according to (Stanescu, 2015), between 1.04-1.05 g / cm³. The individual weight of the samples ranged between 22.5-22.9 g.

In the second step, we made two plates of green-composites with matrix from bio-resin reinforced with:

- 8 almost compact layers of *Typha latifolia* leaves (the plate dimensions were: 250 mm long, 200 mm wide and 5.0 mm thick);
- 8 almost compact layers of *Schoenoplectus lacustris* leaves (the plate dimensions were: 250 mm long, 200 mm wide and 4.7 mm thick).

The plates small thickness resulted from the following action, carried out immediately after casting. More specifically, on each plate we applied a uniformly distributed force in a perpendicular direction to its surface with the value of 0.0343 MPa. The composite plates were made by superimposing three resin-imbued layers, which were compressed subsequently. The pressure was applied by means of a few calibrated weights of 1, 2, 5, 12 and 25 kilograms. The compaction pressure was calculated by dividing the pressure on the plate surface. The uniform thickness of the plate was obtained by employing gage blocks, on which the plate used for pressure was laid.

Individually, from these two plates, we cut a set of samples with the following dimensions: length 200 mm, width 25 mm. More specifically, we produced seven samples reinforced with *Typha latifolia* and eight samples reinforced with *Schoenoplectus lacustris*. The weight of the *Typha latifolia*-reinforced samples ranged from 16-16.5 g, and that of the samples reinforced with *Schoenoplectus lacustris* was between 17.1 and 17.6 g. We mention that the "almost compact layers" of *Typha latifolia* and *Schoenoplectus lacustris* leaves have an unidirectional orientation of fibers toward the tensile loading direction. We resorted to the expression "almost compact layers" because even though the cattail and bulrush leaves that we used stuck very well to the resin, they did not allow its passage from one side to the other. Therefore, in order to ensure the adhesion between the layers and avoid their exfoliation, when placing the leaves in a layer, an interspace was left in between so as to permit the resin to stick to both sides of the leaves.

In order to increase the green-composites performances, depending on the loading type, we can

modify the fibers orientation. For example, we can bind the *Typha latifolia* and *Schoenoplectus lacustris* leaves toward many directions in the same layer, or to dispose the reinforcement successive layers with a tilt angle between them.

In addition, we can noticed that the *Typha latifolia* leaf mass has a high porosity, low density of about $30\text{-}35 \text{ kg m}^{-3}$ (Georgiev et al., 2013) and lower thermal conductivity than polystyrene ($\lambda_{\text{Typha latifolia leaf}} = 0.032 \text{ W m}^{-1} \text{ K}^{-1}$ and $\lambda_{\text{polystyrene}} = 0.04 \text{ W m}^{-1} \text{ K}^{-1}$). In Fig. 1 we presented the samples set made from bio-resin reinforced with *Typha latifolia* (a) and *Schoenoplectus lacustris* (b).

2.2. Devices for testing and verification

With a PHENOM PURE PRO X electronic scanning microscope (which has high SEM performance for imaging and analysis, with the ability to determine the chemical composition and phases in the material structure, with a magnification capacity of 80 to 150,000 times, with an integrated spectrometer for analysis EDS) we determined the chemical composition of a bio-resin specimen. Also using the microscope, we performed a roughness analysis of a bio-resin specimen.

The roughness value is determined by calculating some parameters defined by ISO 4287 (1997). The distance between the electron gun and the analyzed sample was 4 mm and the beam energy was 15 KeV. All three sets of samples were subjected to tensile test with the 300 KN universal Walter Bai machine, which can be used for static and dynamic tests. This test was performed according to ISO 527-4 (1997). The strain rate was 20 mm/minute.

2.3. Main parameters studied

The parameters that characterize the roughness are:

R_z - average height of irregularity (represents the mean of absolute values of heights of the top five prominent

and the heights of lowest five goals, within the baseline length);

R_a - arithmetic mean of absolute values of the profile deviations within the boundaries of basic length.

Based on traction test of the three sets of samples, we determined the following mechanical characteristics:

- the yield stress $R_{p,0.2}$ which is the conventional mechanical stress σ that correspond to a specific elongation (of plastic nature), $\varepsilon = 0.2 \%$;
- the tensile strength (Eq. 1)

$$R_m = \frac{F_{\max}}{S_0}, \quad (1)$$

where F_{\max} is the maximum tensile force reached at the sample's breakage, and S_0 is the initial sample transversal section;

- the percentage elongation after fracture (Eq. 2)

$$A_f = \frac{[(L_u - L_0)100]}{L_0}, \quad (2)$$

where L_0 is the distance between the landmarks labeled before the test, in calibrated portion of each sample ($L_0 = 50.8 \text{ mm}$), and L_u is distance between the same landmarks after the sample is broken. The distance between the machine jaws is 100 mm.

For both *Typha latifolia* and *Schoenoplectus lacustris*, we determined the moisture coefficient based on the Eq. (3):

$$MC (\%) = \frac{[(m_1 - m_2)100]}{m_2}, \quad (3)$$

where m_1 is mass before drying, and m_2 is mass after drying.

Thus, we obtained $MC_T = 292.59 \%$ and $MC_S = 213.12 \%$ for *Typha latifolia* and *Schoenoplectus lacustris* respectively.



(a)



(b)

Fig. 1. The samples made from bio-resin reinforced with *Typha latifolia* – cattail (a) and *Schoenoplectus lacustris* – lakeshore bulrush (b)

3. Results and discussion

3.1. Roughness analysis

In Fig. 2 we showed the roughness analysis for a bio-resin specimen. Roughness parameters were shown in Table 1. The roughness analysis gives information on the surface quality of the studied materials, with applications for the finished products made of these materials. The results we obtained show that, for the studied resin, we can get good quality surfaces that do not need further processing.

3.2. EDS analysis

In Fig. 3 we presented chemical composition spectrum for a bio-resin specimen. This chart was obtained using the EDS analysis. Using the EDS analysis, we presented in Table 2 the chemical composition of a bio-resin specimen. It is notice that the main chemical elements in the composition are carbon, oxygen and nitrogen. For the others elements the atomic concentration is small. The EDS analysis shows the chemical elements in the composition of the natural resin under study.

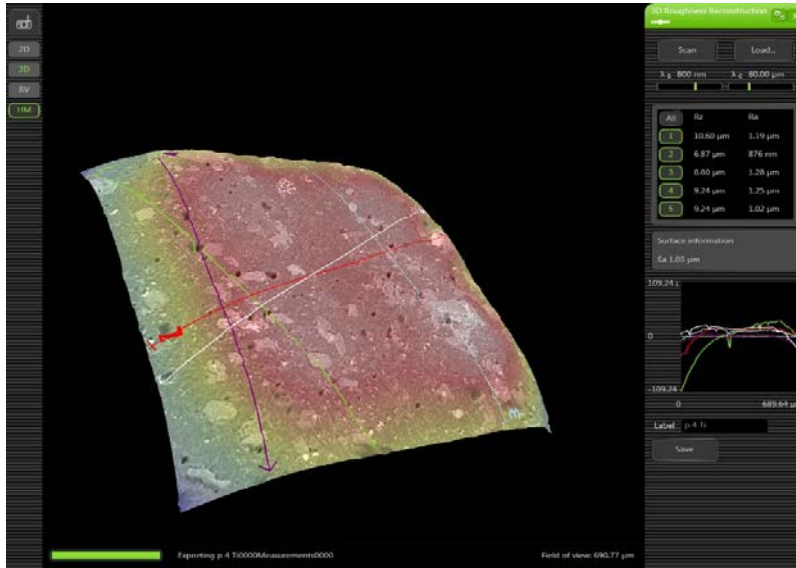


Fig. 2. The roughness analysis for a bio-resin specimen

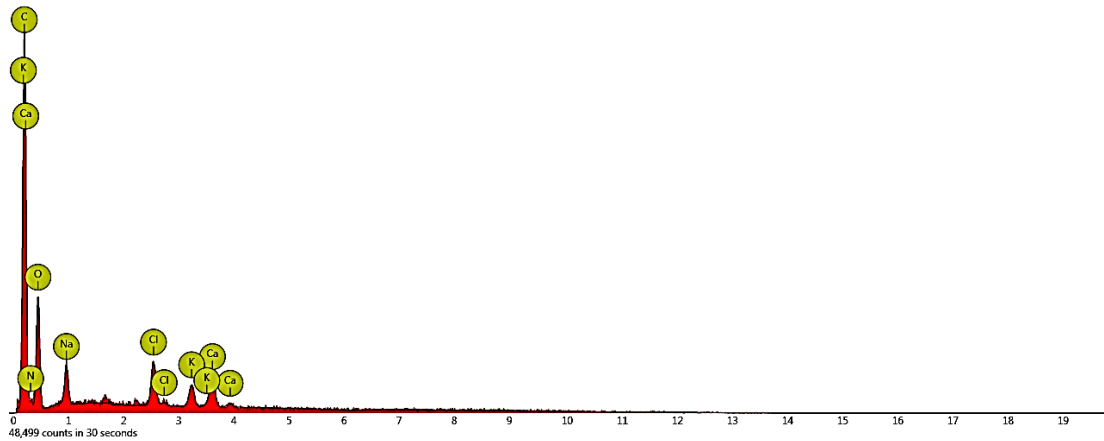


Fig. 3. The EDS analysis spectrum of chemical composition for a bio-resin specimen

Table 1. Roughness profile parameters

No.	R_z	R_a
1	10.60 μm	1.19 μm
2	6.87 μm	876 nm
3	8.80 μm	1.28 μm
4	9.24 μm	1.25 μm
5	9.24 μm	1.02 μm

Table 2 Chemical composition of a bio-resin specimen

Element Name	Element Symbol	Element Number	Atomic Concentration	Error [%]
Carbon	C	6	53.9	0.2
Oxygen	O	8	29.4	0.3
Chlorine	Cl	17	1.9	0.2
Calcium	Ca	20	1.9	0.2
Sodium	Na	11	2.3	0.2
Potassium	K	19	1.1	0.1
Nitrogen	N	7	9.5	1.5

The use of Dammar from different origin regions may lead to different properties. The properties may depend on the turpentine amount used for diluting the Dammar and, also, on the epoxy resin proportion, used with a view to obtaining polymerization points, which can lead to a hardening process that is short enough for industrial applications. The presented chemical composition is necessary so that the utilized resin may be identified because its modifications can lead to significant differences in the properties.

3.3. Tensile test results

Based on the tensile tests for the three samples types, we presented the obtained results in Table 3. As for the materials showing non-linearity from the start of the stress, the modulus of elasticity is calculated on the basis of the slope of the tangent to the characteristic curve. The experimental results shown in Table 3 are automatically determined by the software of the trial machine (NEXYGEN). We measured the main mechanical properties (tensile strength, elongation at break, modulus of elasticity, ratio of transverse contraction) of the studied materials because they are common in the study of the mechanical behavior of the composite materials. The energy stored during the tensile test is not an essential characteristic and the software of the tensile testing machine did not automatically give it.

Table 3. The main mechanical properties of test samples

Sample	Test sample dimensions (mm) $l \times w \times t$	Traction test results			
		R_m [MPa]	A [%]	μ	E [N/mm ²]
Bio-resin	200 x 25 x 5.3	21-22	1.98-2.24	0.49-0.61	1130-1220
Bio-resin reinforced with <i>Typha latifolia</i>	200 x 25 x 5.0	38.5-39.5	1.1-1.3	0.50-0.55	5959-6364
Bio-resin reinforced with <i>Schoenoplectus lacustris</i>	200 x 25 x 4.7	34-35	0.83-0.89	0.63-0.69	4174-4319

A statistical analysis is not necessary because the properties of natural materials, resin, or reinforcing material, depend on several factors (the picking area, weather conditions etc.). A statistical analysis would

highlight the average material properties that would be valid only for the studied sample sets. Other studies might lead to different results. This is why a presentation of the limit ranges within which the mechanical properties of the studied materials vary is preferable. Furthermore, by a representative sample we understood the sample characterized by average values of main mechanical properties studied.

Since the amount of epoxy resin used to initiate the polymerization process was less than in bio-resin obtained by (Stanescu, 2015), we obtained smaller elastic and strength properties of new bio-resin. In Fig. 4 we showed the characteristic curve for a representative sample of Dammar bio-resin. Fig. 5 presents the characteristic curve for a representative sample of bio-resin reinforced with *Typha latifolia* (a) and *Schoenoplectus lacustris* (b), while Fig. 6 shows the breakage area image for a bio-resin sample reinforced with *Typha latifolia* (a) and *Schoenoplectus lacustris* (b).

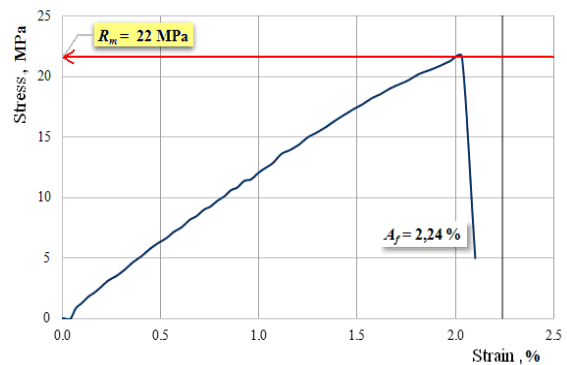


Fig. 4. The characteristic curve for a representative bio-resin sample

4. Conclusions

Based on the breakage area imaging analysis, we noticed that, both for samples reinforced with *Typha latifolia* and those reinforced with *Schoenoplectus lacustris*, the matrix breaking occurs simultaneously with fibers breakage. During the tensile test, due to low adhesion between the *Typha latifolia*, respectively *Schoenoplectus lacustris* layers and resin (which has a relatively low roughness), there was a slight delamination process.

Both in the case of samples reinforced with *Typha latifolia* and also *Schoenoplectus lacustris*, the reinforcement breaking elongation was reduced and thus the characteristic curve was almost linear.

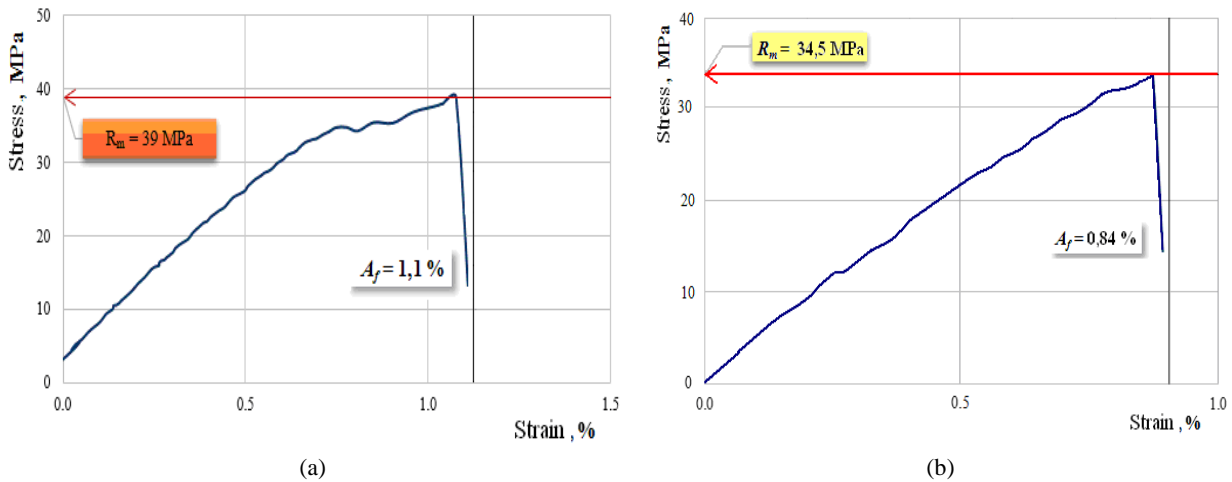


Fig. 5. The characteristic curve for a representative sample of bio-resin reinforced with *Typha latifolia* (a) and *Schoenoplectus lacustris* (b)

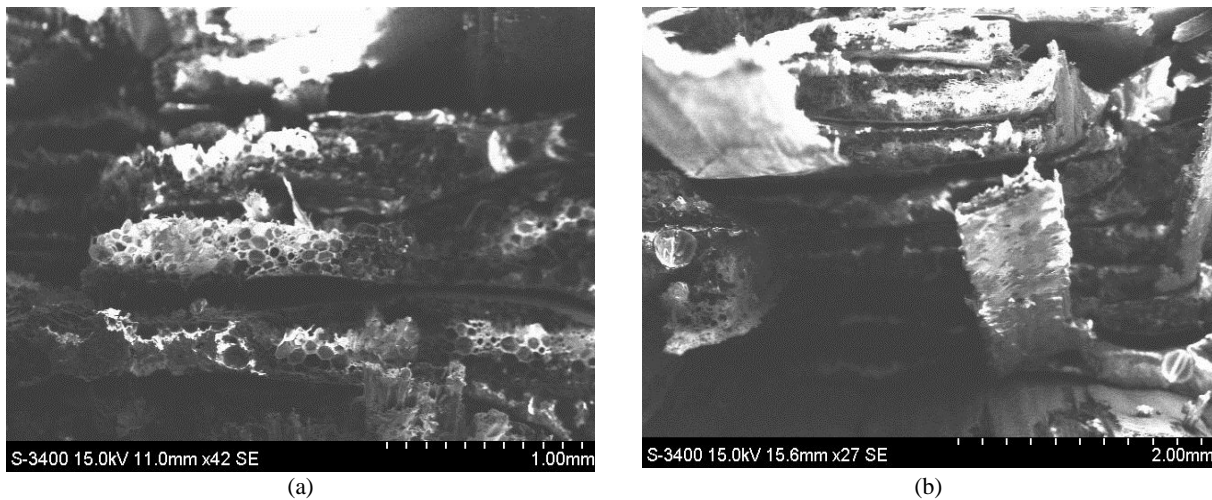


Fig. 6. The breakage area images for a bio-resin sample reinforced with *Typha latifolia* (a) and *Schoenoplectus lacustris* (b)

The explanation for this situation may be that the matrix and the reinforcement took over the external load simultaneously on the entire test duration. The differences on apparent flow limit cannot be high lightened. The ratio between the apparent yield stress limit and tensile stress was in mutual dependence with the fibers breaking elongation. More specifically, this ratio increased with the breaking elongation fibers value.

The behavior of fiber-reinforced composite materials, in sense of their rigidity and tolerance to destruction, it is influenced by the weaker separation interface between the matrix and reinforcement. In many cases, the existence of a weaker separation interface generates a tenacity increase for the composite material as is desirable.

An application where we intended to use these green-composites is in civil engineering. More specifically, we considered replacing laminate flooring, paneling, or possibly formwork structures. Among the advantages of using our composites, we can list: are renewable and bio-degradable, more attractive design, mechanical properties close to those

of laminate flooring, paneling and formwork currently used (made of wood or based on it). We obtained for the used composites almost the same mechanical properties as the ones that use the same reinforcement with synthetic resin.

A further advantage of using aquatic plants leaves, as reinforcement is their low density and consequently the low density of composites in which they are used. For example, in case of *Typha latifolia* leaves, about 85% of their mass is a parenchymatous tissue with large air-filled intercellular spaces (aerenchyma tissue). Therefore, the density of these composites decreases as amount of reinforcement increases and therefore, they will have a low weight, and thermal insulation properties which will be comparable to those of polystyrene.

Another application of these green-composites is manufacturing reusable bio-medical devices instead of the gypsum devices (to immobilize the fractures) and of some medical prostheses (hand, hip, leg etc.). The advantages of using of these devices are: lower production cost; will be better tolerated, more bio-compatible with the human body.

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