Study on the Behaviour of Two Original Biocomposites Subjected to Compression Test

IOANA IULIANA BUSUIOCEANU[1], FLORIN BACIU[2]*, GABRIELA DINU[2], ADINA NEGREA[1], GABRIEL JIGA[2]
[1]Politehnica University of Bucharest, Department of Mechanics, 313 Splaiul Independenței, 060042, Bucharest, Romania
[2]Politehnica University of Bucharest, Department of Strength of Materials, 313 Splaiul Independenței, 060042, Bucharest, Romania

Two types of biocomposite materials are made (original fabrication recipes) containing different resins as base: version A with natural resin and version B with polyester resin. For both of them the reinforcement element is a natural one namely fir needles. The samples are mechanically tested by compression in order to obtain the stress-strain curves and the elasticity longitudinal modulus.

Keywords: biocomposites, natural resin content, polyester resin content, fir needles, compression test

Nowadays, an increased demand to develop products based on natural fibers is encountered everywhere. Natural fibers abundantly found in nature are now considered as most suitable materials for use in various fields due to their enormous eco-friendly advantages and their renewable nature [1-3].

Biocomposites have replaced various conventional materials in several applications during the last decades. Some of the most significant benefits of using resins in place of the traditional materials are the facility of processing, no health hazards, low price, relatively high specific strength, light weight and productivity [4-6].

In the same time, the bio-fibers have represented a special attraction for different scientists due to the advantages that these fibers offer over what the conventional materials do. These biofibers present low density, easy availability, biodegradability, non corrosiveness, friendly to health possessing highly specific properties [7-9].

It has been observed that the behavior of natural fibers depends generally on different factors such as the source of fiber, weather variability, soil quality, climate and specific geographic location [10].

Natural fibers such as pine needles, flax, jute, straw, etc., all have been proved to be good reinforcements in resin matrices.

The commercial importance of the biocomposites has greatly increased, being used in the aerospace and automotive industry, civil infrastructure, where different products can be produced by using materials with fibres as reinforcement [11-13].

The present paper deals to create two types of samples, version **A** (using natural resin) and version **B** (using polyester resin) and then to test their mechanical characteristics.

Experimental part

Materials and Methods

As it has already said, the resin, as main component of a bio-composite, has two different proveniences: a natural resin and a synthetic one. Both of them are embedded with fir needles as reinforcement.

The natural resin (fig. 1) was collected from the fir forests situated in the APUSENI Mountains area. The percentage of impurities in the resin was relatively small (below 10%). The resin was melted in the oven (the melting temperature being 108^{o} C), then has been squeezed.

The second used resin was a pre-accelerated polyester resin, LERPOL TIX 3603. This type is known as an orthophthalic resin in a reduced styrene emulsion, thixotropized and pre-accelerated with a cobalt accelerator in 1% proportion, having a good mechanical strength.

**email: florin.baciu@upb.ro, Phone: +40726268078*


a b

Fig. 1. a) Natural flow of the resin and b) resin collection

Studies on the total content of the chemical elements showed no difference depending on the needle age or the distance from the source of the pollution. An examination carried in 2007 revealed that the total element concentration in one-year-old fir needles was higher than that in older ones [13]. The gross content of the mineral nutrition elements such as potasium, calcium, magnesium, phosphorus, manganese, iron, sodium and aluminium in fir needles samples was detected.

The fir needles were picked from the fir forests of the BUCEGI Mountains. They were naturally dried and then transformed into powder by means of an electric mixer. In figure 2b it is shown this powder.


a b

Fig. 2. a) Whole fir needles and b) Fir needles powder

For each of the two types of biocomposites (natural resin-version A; and polyester resin-version B), two types of specimens was manufactured in order to be tested:

a) whole fir needles in a percentage of 12.5% (fig. 2a);
b) fir needles powder in two different percentages of 12.5% and 25% (fig. 2b).

Biocomposites based on natural resin

The natural resin was mixed with each reinforcing component in part under the following conditions: the mixing time is 5 minutes because of cooling and rapid strengthening, the mixing temperature is 100 °C and the mixing velocity is 50 rpm. The mixture was then poured into stainless steel molds to obtain the specimens.

Due to the fact that the natural resin, collected directly from the tree, is quite soft, a BUTANOX® hardener has been used also in several percentages: 1%, 1.5% and 2%. BUTANOX® is the world's leading brand of the methyl ethyl ketone peroxides (MEKP). BUTANOX® is the most stable MEKP on the market, with guaranteed low water content.

The specimens representing natural resin biocomposites (version A) manufactured according to ASTM E9 - 09 (2018) are depicted in figure 3 and in figure 4.

a b

Fig. 3. Two types of version **A** specimens with the next fir needles powder content percentages:
a) 25%, respectively b) 12.5%

Fig. 4. Version **A** specimens with different whole
fir needles content percentages

Biocomposites based on the polyester resin

As stated above, the polyester resin is a pre-accelerated one, LERPOL TIX 3603. Regarding to hardener, MEK Peroxide is selected, used in different percentages: 1%, 1.5%, 2%.

Samples of the biocomposites consisting polyester resin as the main component (version **B**) were mixed manually with the two reinforcement materials (whole fir needles content and powder) and then they were poured into stainless steel molds.

All samples were tested at the compression test on the universal machine INSTRON 8801 (fig. 5).

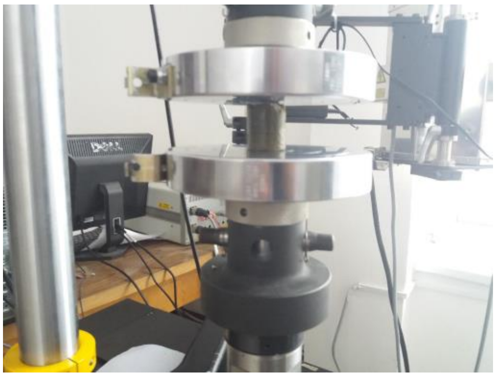


Fig. 5. Specimen mounted on the INSTRON testing machine

Results and discussions

Biocomposites based on natural resin, version ***A***

In table 1 four samples of biocomposites P1 to P4 are presented and for each of them the content of the fir needles-powder and the hardener content are specified.

Table 1
COMPOSITION OF THE BIOCOMPOSITES BASED ON NATURAL RESIN

BioComposite type	Fir needles	Hardener
P1	Powder 12.5 %	1 %
P2	Powder 25 %	1 %
P3	Powder 12.5 %	1.5 %
P4	Powder 12.5 %	2 %

All samples were mechanically tested for compression and below the stress-strain variation curves are plotted.

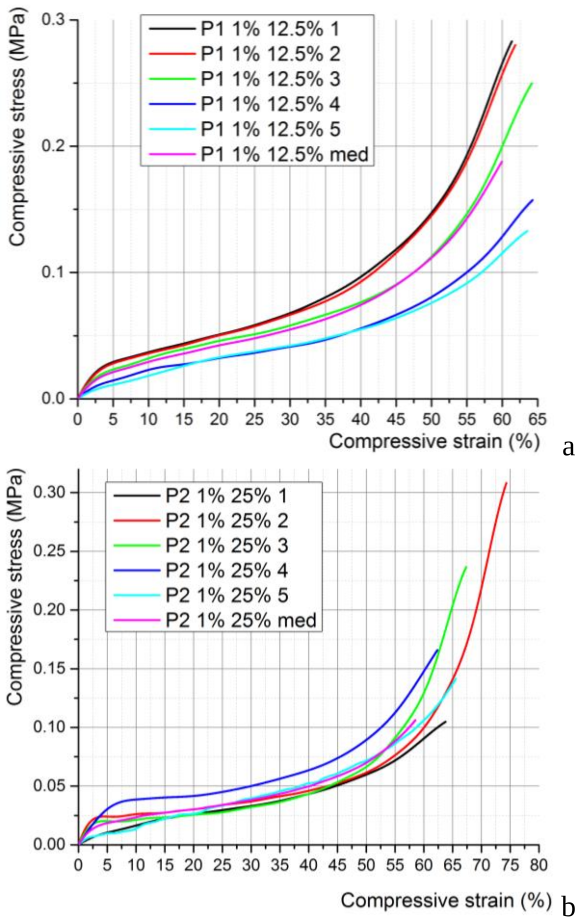


Fig. 6. Stress-strain variation for the specimens (version **A**) characterized by different fir needles powder content (12.5% a) and 25% b)) and with 1% hardener

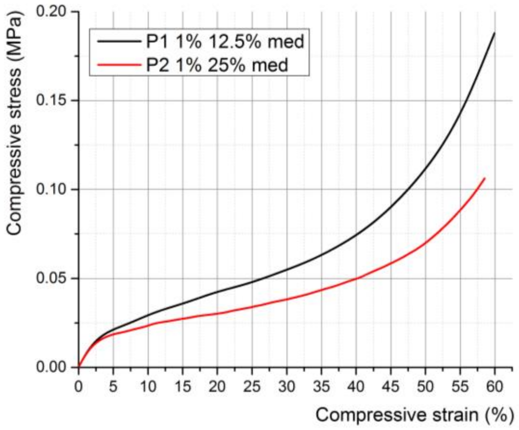


Fig. 7. Comparison between the mean values of the compressive test results for the specimens (version **A**) with 12.5% and 25% fir needles powder content and with 1% hardener

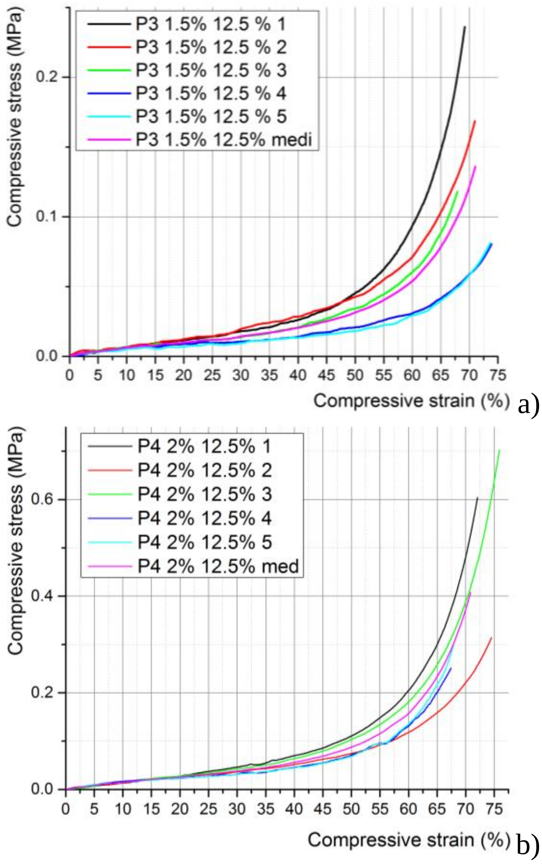


Fig. 8. Stress-strain variation for the specimens (version **A**) having 12.5% fir needles powder content and with 1.5% hardener a) respectively 2% hardener b)

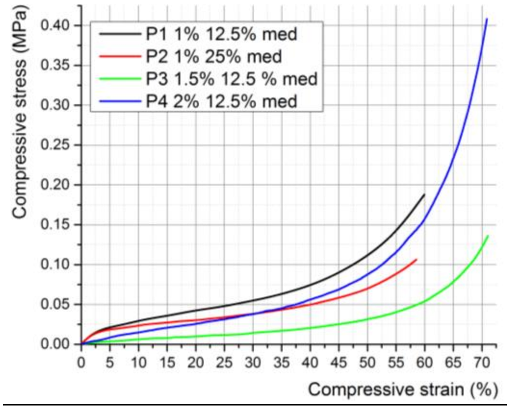


Fig. 9. Comparison between the mean values of the compressive test results for the specimens (version A) having 12.5 % and respectively 25% fir needles content and with different percentages of hardener

The modulus of longitudinal elasticity calculated for each biocomposite is shown in table 2.

Table 2
MODULUS OF LONGITUDINAL ELASTICITY

Bio-composite / Hardener concentration	Young modulus E [kPa]		
	1% hardener	1.5% hardener	2% hardener
12.5% reinforcement element (fir needles powder)	715.7	125.8	211.5
25% reinforcement element (fir needles powder)	698.7	-	-

*Polyester resin biocomposites, version **B***

For this version, six biocomposite samples were prepared, with whole fir needles or powder and different reinforcement and hardening material contents. The content of each sample is specified in table 3.

Table 3
COMPOSITION OF BIOCOMPOSITE VERSION B

Composite type	Fir needles	Hardener
P I	Powder 12.5 %	1.5 %
P Ib	Whole 12.5 %	1.5 %
P II	Powder 12.5 %	3 %
P3	Powder 12.5 %	2 %
P4	Powder 25.5 %	5 %
P5	Powder 25 %	3 %

Also, all of these samples were mechanically tested for compression and below the stress-strain variation curves are plotted.

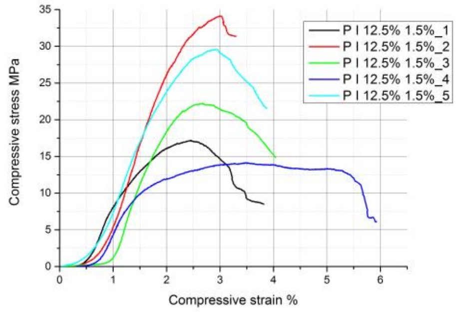


Fig. 10. Stress-strain variation for the specimens (version **B**, P I) having 12.5 % fir needles powder content and with 1.5% hardener

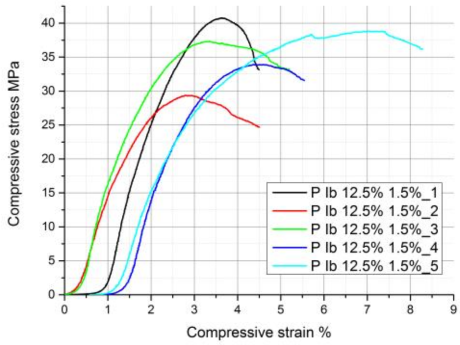


Fig. 11. Stress-strain variation for the specimens (version **B**, P Ib) having 12.5 % whole fir needles content and with 1.5% hardener

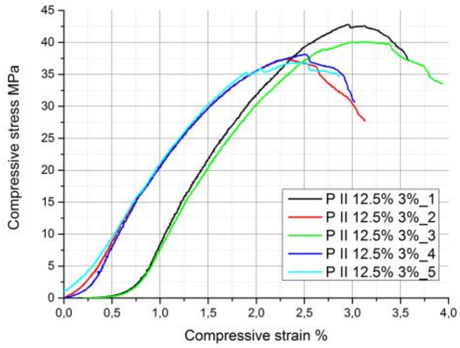


Fig. 12. Stress-strain variation for the specimens (version **B**, P II) having 12.5 % fir needles powder content and with 3% hardener

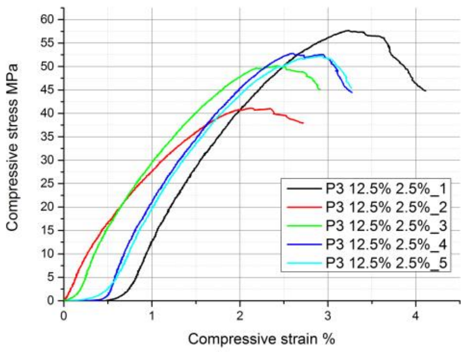


Fig. 13. Stress-strain variation for the specimens (version **B**, P 3) having 12.5 % fir needles powder content and with 2.5% hardener

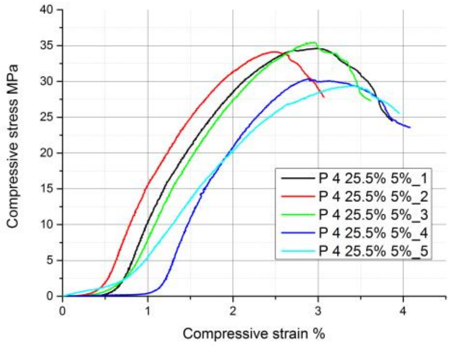


Fig. 14. Stress-strain variation for the specimens (version **B**, P 4) having 25.5 % fir needles powder content and with 5% hardener

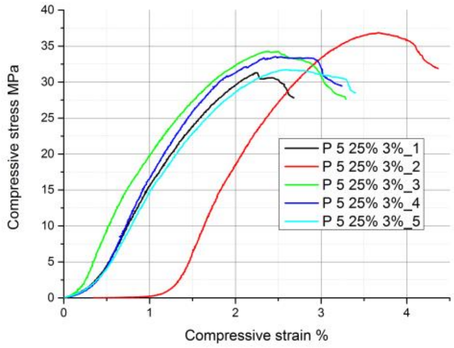


Fig. 15. Stress-strain variation for the specimens (version **B**, P 5) having 25 % fir needles powder content and with 3% hardener

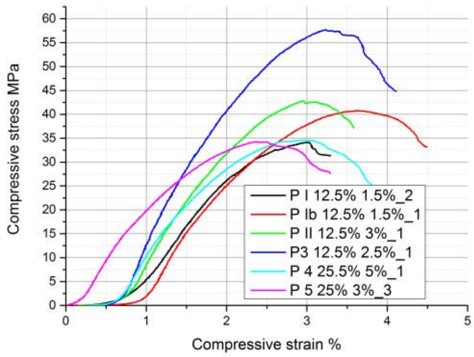


Fig. 16. Comparison between the mean values of the compressive test results for all specimens (version **B**) having different fir needles powder content and with different percentages of hardener

The modulus of longitudinal elasticity and the ultimate tensile strength calculated for each biocomposite are shown in table 4.

Table 4
MECHANICAL PROPERTIES

Biocomposite	Young modulus E [MPa]	Tensile Strength, Ultimate [MPa]
P I	1737.919	34.102
P Ib	2315.528	40.730
P II	2505.424	42.505
P3	2856.943	57.632
P4	2457.253	35.411
P5	2357.379	36.831

Conclusions

In order to ensure a better environmental protection it stands to reason that the technical materials in general and the bio-composites in particular should be entirely natural or at least partly natural. In this paper it is studied the behavior of two original types of bio-composites made by original fabrication recipes, the version **A** containing natural resin and the version **B** based on polyester resin but comprising a natural component as reinforcement element, fir needles. Actually, both versions carry fir needles as reinforcement material.

Testing the version **A** samples from the rheological point of view, one can conclude that any available information is not possible to be provided, due to the fact that the natural resin is sticky, adhering to the walls of the rheometer during the experiments. In conclusion, the main remarks are the following:

1. Doubling the reinforcement element concentration in the bio-composite matrix, the compression behavior of the material does not improve, the Young modulus having approximately the same value (715.7 kPa for the P1 composite sample meaning 12.5% reinforcement element and 1% hardener versus 698.7 kPa for the P2, meaning 25% reinforcement element for the same percentage of the hardener).

2. As one can see in figure 9, even if the hardener concentration has been increased, the elastic properties of the biocomposite are seriously affected. For a better result, it is essential that the mixture between the resin and the hardener to be made in a strictly stable ratio, ensuring that a complete chemical reaction takes place. If one of the two components are not mixed correctly, the final properties will be seriously affected after curing, phenomenon which could occur in reality due to the thermal instability of the natural resin.

3. The worst behavior was observed in the case of the 1.5% hardener concentration (P3 sample in figure 9), where the elastic modulus decreased approximately 5.6 times attaining the value of 125.8 kPa, much lower compared to 715.7 kPa corresponding to the 1% hardener concentration (P1 sample in figure 9).

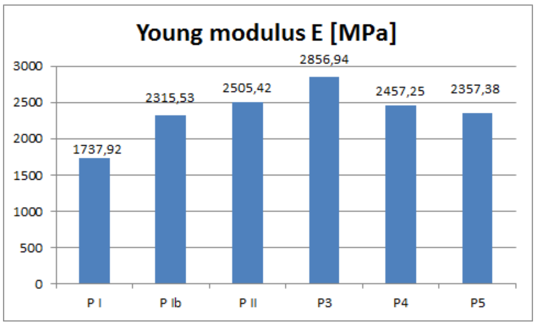
As much as we would like a 100% biodegradable biocomposite material, such as a material with a natural resin base is, this material cannot be yet used in industrial applications because it is very soft. Perhaps, over time, the organic chemistry will provide us a suitable hardener.

Hereinafter the version **B** samples are analyzed, these samples being the bio-composites based on polyester resin.

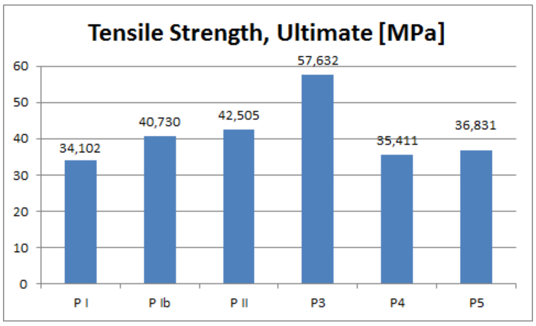
1. Figure 16 shows that all values of the compressive stress corresponding to the polyester resin bio-composites (version **B**) are much higher compared to those based on natural resin (version **A**): 58 MPa versus 0.4 MPa (see figure 16 and respectively figure 9).

2. Comparing the curves for the PI and PIb samples from the same figure 16 it can be seen that the use of the whole fir needles as reinforcement improves the behavior of the bio-composite compared to the other sort of reinforcement as the fir tree powder is. The compressive stress values are nearly 41 MPa for the PIb sample versus 34.1 MPa for the PI sample.

3. Increasing the percentage of the hardener for the polyester resin bio-composites, the compression behavior does not enhance if the percentage exceeds (see PI, PII, P3, P4 and P5 curves from figure 16).



a



b

Fig. 17. a) Young modulus variation and b) Ultimate tensile strength variation

Increasing the percentage of the reinforcement material, the compression behavior of the biocomposite does not improve and even its performance is weaker (the diagrams from figure 17 highlight such remarks concerning the samples PII-12.5% and respectively P5-25% where the percentage represents the fir needles powder concentration for the same percentage of hardener 3%).

Comparing to the other biocomposite materials tested in this paper, the biocomposite with the best behavior is P3 containing 12.5 % fir needles powder and 2% hardener, see figure 17.

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Natural fibers are particularly used in the automotive industry, where a number
of products can be produced by using natural fibers as reinforcement.

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