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Optimization of PHBV-Hemp Fiber Biocomposite Manufacturing Process on the Selected Example

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ABSTRACT

In this work, a modern biocomposite on the base of poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) matrix was produced in the extrusion process, containing 30% by weight of hemp fibers. The use of the above-mentioned filler allowed to reduce the producing costs of the composite material compared to pure PHBV, improving, among others, some mechanical properties of products made of this biocomposite while maintaining full biodegradation. The obtained biocomposite can be successfully used for the production of injection molded products, but its processing properties are not yet fully known and consequently it is difficult to obtain the optimal performance properties of the products. As part of this study, the process of optimization of the production process of products from the PHBV-hemp fiber biocomposite was carried out on the example of samples intended for testing in the uniaxial tensile test. By using orthogonal planes, widely used in optimization process, the required number of injection molding tests was reduced. Input data values were determined by the factorial planning method that is commonly used in designing experiments. The calculations were carried out in the Minitab 18 software. Six controlling factors were used in the analyzes, each of which was subject to changes on three levels. When selecting the range of controlling factors, it was initially assumed that for all assumed levels of variability it must be possible to fill the mold cavity completely. The orthogonal plan of the L27 type was used in the research. For the purposes of the method, an orthogonal table was built containing 27 combinations of parameters subject to optimization. Optimization was undertaken for two main criteria: shrinkage of the „dog-bone" samples (primary and secondary volumetric shrinkage), mechanical properties (Young's modulus, tensile strength, elongation at break). By means of Taguchi method, a significant improvement of some product mechanical properties made of biocomposite was noted and the effective reduction of the processing shrinkage was observed.

Keywords: biocomposites, PHBV, hemp fibers, optimization, injection molding.

INTRODUCTION

Humans have long used biodegradable biocomposites of natural origin, relying on intuition and experience. In the Middle East from 800 B.C.E. clay bricks were reinforced with straw. Such bricks dried in the sun reached the compressive strength of approx. 25% of the value for fired bricks. Mongolian bows were made by joining with glue: wood, animal tendons and silk. The use of natural polymers is also not new, e.g. paper, silk, etc. have been used from historical times to the present [1÷4].

In the twentieth century, the use of natural polymers was significantly reduced due to the development of a wide range of cheap synthetic polymers and their composites, based on raw materials derived from crude oil. These materials are now well known worldwide in a wide range of applications due to their good performances, mechanical properties and long durability. In the last decades, polymer composites reinforced with synthetic fibers have started to enjoy research and engineering interest. Their main advantages should be presented, such as: high fracture strength, anti-corrosion properties, high strength-to-weight

ratio. Their disadvantages should also be mentioned. In these materials, it is difficult to separate the fiber from the matrix after use, they are not biodegradable, there is a health risk when inhaled during production, moreover, the production is characterized by high energy expenditure. Moreover, the use of large amounts of polymers reinforced with synthetic fibers has led to the problem of their waste management and recycling [5÷9].

The introduction of regulations on the appropriate collection and processing of biodegradable and composted polymers may encourage the development of technology for the development of new materials based on natural resources - materials of natural origin such as cellulose fibers and green polymers, because after fulfilling their application, they can be decomposed to the first factors. This type of waste is also a source of carbon and many other substances that are further used, e.g. for the re-production of green polymers and natural fibers. Additionally, cellulose fibers have some positive features, such as: lower tooling abrasion during processing, they are inexpensive to produce, they do not cause breathing problems for workers employed in processing companies [5, 10÷12].

Due to the above-mentioned issue, advanced biodegradable polymer materials of natural origin are sought and developed. One of such materials is PHBV (poly(3-hydroxybutyrate-co-3-hydroxyvalerate) - a natural biopolymer, fully biodegradable produced as a backup material in the mitochondria of bacteria. The wider commercial application of this biopolymer is still difficult due to the high production costs, as well as the small difference between the melting point and the degradation point of this polymer, and low flexibility and quite high brittleness [13÷16]. For this reason, further plans in the research of scientists are to improve the mechanical properties and processing window of this biopolymer, as well as the possibility of producing composites with the PHBV matrix [17, 18].

The tensile strength as well as the Young's modulus of natural fibers such as kenaf, hemp, flax, jute and sisal are usually lower than the glass fiber used in composites. However, the density of glass fiber is high, approximately 2500 kg/m^3, while the densities of natural fibers are much lower (~ 1500 kg/m^3). This becomes especially important in the case of the weight of products made of composites, where the weight must be significantly reduced [5, 19]. The mechanical properties of plastics are in most cases improved by adding fibers to the polymer matrix, since the fibers have a much higher strength and stiffness than the polymer matrix. Therefore, the influence of the fiber content on the strength properties of fiber reinforced composites is of particular interest and importance to many researchers [20÷24].

One of the possible ways of commercializing green composites, and PHBV in particular, may be the use of natural fibrous fillers in a biopolymer matrix. As a result of their use, it is expected to improve the mechanical properties of the formed composites while maintaining full biodegradability and with reduced production costs compared to pure biopolymer [25÷29]. One of the possibilities for the development of the current trend may be the use of short hemp fibers as a PHBV filler in order to produce, test and optimize some of the properties of the newly produced biocomposite. Once such a biocomposite has been manufactured, it is important to optimize the processing parameters of manufacturing process. For this purpose, factor planning methods are used, such as Taguchi orthogonal plans. The aim of the research presented in the paper was to produce a modern biocomposite and to analyze its selected mechanical properties for products manufactured with the use of optimized processing parameters.

RESEARCH MATERIAL

PHBV under the trade name Enmat Y1000 as a powder was used as the polymer matrix. The molar proportion of HV in the biopolymer is 8%, the density of the biopolymer is 1250 kg/m^3, and the softening point ranges from 165 °C to 175 °C. Hemp fibers with a length of approx. 1 mm and an average length-to-diameter ratio (L/d) of approx. 10 were used as the filler in the polymer matrix. The produced biocomposite contained hemp fibers with a mass fraction of 30%. A 10% aqueous sodium hydroxide solution was used for the surface modification of the fibers.

PRODUCTION OF BIOCOMPOSITES

The PHBV-hemp fiber biocomposite was produced by means the extrusion process using a ZAMAK EHP-25E single-screw extruder. The extrusion speed was 100 rpm. The machine's adjustable parameters are presented in Table 1. Extrusion

Table 1. The temperature of extruder heating zones

Head	Zone 3	Zone 2	Zone 1	Feed hopper zone
175	170	160	150	35

Table 2. Initial processing parameters of injection molding process

Parameter	Value
Mold temperature [°C]	85
Melt temperature [°C]	185
Cooling time [s]	25
Packing time [s]	25
Packing pressure [MPa]	30
Injection speed [cm³/s]	35

was carried out using an technological line for extrusion equipped with a cooling bath and a granulator. The temperature of the water in the bath for cooling during the granulation of composites was kept at 30 °C. It should be noted that both the polymer matrix and the plant fibers were dried for 3 hours at a temperature of 90 °C before the extrusion process. The obtained granulate was used for the production of specimen applied for testing of mechanical properties. Specimens were produced by means of injection molding process.

INJECTION MOLDING OF SPECIMENS

A DrBoy 55E injection molding machine equipped with a Priamus system for monitoring and controlling the injection molding process was used for the injection molding of the samples. The tests used an injection mold with inserts for uniaxial tensile test specimen (in accordance with EN ISO 527-1). The "dog-bone" geometry of specimens were pre-fabricated using adjustable parameters listed in Table 2.

RESEARCH METHODS

The Zwick Z030 testing machine was used to determine the strength properties of the obtained composites. The uniaxial tensile test was carried out in accordance with the EN ISO 527-1 standard for specimens with "dog-bone" geometry. Each series of specimens consisted of 7 pieces for subsequent statistical analysis. On the basis of the obtained test results, the following were analyzed: Young's modulus (E), tensile strength (σ_M), elongation at break (ε_M). The results were statistically processed, where the arithmetic mean (AM), standard deviation (SD) and the coefficient of variation (CV) were determined.

The shrinkage of the obtained details with the "dog-bone" geometry was tested on the basis of the EN ISO 294-4 standard. The primary shrinkage was tested after approx. 3 hours, and the secondary shrinkage after approx. 14 days from receiving the "dog-bones" specimens.

MECHANICAL PROPERTIES COMPARISON

The properties of the obtained composite (marked with the symbol K30) were assessed and compared with the given set parameters (Table 2) in relation to the properties of pure PHBV (Enmat Y1000) and polypropylene (Moplen HP648T). The strength properties obtained in the uniaxial tensile test were analyzed (Table 3). Representative stress-strain characteristics are shown in Figure 1. A significant increase in the value of Young's modulus in relation to polypropylene - by approx. 348% and tensile strength by approx. 24% was observed. On the other hand, compared to pure PHBV, the Young's modulus value increased by approx. 167% and the tensile strength by approx. 21%. The significantly lower value of elongation at break compared to both unfilled polymers may result from the reduced proportion of the polymer matrix in the biocomposite.

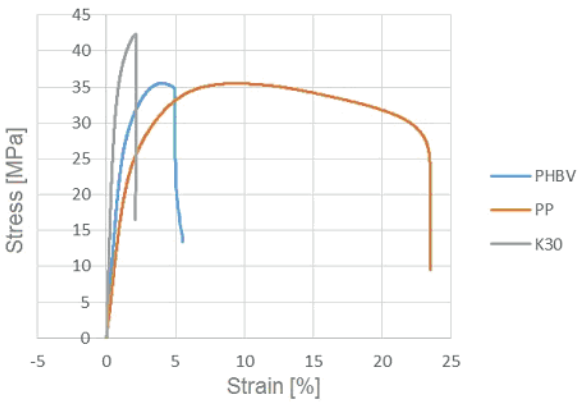

Fig. 1. Stress-strain characteristics of the K30 biocomposite, PP and pure PHBV

Table 3. Results from the uniaxial tensile test of the K30 biocomposite, PP and pure PHBV

Type of material	Statistics	E [MPa]	σ_M [MPa]	ε_M [%]
PP	AM	1561.61	34.68	9.48
	SD	24.65	0.69	0.27
	CV	1.58	1.98	2.85
PHBV	AM	2617.37	35.48	4.12
	SD	112.02	0.86	0.15
	CV	4.28	2.42	3.63
K30	AM	6992.31	42.90	2.28
	SD	199.44	0.71	0.06
	CV	2.85	1.65	2.60

A comparative analysis was also made of the shrinkage values of samples with the „dog-bone" geometry (Fig. 2). The use of hemp fibers in the PHBV matrix significantly reduces the value of the volumetric shrinkage (primary by about 71% and secondary by about 81%) compared to pure PHBV. Moreover, for the obtained biocomposite, lower shrinkage values (primary by approx. 69% and secondary by approx. 44%) in relation to polypropylene were noted.

The use of hemp fiber with a mass content of 30% results in a significant improvement in most of the mechanical properties in the uniaxial tensile test and the shrinkage values in relation to pure PHBV and polypropylene. Moreover, the addition of natural fibrous filler to PHBV allows for full biodegradation and enables the use of widely available and cheap hemp fibers in order to reduce the costs of producing a PHBV based biocomposite. Therefore, when obtaining a biocomposite with interesting and satisfactory mechanical and functional properties, it is worth optimizing in the direction of further improvement of mechanical properties and reduction of processing shrinkage of moldings.

OPTIMIZATION OF THE INJECTION MOLDING PROCESS

The production analysis of composite products was limited to the injection molding technology, in which samples for uniaxial tensile tests were produced. In order to optimize the analyzed injection molding process, the Taguchi method was used. Input data values were determined by the factorial planning method that is commonly used in designing experiments. The calculations were carried out using the Minitab 18 software. Six controlling factors were used in the analyzes, each of which was subject to changes on three levels (Table 4). When selecting the range for the controlling factors, it was initially established that for all assumed levels of variability it must be possible to fully fill the cavity. The range of input parameters was established on the basis of injection parameters already used in order to obtain a biocomposite containing 30 wt. hemp fiber.

Due to the use of Tauguchi orthogonal arrays, widely used in process optimization, the required

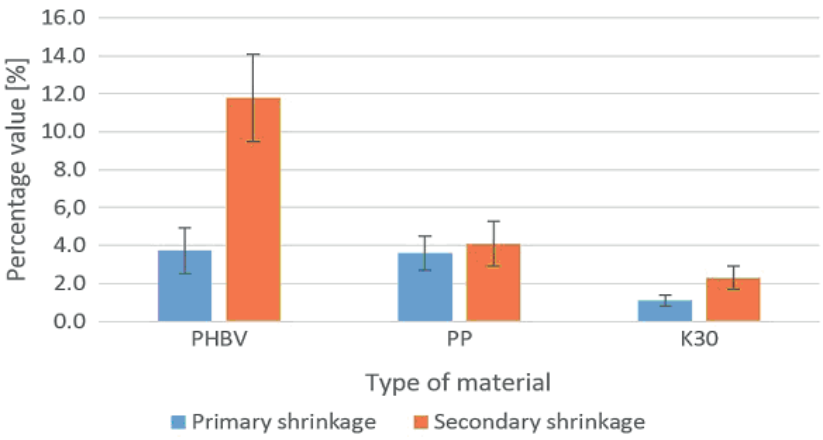

Fig. 2. Primary and secondary shrinkage values for PHBV, polypropylene and K30 biocomposite

Table 4. The control factors and the levels of variability

Control factor	Level 1	Level 2	Level 3
Cooling time [s]	20	25	30
Mold temperature [°C]	75	80	85
Melt temperature [°C]	180	185	190
Packing time [s]	20	25	30
Packing pressure [MPa]	28	30	32
Flow rate [cm^3/s]	25	35	45

number of tests was reduced. The L27 plan was used in the research. For the purposes of the method, an orthogonal table (Table 5) was built, containing 27 combinations of parameters subject to optimization. Optimization was carried out for two main criteria:

- shrinkage of specimens (primary and secondary volumetric shrinkage),
- mechanical properties (Young's modulus, tensile strength, elongation at break).

THE RESULTS ANALYSIS AND INTERPRETATION

On the basis of the prepared 27 configurations of setting parameters, injection tests were carried out. The obtained molded pieces were tested for shrinkage and mechanical properties in a uniaxial tensile test. The results are summarized in tables 6 and 7. When analyzing the obtained data on the shrinkage value, one can notice the lowest value of

Table 5. Input parameters for 27 injected sample configurations

Sample number	Coolling time [s]	Flow rate [cm^3/s]	Packing pressure [MPa]	Packing time [MPa]	Mold temperature [°C]	Melt temperature [°C]
1	20	25	28	20	75	180
2	20	25	28	20	80	185
3	20	25	28	20	85	190
4	20	35	30	25	75	180
5	20	35	30	25	80	185
6	20	35	30	25	85	190
7	20	45	32	30	75	180
8	20	45	32	30	80	185
9	20	45	32	30	85	190
10	25	25	30	30	75	185
11	25	25	30	30	80	190
12	25	25	30	30	85	180
13	25	35	32	20	75	185
14	25	35	32	20	80	190
15	25	35	32	20	85	180
16	25	45	28	25	75	185
17	25	45	28	25	80	190
18	25	45	28	25	85	180
19	30	25	32	25	75	190
20	30	25	32	25	80	180
21	30	25	32	25	85	185
22	30	35	28	30	75	190
23	30	35	28	30	80	180
24	30	35	28	30	85	185
25	30	45	30	20	75	190
26	30	45	30	20	80	180
27	30	45	30	20	85	185

Table 6. Shrinkage of received samples

Sample number	Primary shrinkage [%]				Secondary shrinkage [%]			
	Longitudinal	Transverse	Thickness dir.	Volumetric	Longitudinal	Transverse	Thickness dir.	Volumetric
1	0.69	0.78	1.83	1.06	1.92	2.79	2.93	4.18
2	0.72	0.80	1.85	1.05	1.97	2.87	2.95	4.54
3	0.73	0.82	1.83	1.04	1.95	2.85	2.98	4.48
4	0.58	0.67	1.68	1.09	1.78	2.67	2.78	3.32
5	0.61	0.64	1.60	1.08	1.71	2.61	2.73	2.98
6	0.68	0.79	1.82	1.06	1.90	2.75	2.88	3.96
7	0.50	0.56	1.45	1.10	1.56	2.43	2.57	2.26
8	0.53	0.59	1.48	1.09	1.60	2.48	2.60	2.42
9	0.70	0.78	1.73	1.05	1.86	2.68	2.75	3.53
10	0.54	0.59	1.50	1.09	1.58	2.45	2.58	2.33
11	0.62	0.73	1.82	1.08	1.94	2.82	2.93	4.30
12	0.52	0.57	1.46	1.09	1.60	2.46	2.62	2.42
13	0.62	0.69	1.52	1.06	1.62	2.55	2.63	2.57
14	0.57	0.61	1.50	1.08	1.59	2.51	2.69	2.51
15	0.52	0.58	1.48	1.10	1.61	2.47	2.67	2.50
16	0.67	0.75	1.73	1.06	1.81	2.75	2.83	3.59
17	0.74	0.82	1.85	1.04	1.98	2.88	2.97	4.63
18	0.65	0.78	1.79	1.06	1.88	2.82	2.93	4.09
19	0.63	0.72	1.67	1.07	1.72	2.62	2.83	3.13
20	0.51	0.55	1.47	1.10	1.59	2.45	2.59	2.36
21	0.59	0.68	1.55	1.07	1.64	2.49	2.67	2.59
22	0.70	0.79	1.78	1.05	1.90	2.79	2.89	4.04
23	0.66	0.77	1.82	1.06	1.91	2.79	2.88	4.06
24	0.73	0.84	1.82	1.04	1.96	2.85	2.96	4.48
25	0.67	0.79	1.60	1.04	1.76	2.60	2.73	3.10
26	0.65	0.73	1.56	1.05	1.72	2.57	2.65	2.87
27	0.66	0.76	1.59	1.05	1.78	2.61	2.71	3.15

the primary volume shrinkage for sample no. 17, 24 and 25 (decrease in value by approx. 5,5% in relation to biocomposites processed with the initial parameters). The lowest value of volumetric secondary shrinkage was obtained for test no. 7 (decrease in value by approx. 2% in relation to biocomposites processed with the initial parameters).

Comparing the results regarding the mechanical properties, it was noted that the highest value:

- Young's modulus was obtained for test no. 15 (increase in value by approx. 3% in relation to biocomposites processed with the initial parameters),
- tensile strengths were obtained for test no. 13 (increase in value by approx. 3% in relation to biocomposites processed with the initial parameters),
- elongation at break obtained for test no. 18 (increase in value by approx. 36% in relation to biocomposites processed with the initial parameters).

After entering 18 output data into Minitab, a criterion describing the type of the analyzed problem was selected. The Taguchi method uses the so-called „Signal to noise ratio" (S/N). This parameter takes into account both the mean value of the measured signal and its standard deviation.

The method of calculating S/N depends on the tested quality criterion [30, 31]:

- in the case of optimization of the shrinkage of samples, the values of primary and secondary shrinkage should be as low as possible, so the criterion: "the smaller - the better" was selected. The quality characteristics and the „Signal-to-noise ratio" (S/N) were calculated from the equation:

$$S/N = -10 \cdot \log\left(\frac{1}{n}\sum_{i-1}^{n} y_i^2\right) \quad (1)$$

where: y_i - value of primary or secondary shrinkage.

- in the case of mechanical properties, the values of: Young's modulus, tensile strength, elongation at break should be as high as possible,

Table 7. The results summary of uniaxial tensile test

Sample number	E [MPa]	σ_M [MPa]	ε_M [%]
1	7095.93	43.82	2.68
2	6962.16	43.21	2.73
3	6658.64	41.64	2.57
4	6980.12	42.94	2.71
5	7025.69	42.63	2.66
6	6781.57	42.77	2.69
7	6855.86	42.90	2.92
8	6599.35	42.35	2.13
9	6491.20	41.90	2.86
10	6543.98	43.34	2.57
11	6932.17	41.56	1.96
12	7124.86	42.71	2.78
13	6841.03	44.12	2.62
14	6738.74	43.15	2.76
15	7195.99	43.76	2.31
16	6335.14	42.05	2.99
17	6237.25	41.66	3.00
18	6145.25	40.84	3.10
19	6911.98	43.55	2.86
20	6947.06	42.91	2.81
21	7056.49	43.85	2.73
22	6786.07	41.02	2.58
23	6499.75	41.81	2.59
24	6444.48	42.41	2.77
25	6692.90	42.14	2.41
26	6511.82	40.84	2.51
27	6344.72	40.48	2.62

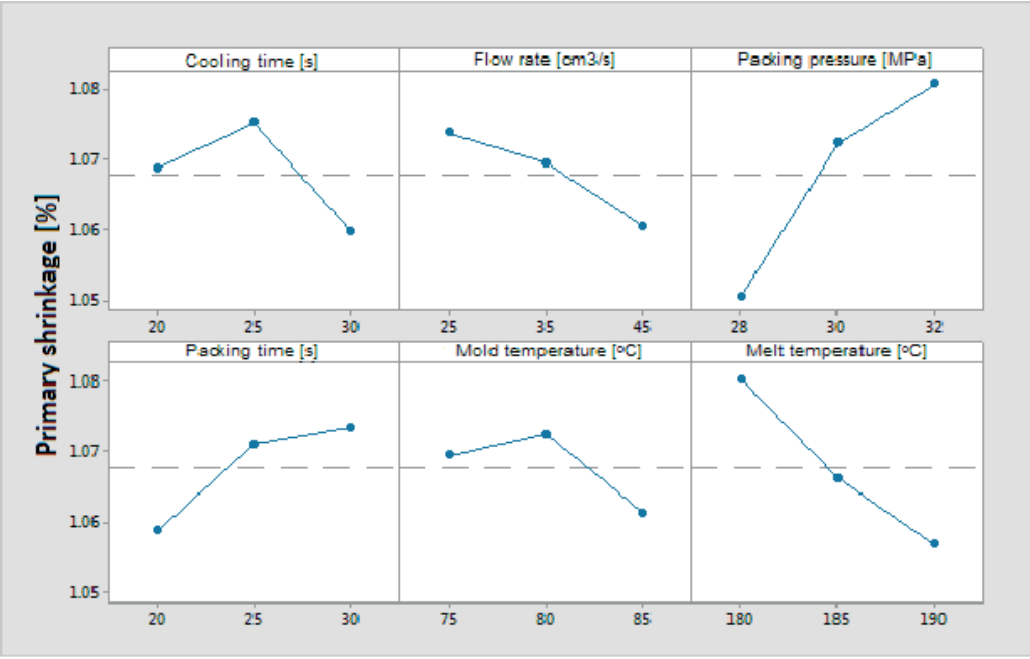

Fig. 3. Influence of controlling factors on the value of primary volumetric shrinkage

so the criterion "the greater - the better" was selected. The quality characteristics and the „Signal-to-noise ratio" (S/N) were calculated from the equation:

$$S/N = -10 \cdot \log\left(\frac{1}{n}\sum_{i-1}^{n}\frac{1}{y_i^2}\right) \quad (2)$$

where: y_i - Young's modulus or tensile strength or elongation at break.

The obtained response characteristics concerning the primary (Fig. 3) and secondary (Fig. 4) shrinkage illustrate the influence of the controlling factors on the value of shrinkage.

When analyzing the obtained results (Fig. 5-7) as response characteristics, it can be noticed that to increase the Young's modulus, the injection speed must be reduced and the packing

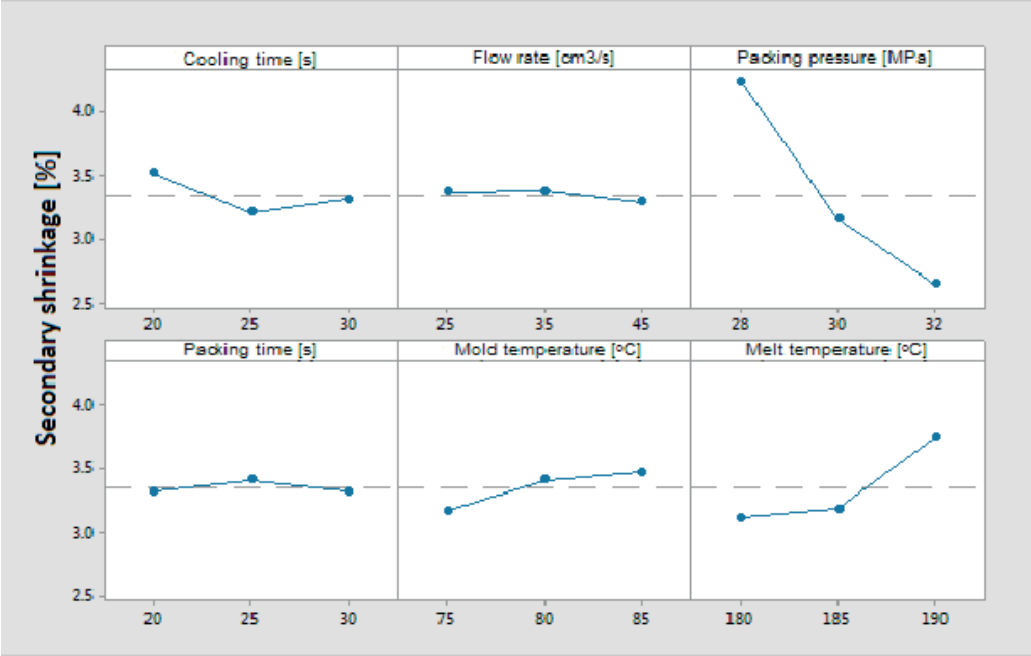

Fig. 4. Influence of controlling factors on the value of secondary volumetric shrinkage

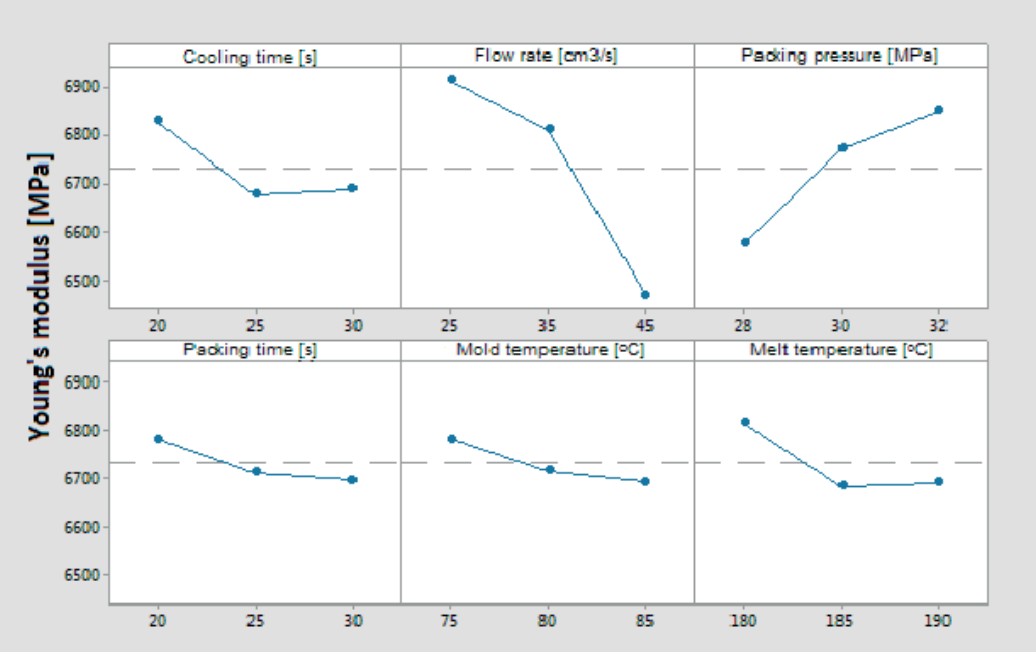

Fig. 5. Influence of controlling factors on the Young's modulus

pressure increased. Lowering the value of the other controlling factors slightly increases the value of the elastic modulus. To increase the tensile strength the greatest impact have: lowering the injection speed (flow rate) and increasing the packing pressure. Lower values of the cooling time and the packing time slightly increase the value of the tensile strength. In order to increase the elongation at break the melting temperature should be lowered.

In addition, on the basis of the above-mentioned results, it is possible to estimate the optimal setting parameters for individual input criteria - they are presented in Table 8.

Injection molding tests were carried out for the recommended setting parameters for

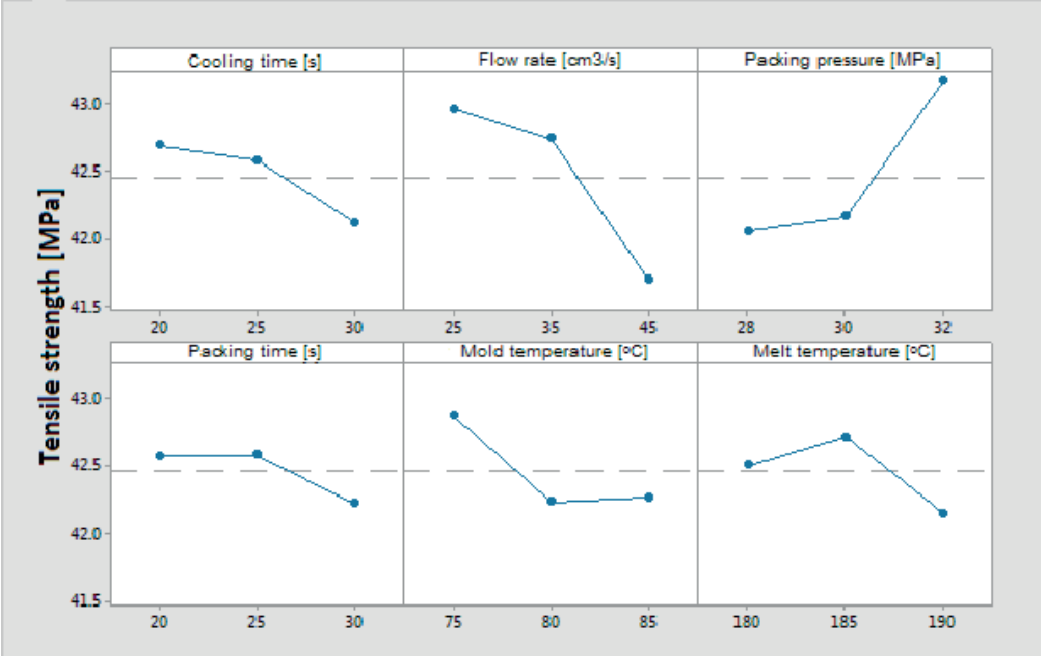

Fig. 6. Influence of controlling factors on the tensile strength

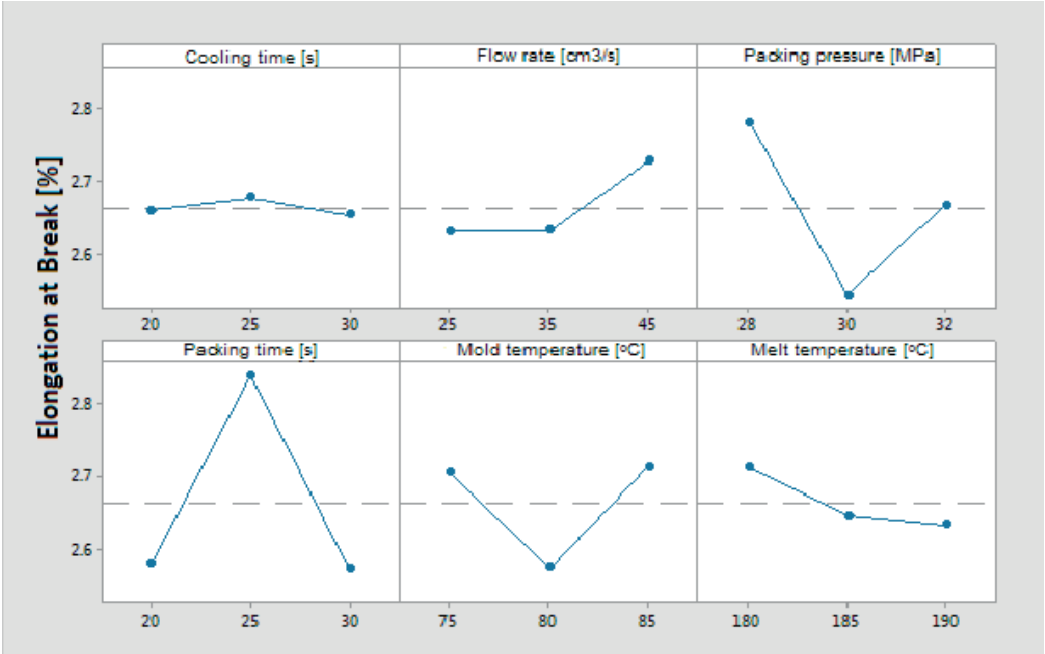

Fig. 7. Influence of controlling factors on the elongation at break

Table 8. The summary of the optimal setting parameters/control factors

Control factor	Output criterion				
	Primary shrinkage	Secondary shrinkage	Young's modulus	Tensile strength	Elongation at break
Cooling time [s]	30	25	20	20	25
Flow rate [cm^3/s]	45	45	25	25	45
Packing pressure [MPa]	28	32	32	32	28
Packing time [s]	20	30	20	20	25
Mold temperature [°C]	85	75	75	75	85
Melt temperature [°C]	190	180	180	185	190
Output properties					
	1.07%	2.28%	7218.1 MPa	45.1 MPa	3.21%

individual injection molding cycles. Slight improvement of primary and secondary shrinkage as compared to initial process parameters setting was noted. Interestingly, these results are worse than the shrinkage of specimens produced by the best process parameters configurations from the orthogonal array. This phenomenon may result from the fact that the composite is made of a 70% share of the PHBV matrix, whose shape and dimensional properties are characterized by low repeatability, which may directly affect the lack of effective forecasting of the shrinkage value of the tested amount of biocomposite.

On the other hand, when analyzing the mechanical properties, for each of the analyzed output criteria, the highest results were recorded in relation to injection moldings for the initial adjustable parameters and from the orthogonal table of the Taguchi. Compared to the primary setting parameters used, the Young's modulus value improved by over 3.5%, tensile strength by over 5% and elongation at break over 40%.

CONCLUSIONS

Analyzing the obtained results as response characteristics, it can be noticed that:

1. To increase the Young's modulus, the injection speed must be reduced and the packing pressure increased. Lowering the value of the other controlling factors slightly increases the value of the elastic modulus.
2. To increase the tensile strength the greatest impact have: lowering the injection speed (flow rate) and increasing the packing pressure. Lower values of the cooling time and the packing time slightly increase the value of the tensile strength.
3. In order to increase the elongation at break the melting temperature should be lowered.

After the injection molding process for the recommended setting parameters, a significant improvement of some mechanical properties was noted. On the other hand, for the analyzed shrinkage, only a slight improvement in the value was noted in relation to the specimens produced at the initial parameters. The use of the Taguchi method in planning and optimization of the injection molding process of the PHBV-hemp fiber products from biocomposite makes it possible to reduce the number of tests needed to perform the tests, moreover, on the basis of the signal to noise ratio, it is possible to estimate the influence of the relevant controlling factors on the change in the values of the tested parameters.

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