Basic mechanical analysis of biodegradable materials

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Abstract. The field of polymeric materials and manufacturing technologies is constantly evolving, offering the possibility to prototype 3D products in a responsible and ecological way, thus aiming to replace on a large scale the filaments of nonbiodegradable synthetic polymers (from fossil resources) with filaments of biodegradable materials, obtained from renewable resources. The paper supports the development mentioned above and follows to characterize biodegradable materials from the mechanical behavior point of view, tensile, bending, and impact tests. Also, the study reflects the influence of the technological parameters on the tensile test obtained results and also aims to optimize the obtained results. The studied materials were Extrudr Green-TEC Anthracite and Extrudr BDP Pearl which according to the obtained basic mechanical results can successfully replace conventional polymers such as Flexible, HIPS, PP and other ones.

1. Introduction
As biodegradable thermoplastic materials are increasingly entering on the filaments market used in 3D printing, the FDM method (Fused Deposition Modeling), knowing and understanding their functional characteristics is a very important aspect especially when it comes to the optimal selection of the material for specific applications of a certain field (engineering, medicine, food service, automotive etc).

Also, the study of the process parameters influence (printing speed, part orientation, layer thickness, printing temperature, infill degree, etc.) on the obtained results must be studied in order to optimize the 3D printing environment and at the same time the mechanical, thermal and structural characteristics of printed parts, [1, 2, 3].

To be considered filaments biodegradable, according to the FTC’s Green Guide (Federal Trade Commission), [4] they must decompose in nature under the action of the microorganisms, into chemical elements that are found naturally in the environment (oxygen, carbon, etc.). It should be noted that these materials usually contain additives that allow them to decompose faster than conventional plastics, however, it is not recommended to be stored in nature to decompose but in well-established locations as the speed and type of biodegradation differ and depends on the constituents used in their production, [5].
The obvious advantage of the biodegradable polymeric materials is the possibility of degradation, but this is not the only one, other strengths are: the possibility of processing/manufacturing by using the same technologies as in the case of conventional polymers, reduced energy consumption due to the low printing temperatures, rigidity, and mechanical strength, pleasant aesthetic appearance, etc.

The results from the literature regarding the characterization of printed parts from biodegradable materials by the FDM technology look promising due to the relevant number of scientific articles published in this field recently, [5 - 12]. However, not all aspects have been examined and conclusive information can be obtained by understanding and improving the printing of biodegradable materials by the FDM method.

The novelty of the present manuscript consists of the fact that characterizes mechanically two biodegradable thermoplastic materials that until now were not study from any point of view. The objectives pursued by the authors are to determine the behavior of the Extrudr GreenTee Anthracite and Extrudr BDP Pearl materials during tensile, bending and impact tests. The tests were performed in order to make some recommendations regarding a possible replacement of conventional plastics with the studied materials.

2. Materials and methods

The prototyping of the samples necessary for the mechanical determinations was performed on a series of biodegradable materials selected according to their utility and the possibility of substituting nonbiodegradable synthetic plastics. Biodegradable materials in the form of filaments used in the study of this paper were Extrudr Green-TEC Anthracite and Extrudr BDP Pearl.

Extrudr Green-TEC Anthracite and Extrudr BDP Pearl filaments are produced by Extrudr company (Lauterach, Austria). Extrudr Green-TEC Anthracite is part of the BIO Performance products range, is made from renewable raw materials - contains PLA (based on corn starch, sugar cane or tapioca roots), copolyester and additives, [13]. Is biodegradable according to DIN EN ISO 14855, being created to be used in applications that require high tensile strength (excellent flexural strength), low deformation and high product surface quality. This material has no smell and is safe in the food field. Extrudr BDP Pearl is a material 100% from renewable resources, used mainly in applications where the aesthetic part is important (BIO Design) and with mechanical and thermal characteristics comparable to those of PLA. According to the manufacturer, its chemical composition is similar to that of the Extrudr Green-TEC Anthracite material, [14].

3D printing of specific tensile test samples was performed according to ISO 527, [15, 16], using Raise3D Pro2Plus printing equipment, available in the Laboratory of Fine Mechanics and Nanotechnology, Faculty of Mechanical Engineering and Industrial Management, "Gheorghe Asachi" Technical University of Iasi.

Prototyping of biodegradable materials was performed according to a complete factorial experimental plan of $2^3$ type (8 experiments), in which the factors were the layer thickness (0.1mm, 0.2mm), the infilling speed (40mm / min, 80mm / min) and the sample orientation on the printing table (flat - and on edge - ), using the ANOVA method, [17] and the Minitab 17 program. The samples are dumbbell shaped and the dimensions according to the standard mentioned above.

The tensile test samples were printed from Extrudr Green-TEC Anthracite and Extrudr BDP Pearl materials. The diameter of the nozzle was 0.4 mm. All samples were printed with three shell layers and a 100% filling degree. The temperatures of the extrusion nozzle and of the printing bed for the two printed materials were:
- For Extrudr Green-TEC Anthracite material, the nozzle temperature was 220°C and printing bed temperature 60°C;
- For Extrudr BDP Pearl material, nozzle temperature 180°C and printing bed temperature 55°C.

Uniaxial tensile tests were performed at the "Gheorghe Asachi" Technical University of Iasi, Faculty of Materials Science and Engineering, on the Instron 3382 universal test machine, with a constant
transverse speed of 5mm/min according to ISO 527-3: 2003 (table 1), the distance between the jaws was 115mm, the data acquisition rate 10Hz and the determinations were performed at room temperature (23°C).

TableCurve3D v.4.0 application was used to optimize the technological parameters for 3D printing with filament from biodegradable materials.

In order to achieve a three-point bending test, three specimens were prepared (to demonstrate that the printing process is stable and reproducible) from each biodegradable material. A single test was performed on materials with a deformation greater than 5 mm. The used specimens were cut from those specific to tensile test and have been determined the distance between the sample placement supports, L = 60mm, the length of the tested sample 50mm, the thickness h = 4mm and its width b=10mm. A WTW 50 universal test machine was used to determine the bending strength, using a 1kN load and a 2mm/min load rate.

The impact-specific samples were printed according to the SR EN ISO 179 standard (table 1), with the following dimensions: length (L) = 80 ± 2 mm, width (l) = 10 ± 0.2 mm, thickness (h) = 4 ± 0.2 mm. The test parameters used to determine the impact resistance of the two biodegradable materials were: speed 2.9 m/s, hammer weight 1.189 kg, energy 5J.

| Material name          | Tensile-specific sample | Impact-specific samples |
|------------------------|-------------------------|-------------------------|
| Extrudr Green-TEC Anthracite | ISO 527-3: 2003        | SR EN ISO 179           |
| Extrudr BDP Pearl      |                         |                         |

The temperature at which the tests were performed was 20°C. Three biodegradable samples of each material were printed for the impact test. The printing parameters set for obtaining samples were: printing temperature and printing bed temperature according to the statements made for the printed specimens for the tensile test; infill speed 60mm/s; the deposited layer thickness 0.2mm; filling type - grid; filling degree- 100%; flat orientation of the sample on the printing table; the temperature inside the printer was 23°C.

The test results are used to select a biodegradable material for a required load, to control the quality and to anticipate how this material will react under the action of different types of forces.

3. Results and discussions

3.1. Tensile strength

For each experiment, three samples were printed to highlight the process stability by calculating the mean and the dispersion of the values for each input parameters values set from the experimental plan.
Each test was generated responses regarding the tensile strength, $\sigma$ [MPa], elongation, $\varepsilon$ [%] and modulus of elasticity, $E$ [MPa] of the analyzed biodegradable material, calculating for each one the mean value and dispersion. Using the ANOVA method, the influences of the input parameters on the tensile strength, on the elongation and on Young's modulus were determined, which allowed a hierarchy of the influence of these factors.

In table 2, the values of the mechanical characteristics obtained for the two studied materials are shown. The characteristic strength-strain curves for the two biodegradable materials Extrudr Green-TEC Anthracite (experiment 6) and Extrudr BDP Pearl (experiment 6), highlighted the relatively homogeneous behavior of the samples, figure 1(a) and figure 1(b), demonstrating the fact that the prototyping process, by the FDM method, was stable and reproducible, the mechanical properties of the materials varying within acceptable limits.

**Table 2.** Experimental results of uniaxial tensile tests of printed samples from Extrudr Green-TEC Anthracite and Extrudr BDP Pearl.

| Exp. no. | Material          | $l_t$ [mm] | $i_s$ [mm/min] | $s_o$ | $\sigma_{\text{max}}$ [MPa] | $\varepsilon$ [%] | $\varepsilon_t$ [%] | $E$ [MPa] |
|----------|------------------|------------|----------------|------|-----------------------------|------------------|-------------------|----------|
| 1        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 2        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 3        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 4        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 5        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 6        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 7        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |
| 8        | Extrudr BDP Pearl | 0.2        | 40             | on edge | 28.63±1.10                 | 5.44±0.9      | 12.71±3.35         | 1036.86±4.83 |

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not: ($l_t$) - layer thickness; $i_s$ - infill speed; $s_o$ – sample orientation.

Analyzing the obtained results from the tensile tests it can be seen that the experiments with the best results, the highest values of tensile strength, $\sigma_{\text{max}}$, were recorded by the samples oriented "on edge" on the printing bed, Extrudr Green-TEC Anthracite - experiment number 6, (30.26 ± 1.89)MPa, Extruder BDP Pearl - experiment number 6, (28.63 ± 1.10)MPa.

Following the results dispersion for the three samples of each material studied, it can be concluded that the material Extrudr BDP Pearl figure 1(b) has the smallest dispersion, followed by the material, Extrudr Green-TEC Anthracite - experiment 6, which shows a higher dispersion of the results.

According to the obtained results regarding the strain at break ($\varepsilon_{\text{max}}$) of the printed samples from the studied materials, it was found that the material that showed the most rigid behavior was the
Extrudr Green-TEC Anthracite biopolymer (4.84 ± 0.14)% having a value of the total strain at break of (5.51 ± 0.61)%. The Extrudr BDP Pearl material showed slightly lower elasticity than the Extrudr Green-TEC Anthracite material, the value of strain at break being (5.44 ± 0.9)%, and the total strain at break of (12.71 ± 3) 35)%.

The modulus of elasticity of the analyzed polymers is higher for the „on edge” oriented samples during the prototyping process. The highest mean value is presented by the material Extrudr Green-TEC Anthracite (1232.48 ± 75.30)MPa which reveals that this material is stiffer than the other material, Extrudr BDP Pearl (1062.65 ± 65.13)MPa which deform more because it has a smaller modulus of elasticity.

The behavior of the materials, Extrudr GreenTEC Anthracite, figure 1(a), and Extrudr BDP Pearl, figure 1(b), is approximately similar, their curves being characteristic for hard and resistant materials.

The mechanical properties obtained for the studied materials are comparable to those of synthetic materials (Flexible - tensile strength 26-43MPa, HIPS (High Impact Polystyrene) - tensile strength 32MPa, PP (Polypropylene) - tensile strength 32MPa, Metal Filled - tensile strength 20-30MPa, etc.), [18] so that they can be used in various industrial fields and not only.

3.2. The influence of technological parameters on tensile behavior
The influence of the technological parameters on the mechanical behavior of the printed samples was made only for the Extrudr Green-TEC Anthracite material because for it the best results in tensile and impact tests were obtained.

Table 3 presents the influence variance analysis results of the considered input parameters on the tensile strength, modulus of elasticity and strain of the Extrudr Green-TEC Anthracite material. The biggest influence on the tensile strength, but statistically insignificant (p = 0.095> 0.05), is the sample orientation (F = 4.76). The infilling speed and the deposited layer thickness also do not have a statistically significant influence since p> 0.05. The same happens both in the case of the modulus of elasticity and in the case of strain, according to the obtained results no parameter has statistically significant influence (p> 0.05). For this reason, the influence of the parameters on the strain was no longer graphically represented.

Figure 1. Strain-strength curve of materials: (a) Extrudr GreenTec Anthracite, experiment no. 6; (b) Extruder BDP Pearl, experiment no. 6.
Table 3. Results of the variance analysis on the tensile strength by the ANOVA method for the mechanical responses of the Extrudr Green-TEC Anthracite sample.

| Parameter               | Tensile strength | Modulus of elasticity | Strain |
|-------------------------|------------------|------------------------|--------|
|                         | Fisher-value     | Probability of Fisher value, p-value | Fisher-value | Probability of Fisher value, p-value | Fisher-value | Probability of Fisher value, p-value |
| Sample orientation      | 4.76             | 0.095                  | 44.27  | 0.003                  | 0.06        | 0.821                  |
| Infill speed, [mm/min]  | 0.64             | 0.468                  | 0.36   | 0.582                  | 0.08        | 0.791                  |
| Layer thickness, [mm]   | 0.16             | 0.708                  | 1.55   | 0.281                  | 1.85        | 0.245                  |

Figure 2 shows the main effects of the factors on the obtained mechanical responses for the Extrudr Green-TEC Anthracite material. For the tensile strength (mean value of the model 23.18MPa) the samples deposited „on edge“ (variation +1 level) have a higher influence, with a higher infill speed (80mm / min) and at a higher deposition thickness (0.2mm).

The higher modulus of elasticity (model mean value 988.91MPa) was evidence by the „on edge“ printed samples. A larger print layer thickness (0.2mm) and a higher infill speed (80mm / min) ensure the samples a higher modulus of elasticity.

3.3. Optimization of technological parameters in order to maximize mechanical responses

To optimize the technological parameters for 3D printing with filament from biodegradable materials, the application TableCurve3D v.4.0 was used and the same material was considered for the same reasons presented for the influence of technological parameters.

The optimization criteria are the mechanical responses maximization received from the tensile tests of the Extrudr Green-TEC Anthracite samples for tensile strength, modulus of elasticity and strain.

Quantitative technological parameters were taken into account to optimize the tensile strength: infill speed, s [mm/min], and the printing layer thickness, t[mm]. The printing direction was considered „on edge“, for which, according to the analysis of the factors influence, the best results emerged. Table 4, the (a) graph shows the plan obtained by regression. The correlation coefficient is R² = 0.383 - being very small.
The equation that approximates the influence of the deposition layer thickness, \( t \), and the infill speed, \( s \), on the tensile strength, is presented in table 4, whose maximum in the experiments field is for \( t = 0.2 \, \text{mm} \) and \( s = 80 \, \text{mm/min} \). For these parameters and „on edge” printing was obtained \( \sigma = 29.865\,\text{MPa} \). The deviation from the experimental data in this case is 12.32%.

For the modulus of elasticity quantitative factor, the considered printing direction was „on edge”, because, according to the analysis of the factors influence, the best results emerged.

**Table 4.** Results regarding the optimization of the technological parameters in the case of the printed samples from Extrudr Green-TEC Anthracite.

| Parameter | The regression equation | \( R^2 \) | Max. \( \sigma \) [MPa] | Error [%] | \( t \) [mm] | \( s \) [mm/min] |
|-----------|-------------------------|---------|------------------------|----------|-------------|-------------|
| \( \sigma \) | \( \sigma = 15.545 + 42.8 \cdot t + 0.072 \cdot s, [\text{MPa}] \) | 0.383 | 29.865 | 12.32 | 0.2 | 80 |
| \( E \) | \( E = 1378.91 - 395.85 \cdot t - 2.559 \cdot s, [\text{MPa}] \) | 0.71 | 1235.5 | 2.8 | 0.1 | 40 |
| \( \varepsilon \) | \( \varepsilon = 0.855 + 16.8 \cdot t + 0.0305 \cdot s, [%] \) | 0.55 | 6.65% | 12.32 | 0.2 | 80 |

Note: \( \sigma \) - tensile strength [MPa]; \( E \) - modulus of elasticity; \( \varepsilon \) - strain; \( R^2 \) - correlation coefficient;

In table 4, graph (b), is graphically represented the regression equation of the plane that provides information related to the infill speed and layer thickness influence on the modulus of elasticity in the case of „on edge” 3D printed samples made of Extrudr Green Anthracite material.
Also, the regression equation for the modulus of elasticity ($R^2 = 0.71$) is presented in table 4, whose maximum in the experiments field is for $t = 0.1\text{mm}$ and $s = 40\text{mm/min}$. For these parameters and “on edge” printing, $E = 1235.5\text{MPa}$ is obtained, with an error of 2.8% compared to the experimental data. The best results for elasticity came out when the printing direction was considered “on edge”. The equation of the plane is for $R^2 = 0.552$ (weak correlation) being presented in table 4, graph (c), whose maximum in the experiments field is for $t = 0.2\text{mm}$ and $s = 80\text{mm/min}$. For these parameters and “on edge” printing is obtained $\varepsilon = 6.655\%$. The error compared to the experimental data is 12.32%.

3.4. Bending strength

Before starting the bending test, certain conditions have been established according to the literature. Thus, it was considered that for materials with the maximum arrow in the centre of the sample (linear displacement in the direction of the application force) greater than 5 mm, the results obtained cannot be taken into account for this type of test.

Biodegradable materials, Extrudr Green-TEC Anthracite, figure 3 (a), Extrudr BDP Pearl, figure 3 (b), according to the characteristic bending test curves, are not suitable for this type of test as it does not yield easily under the action of a progressive load, because they are elastoplastic materials, with a maximum arrow greater than 5mm.

![Figure 3. The bending strength curve depending on: (a) Extrudr Greentec, exp. no. 8; (b) BDP Pearl Extruder, exp. no. 8.](image)

3.5. Impact strength

Determination of the impact strength, by the Charpy method, of the samples from Extrudr Green-TEC Anthracite and Extrudr BDP Pearl materials was realized in accordance with the SR EN ISO 179 standard, with the aim of observing their impact behavior. The obtained values during the impact test for Extrudr GreenTec and Extrudr BDP Pearl materials are shown in table 5.

The best value of impact strength was recorded by the Extrudr Green-TEC Anthracite material $(8.87 \pm 1.02)\text{kJ/m}^2$, followed by the Extrudr BDP Pearl material $(7.98 \pm 0.26)\text{kJ/m}^2$. The obtained values are much lower than in the case of other printed samples made of biodegradable materials, for instance, compared to PLA (polylactic acid), which according to the literature is the most widely used biodegradable material in FDM printing and has an impact resistance of about $38\text{kJ/m}^2$, [19]. The obtained results for the studied materials are up to five times lower than the material selected as reference (PLA). The major differences between the obtained results from the literature for PLA and the Extrudr Green-TEC Anthracite and Extrudr BDP Pearl biodegradable materials are cause by the different chemical compounds found in their structure. According to previous studies on the impact strength of biodegradable materials, the presence of renewable raw materials (lignin) and/or natural plant fibres (a substance that gives hardness) decreases the impact strength of polymers/composites, [20, 21]. The viscoelastic hardness is explained by the fact that the lignin molecule is a complex
molecule which in its turn is made up of three other complex molecules and which, depending on an external deforming cause, tries to shorten the chemical bonds between them so that the lignin molecule to occupy the same volume.

Table 5. Mean values of the Charpy strength for printed samples from Extrudr GreenTec and Extrudr BDP Pearl.

| Material                  | Sample | Sample dimensions [mm] | Impact strength (kJ/ m²) | Mean values of impact strength (kJ/ m²) |
|---------------------------|--------|------------------------|-------------------------|----------------------------------------|
|                           |        | length | width | | | |
| Extrudr Green-TEC Anthracite | 1      | 36.4   | 100.0 | 8.15 | | |
|                           | 2      | 35.9   | 99.9  | 14.99 | | |
|                           | 3      | 37.0   | 99.6  | 9.59  | | |
| Extrudr BDP Pearl         | 1      | 36.3   | 100.3 | 13.60 | | |
|                           | 2      | 36.1   | 100.4 | 7.79  | | |
|                           | 3      | 36.3   | 100.1 | 8.16  | | |

*mean value achieved between the impact strength of two samples, sample three being excluded from the study due to the very large difference in the value obtained for the impact strength (difference probably caused by possible temperature variations during printing)

Also, from the graph analysis presented in figure 4, the variations of the mean values of the impact strength but also can be observed the standard deviations calculated for each material. The mean value of the impact strength deviations is ± 0.73 kJ/m².

Figure 4. Impact strength diagram for the printed samples made of Extrudr Green-TEC Anthracite and Extrudr BDP Pearl.

Following the analysis of the obtained results, the printed samples from Extrudr Green-TEC Anthracite and Extrudr BDP Pearl materials can successfully substitut other plastics that have in their composition lignin and natural fibers but also polymers reinforced with glass microfibers or metal powders, [18, 22].

4. Conclusions

Samples obtained using FDM three-dimensional printing technology are significantly influenced by the process parameters that can be controlled directly or indirectly before the beginning of model printing. The selection of the optimal printing parameters is quite difficult if the aim is to obtain a sample with the highest possible mechanical characteristics, [7, 23].

The following conclusions can be drawn from the analysis of tensile tests:
- tensile strength is the mechanical property often used to evaluate the usefulness of the product manufactured by prototyping, and not only. Thus, the best values of tensile strength were obtained for
“on edge” oriented samples. The values of the tensile strength in the case of the “flat” orientation of the sample, varied/decreased compared to the other type of sample orientation by up to 40%;
- “flat” oriented samples have a more fragile behavior, offering lower strain results;
- the best mechanical behavior was highlighted by the biodegradable material Extrudr Green-TEC Anthracite, the material Extrudr BDP Pearl offering slightly lower results;
- also, at the moment of the mechanical determinations, the material interruptions must be taken into account, due to the layer’s adhesion lack, which has a significant effect on the general resistance of the sample.

The mechanical properties obtained for the studied materials are comparable to those of synthetic materials such as Flexible - 26-43MPa tensile strength, HIPS - 32MPa tensile strength, PP - 32MPa tensile strength, Metal Filled - 20-30MPa tensile strength, but also with those of other plastics that have in their composition lignin and natural fibers, glass microfibers or metal powders (valid for impact resistance), [82, 94], so they can be used in various industrial fields and beyond.

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