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MECHANICAL CHARACTERISTICS OF ARBOBLEND V2 NATURE COATED WITH SILVER NANOPARTICLES

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Abstract: Composite materials are an increasingly important resource having applicability in a wide range of fields such as automotive, aircraft and even medical one. Their purpose is to improve the functionality of the products by increasing their characteristics. In this sense, this manuscript highlights the mechanical characteristics of the Arboblend V2 Nature bio-polymer, reinforced (by coating the granules) with silver nanoparticles (AgNPs). With the incorporation of nano particles into the structure of the eco-friendly polymer, the resistance of the material to different types of tests changes, specifically, its properties decrease compared to the basic polymer due to the presence of discontinues introduced with the incorporation of silver nanoparticles. But this composite material it is a viable alternative for applications that require antimicrobial characteristics: food packaging, medicine, pharmaceutical industry etc. **Key words:** mechanical characteristics, AgNPs, biodegradable polymer, substitution, applications.

1. INTRODUCTION

The focus on hybrid/composite materials that can successfully replace traditional materials from a functional point of view occupies in the last years the market because they meet the quality requirements. And when referring to quality, it is not talking only about characteristics, whether mechanical or physical ones, a lot of emphasis is placed on the ecofriendly aspect of the newly developed composite materials.

Adding silver or copper nanoparticles in the polymer matrix is a versatile way to take advantage of their strong antimicrobial properties, thus producing new biocide materials and allowing a further expansion of the polymer applications range. These metals, in deficient concentrations, can be extremely toxic to bacteria found in different sectors of activity such as medical care, food packaging, agricultural applications, and industry in general. Compared to other antimicrobial agents, copper and silver are stable under industry-specific working conditions, being used as additives in different forms: micro/nanoparticles, salts, ions. hybrid structures etc. Nano-composites based on

polymer and Ag/Cu nano-powder can be obtained by direct incorporation of nanoparticles into the thermoplastic matrix (injection molding, coating) or by in situ syntheses of nanoparticles in a hydrogel etc., [1,2].

In this context, although there are studies in the literature referring to the incorporation of silver nanoparticles into different polymeric matrices, for instance: surfaces treated with silver ions - for stagnation/eradication of development, distinctive bacterial [3-6]; physical characteristics - depending on nanocomposites manufacturing, [7]; concentration of AgNPs in order to achieve higher antibacterial action, [8-12]; exposure of samples to different bacterial cultures, [13]; inhibition of bacteria/fungi reproduction, [14]; toxic effects to consumers when silver ions are released from AgNPs - pulmonary inflammation and cell necrosis, [15-17]. However, additional research is needed to support the development of new antimicrobial bio-polymeric materials that can be easily used on an industrial scale, [18].

The manuscript aims to investigate the potential advantage, in terms of mechanical characteristics, of parts obtained from injected Arboblend V2 Nature and AgNPs. More exactly, the composite material has in its structure a

lignin/PLA-based material (Arboblend V2 Nature - granules as substrate) and silver nanoparticles (AgNPs coating material) deposited through the Physical Vapour Deposition (PVD) method. The coating layer has very small dimensions - micrometers order, [19], as it was aimed at limiting as much as possible the content of AgNPs in order to maintain the highest biodegrading rate. It should be mentioned that the selected biopolymer as a substate has renewable resources in its structure, [20,21]. The evaluated mechanical characteristics were: tensile test resistance, bending test resistance, impact resistance, and dynamic-mechanical behavior.

2. MATERIALS AND METHODS

The used materials are Arboblend V2 Nature (polymer with lignin/ polylactic acid matrix) and silver nanoparticles - 99.99% purity (AgNP's). Arboblend V2 Nature was purchased from the Tecnaro company which produces and sells it in granular form, [22]. The coating of the granules with silver nanoparticles was carried out by using the PVD (Physical Vapor Deposition) method, on VS-40 MITEC PVD sputtering equipment. The coated granules were produced on SZ-600H injection molding equipment. The injection molding of the Arboblend V2 Nature + AgNP's bio-composite material was performed according to the experimental factorial research plan, using the ANOVA method, and the Minitab 17 software. The experiments were performed according to a complete factorial program of 2^3 type, in which the factors were the injection temperature - T_{top} [°C], the injection pressure - P_{inj} [MPa], and the cooling time – t_r [s]. The two levels for the three input factors are presented in table 1 which highlights the full factorial design used to inject the samples.

For each mechanical test, 3 samples were used in order to highlight the stability of the process through reduced values dispersion recorded for each individual data set.

The test conditions for the four types of mechanical determinations were:

The tensile test was realized on INSTRON 5587 equipment with an extensometer. The

samples were "dog bone" type, dimensioned according to ISO 527: 2, [23]. The process

Table 1

Complete	experimental	plan
Compress.	en per meene	P

Ewn	Input parameters			
no.	Ttop	Pinj	tr	
	[°C]	[MPa]	[s]	
1	155	80	15	
2	155	80	30	
3	155	100	15	
4	155	100	30	
5	165	80	15	
6	165	80	30	
7	165	100	15	
8	165	100	30	

parameters were: linear density - 100tex, sled weight -10 N, loading span -10 mm, support span -100 mm, span ratio -2, fixture type - 4point, extensometer - 0.0125 m.

The bending test was performed by using a three-point loading system, on INSTRON 5587 - equipment adapted for bending testing. The dimensions of the tested samples were in accordance with the ISO 178:2019 standard, [24], (80 x 10 x4) mm, the test was stopped at the time of complete sample failure. Bending tests were performed at room temperature. Process parameters were as follows loading span -10 mm, support span – 56 mm, and span ratio - 2.

Impact test was realized in accordance with SR EN ISO 179 standard, on a CHARPY Impact Tester, [25]. The settled test parameters were: temperature – 20 °C, energy – 5 J, speed - 2.9 m/s and hammer weight - 1.189 kg.

Dynamic Mechanical Analysis (DMA): To obtain specific samples for DMA analysis, samples with dimensions of (25x4x2) mm, standardized by the manufacturer of the DMA 242 Artemis NETZSCH equipment, were cut. Dynamic mechanical testing was performed using the three-point bending method to determine the viscosity of the composite material. The test temperature range was between RT (room temperature) and 373.15 K (100 °C), with a step of three Kelvin degrees, dynamic force of 5 N, strain of 50 μ m and frequency of 1 Hz.

3. RESULTS AND DISCUSSION

3.1 Tensile test

As was specified in the previous chapter, for each tensile test, three samples were used to highlight the stability of the process.

Each test generated responses on modulus of elasticity - E [MPa], elongation - ε [mm] and

tensile strength - σ [MPa]. Following the experimental results (average values and dispersion), highlighted in table 2, it was observed that the best results, the highest values of tensile strength, σ_{max} , were recorded in the experiment with number 1, (12.60 ± 1.73) MPa. The registered value was influenced by the set process parameters, in this case: injection temperature - 155 °C, injection pressure - 80 MPa, and cooling time - 15 s. The significant influence on the recorded results had the injection temperature.

Table 2

Experimental results of tensile tests - samples from Arboblend V2 Nature + AgNPs.

Exp.	Ttop	Pinj	tr	Samula	Ε	σmax	3
No. [°C]	[MPa]	[s]	Sample	[MPa]	[MPa]	[mm]	
			1	1528.49	10.60	0.78	
				2	1665.99	13.58	1.91
1	1 155	80	15	3	1503.48	13.63	1.24
				Average	1565.99	12.60	1.31
				StDev	87.51	1.73	0.57
				1	1665.23	3.62	0.61
				2	1448.76	6.87	0.54
2	155	80	30	3	1591.63	10.05	0.75
				Average	1568.54	6.85	0.63
				StDev	110.06	3.21	0.11
				1	1559.25	10.72	0.76
				2	1796.10	8.33	0.64
3	155	100	15	3	1868.31	4.76	0.38
				Average	1741.22	7.94	0.59
				StDev	161.67	3.00	0.19
		100		1	1609.34	4.99	0.50
			30	2	1554.45	10.64	0.83
4	155			3	1706.16	7.32	0.64
				Average	1623.32	7.65	0.66
				StDev	76.82	2.84	0.17
		80	15	1	1543.24	9.74	0.86
				2	1693.66	9.08	0.71
5	165			3	1539.41	8.34	0.74
				Average	1592.10	9.05	0.77
				StDev	87.97	0.70	0.08
		80	30	1	1533.91	9.41	0.82
				2	1610.29	10.93	0.90
6	6 165			3	1746.09	10.66	0.97
				Average	1630.10	10.33	0.90
				StDev	107.47	0.81	0.08
				1	1687.92	6.02	0.51
7 165	100	15	2	1819.94	13.25	1.07	
			3	1886.14	11.61	0.90	
				Average	1798.00	10.29	0.83
				StDev	100.91	3.79	0.29
		165 100	0 30	1	1665.38	12.65	1.05
				2	1862.37	9.52	0.80
8	165			3	1532.93	10.83	0.93
				Average	1686.89	11.00	0.93
			StDev	165.77	1.57	0.12	

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Analyzing the dispersion of the results for the three tested samples, from the best experiment according to the value recorded for tensile strength it can be concluded that the material Arboblend V2 Nature + AgNPs recorded a low dispersion of results, visible in figure 1 but also in table 2 (according to StDev). Regarding the shape of the curve specific to the tensile test (strength - elongation) but also to the low values of ε it can be stated that the rupture was sudden and the behavior of the material is fragile. Its fragility was given by the incorporation of the silver nanoparticles from the granules' surface layer in the structure of the bio-polymeric material. The effect of embedding was negative in terms of tensile strength because silver nanoparticles created discontinuities in the polymeric structure that led to the quick yield of the material under the action of progressive loads.

Comparing the tensile test results with those obtained for non-reinforced Arboblend V2 Nature, they are much lower, according to the literature the tensile strength values are approximately 44 MPa [21,26].

The possibility of substituting polymers based on fossil resources is not the purpose of this type of mechanical test, but the Arboblend V2 Nature + Ag composite material can replace from this point of view other biodegradable plastics (based on renewable resources) which do not have antimicrobial characteristics like this one.



3.2 Impact strength (Charpy test)

The results regarding the impact behavior of the composite material are centralized in table 3 and the graphical representation of the average



values recorded for the eight experiments is shown in figure 2.

The best value of shock resistance was recorded in experiment number 3 (3.75 ± 0.29) kJ/m², and experiment 2 showed a much weaker behavior, the value of shock resistance being almost 1.5 kJ/m² lower. The presence of vegetal fibers and/or lignin (as matrix) confers hardness the biodegradable composite material, to according to the literature, [27,28]. The complex lignin molecule offers viscoelastic hardness which is in accordance with the external cause of deformation. The lignin during the impact test tends to shorten the chemical bonds in order occupy the same volume. Another factor that led to obtaining these low values of impact resistance compared to other conventional plastics but also other biodegradable materials was the presence of silver particles in the structure of the biopolymer which weakened the stability of its molecular bonds.

Table 3 Charpy resistance values for injected samples from Arbahland V2 Nature - A g papaparticles

Al boblenu V2 Nature + Ag nanopai ticles					
	Samples			Avenage	
Exp. no.	1	2	3	[kJ/m ²]	StDev
1	2.19	2.19	2.19	2.19	0.0
2	2	2.63	1.56	2.06	0.53
3	3.5	3.69	4.06	3.75	0.29
4	3.25	3.81	3.87	3.65	0.34
5	4.88	1.75	1.25	2.63	1.96
6	3.38	3.75	3.38	3.50	0.22
7	4	3.06	2.88	3.31	0.60
8	2.19	3	3.12	2.77	0.51

The non-reinforced material, Arboblend V2 Nature, recorded average impact resistance values of (20-26) kJ/m². Regarding the possibility of replacing polymeric materials, this composite material can only replace other biodegradable materials such as Arbofill Fichte and Arboform LV3 Nature (biopolymers), [30,31].

3.3 Determination of the bending strength

Table 4 shows the average values of the three samples tested in each experiment. Due to the incorporation of nanoparticles in the Arboblend V2 Nature structure the behavior of the material became rigid, an aspect highlighted by the bending-displacement curve, figure 3, the rupture taking place suddenly. Also, the metal particles made the loading sustained by the rectangular samples very low, on average 1.5 N, and the test time very short only a few seconds (12s).

The best results of bending strength and displacement were recorded in experiment number 8: (32.95 ± 0.48) MPa and (4.47 ± 0.19) mm. Another experiment with results closes to those of experiment 8 was experiment number 4, being lower by only 2 MPa.

Table 4

26.76±1.38

24.59±0.81

each sample aspect visible in figure 3 but also in the standard deviation values for each output parameter, table 4. The results for the eight experiments are quite compact, the differences being larger only for experiments 8 and 4. The highest standard deviation is recorded by experiment 5, ± 2.11 MPa, as an effect of the technological parameters, which acted in reverse. Process parameters used for injection of

- 15 s, and high injection temperature – 165 °C. According to previous studies, the bending behavior of the reinforced material decreased compared to that of the base material, as was already mentioned due to the stiffening effect introduced by AgNPs. For example, the bending deflection parameter, in the case of the base material, was approximately 5.5 mm [32]. Regarding the possibility of substituting (based on this characteristic) some conventional plastic materials, it exists, but it will only be able to replace other polymers that have metal micro/nano particles in their structure.

the sample for experiment number 5 were: low

injection pressure 80 MPa, reduced cooling time

reinforced with AgNPs						
Exp.no.	ε _f [mm]	E _f [MPa]	σ _f [MPa]			
1	4.04±0.43	1706.98±58.53	26.15±0.06			
2	3.90±0.24	1723.57±50.81	26.73±0.26			
3	3.60±0.06	1730.04±69.30	25.52±1.63			
4	3.86±0.63	1878.10±184.91	30.49±0.68			
5	3 38+0 34	1901 70+13 78	25 44+2 11			

Randing test regults in ease of Arbahland V2 Nature

1819.52±6.49

1886.18±11.87

6

7

 3.44 ± 0.10

3.30±0.24

The values of these output factors were influenced by the injection parameters, namely the injection pressure at the second variation level, 100MPa, and the cooling time 30s. The higher pressure helped to better compact the biopolymer in the injection mold and the longer cooling time allowed the formation of closer intermolecular bonds.

The structural homogeneity of the samples and the experimental reproducibility is highlighted by the very close values obtained for



3.4 Dynamic Mechanical Analysis (DMA)

DMA diagrams, recorded during temperature scans, show variations in storage modulus (E') and internal friction during a heating cycle. The damping is determined as the ratio between loss modulus and storage modulus, $\tan \delta = E''/E'$. The behavior of Arboblend V2 Nature + AgNPs composite material during DMA analysis can be observed in figure 4. - 1260 -

The damping value of 0.6 recorded at a temperature of 65.9 °C denotes the rigid behavior of the material. Even so, the composite can replace in terms of $tan\delta$ value other biodegradable materials such as Arboblend V2 Nature reinforced with Extrudr BDP Pearl (biopolymer composite), $\tan \delta = 0.88$, [The damping value of 0.6 recorded at a temperature of 65.9 °C denotes the rigid behavior of the material. Even so, the composite can replace in terms of $tan\delta$ value other biodegradable materials such as Arboblend V2 Nature reinforced with Extrudr BDP Pearl (biopolymer composite), $tan\delta = 0.88$, [33], PLA-MFC (conventional fibrillated cellulose) $\tan \delta = 0.44$, PLA-MFLC (partially lignified and subsequently fibrillated beech wood) $tan\delta = 0.53$ and PLA-MFW (micro fibrillated wood) $\tan \delta =$ 0.33 [34].



Fig. 4. DMA termogram of the Arboblend V2 Nature + AgNPs composite.

4. CONCLUSION

The obtained composite material Arboblend V2 Nature + AgNPs highlighted during mechanical tests (tensile, impact, bending, mechanical analysis in dynamic regime) a completely different behavior from that of biobased polymer. The incorporation of silver nanoparticles has given material rigidity, creating structural discontinuities in the polymeric mass. Thus, the effects of AgNPs have put their mark on the output parameters specific to each type of mechanical test:

- from a tensile strength point of view the value decreased up to 3.5 times compared to the base material;

- in terms of bending strength, it was recorded a 24.5% lower value than Arboblend V2 Nature

not reinforced;

- the impact resistance and the material viscosity decreased due to the increased stiffness of the reinforced material;

- the standard deviations calculated for the tensile, bending, and impact average results, highlighted the uniform distribution of AgNPs in the polymer structure, as their values are quite low in the case of all the highlighted parameters; - the composite material withstands transverse stresses better than longitudinal stresses.

Even if the results were not the desired ones, the possibility of substituting polymers remains available, especially for those polymers which also have in their structure micro/nano metal particles. Also, the advantage offered by the composite material (created by coating the polymer granules with AgNPs) must be taken into account - antimicrobial characteristics [19].

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CARACTERIZAREA MECANICĂ A ARBOBLEND-ULUI V2 NATURE ACOPERIT CU NANOPARTICULE DE ARGINT

Materialele compozite sunt o resursă tot mai importantă având aplicabilitate într-o gamă largă de domenii industrial cum ar fi industria auto, aerospațială și chiar domeniul medical. Scopul acestora este de a îmbunătăți funcționalitatea produselor prin creșterea caracteristicilor acestora. În acest sens, prezentul manuscris scoate în evidență caracteristicile mecanice ale bio-polimerului Arboblend V2 Nature, ranforsat (prin acoperirea granulelor) cu nano particule de argint (AgNPs). Odată cu încorporarea nanoparticulelor de argint în structura polimerului eco prietenos rezistența materialului la diferite tipuri de încercări se modifică, mai exact, proprietățile sale scad față de polimerul de bază datorită prezenței discontinuităților introduse odată cu încorporarea nanoparticulelor de argint. Cu toate acestea acest material compozit rămâne o alternativă viabilă pentru aplicații care necesită caracteristici antimicrobiene: ambalaje alimentare, medicamente, industria farmaceutică etc.

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