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Mechanical properties of recycled PLA and PETG printed by FDM/FFM method

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ABSTRACT

Purpose: The aim of this paper is to compare the mechanical properties of selected recycled thermoplastics against their equivalents made from new raw materials manufactured using the FDM/FFF additive method.

Design/methodology/approach: Two materials were tested: recycled polylactide (R-PLA) and recycled poly(ethylene terephthalate) with the addition of glycol (R-PETG). Reference materials are their equivalents made from new raw materials. Both types of materials are widely available on the market. In order to compare their mechanical properties and to check whether recycled materials do not differ in quality from their equivalents made from new raw materials, tensile strength tests were performed. In addition, the Vickers microhardness was measured, and the structure of printed samples using optical microscopy was observed.

Findings: The paper presents the results of the static tensile strength test of samples made by the FDM/FFF technology from the tested materials in accordance with the ISO-00527-2-2012 standard. The samples were manufactured at the average temperature recommended by the producer ±10°C. The results of tensile strength tests indicate that the samples printed at the average temperature show the best tensile strength for both methods of filament deposition.

Research limitations/implications: The recycled materials are not significantly different from the reference materials in terms of tensile strength, microhardness and structure. It is reasonable to test other polymeric materials further and check materials from several consecutive recycling cycles.

Practical implications: Closing the cycle of plastic used in 3D printing. The ability to quickly transform waste products, e.g. PET bottles, into filaments and reuse them to produce full-value products.

Originality/value: The paper presents the results of strength and microhardness tests as well as microscopic investigations of two recycled thermoplastics commonly used in the industry manufactured using the FDM/FFF technology against the background of reference materials made from new raw materials.

Keywords: 3D printing, FDM/FFF, Recycling, PLA, PETG

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PROPERTIES

1. Introduction

Polymers are omnipresent in our lives and have found circa-countless applications in areas such as medicine, electronics, automotive, aerospace, electronics/household appliances, food packaging, etc. The increase in the world's population, together with a growing economy, has led to an increase in the consumption of polymers. According to the European Environment Agency (EEA) [1], in 2020, global plastic production was ca. 450 million tonnes. EEA also states that 85% of marine litter is plastic. Plastic production in Europe in 2021 was 57.2 million tonnes. Plastics Europe [2] reports that recycled plastics, together with biobased plastics, account for only 12.4% of European production. It is estimated that the annual production of plastics in the EU leads to the generation of ca. 13.4 million tonnes of CO_2. As the global climate deteriorates situation, organisations and communities adopt strategies to reduce the generation of plastic waste, mainly by increasing the reuse of recycled plastics and replacing plastic materials with biodegradable ones. A few things could be improved in processing plastics for further reuse. Due to their wide variety and high similarity to each other, plastics are difficult to recycle. Moreover, their degradation causes the quality loss of recycled material. Those problems and related ones by Kampman Eriksen et al. [3] were described.

Nowadays, additive technologies in relation to metal and ceramic materials [4,5], as well as polymers on a large scale, are used. The most widespread methods of polymer additive manufacturing, both in industry and for private use, are extrusion technologies. The basic principle of these is layer deposition, usually in thread form, on the working surface of the machine. The most popular extrusion technologies for manufacturing thermoplastics are Fused Deposition Modelling (FDM) and its derivative, Fused Filament Fabrication (FFF). Today, the terms are used interchangeably because the basis of the technology is the same, but anyway, there are minor differences in machine construction [6]. The FDM method by Stratasys in 1989 was patented. It involves cutting a 3D model into 2D slices and depositing the material by a printhead moving in a Cartesian system (XYZ) in a closed environment at a controlled temperature. However, after the Stratasys patent expired in 2009, the FFF method by the RepRap community was developed and popularised. The group of engineers and hobbyists replicated the FDM technology, omitting expensive components such as the heated chamber and platform. The result of those changes and using cheaper and more widely available components, such as aluminium profiles and stepper motors from inkjet printers, significantly reduced the cost of making the machine and manufacturing objects in this technology. Today, most machines of this type are labelled FDM/FFF; the FDM term is used only for the most professional use. The paper contains research results of materials manufactured using the FDM/FFF printing machine.

3D printing using FDM/FFF technology is applied extensively both in industry and by individual users. It is estimated that the market value of the hardware only in 2020 was equal to €11 billion. Available data shows that in 2022 the market for 3D printers has grown to €22 billion. It is estimated that in 2024 it will be €35 billion. Emergencies, such as the COVID-19 pandemic, have proven the usefulness of additive technologies to fill critical equipment shortages rapidly. Examples include the initiative to manufacture protective visors for medics during the pandemic [7]. FDM/FFF technology is most commonly used in healthcare (44 %), energy industry (22 %) and transport (11 %) [8,9], with 67 % of the overall use cases being prototyped [8]. Prashar et al. [10] point to the advantages of using 3D printing in Industry 4.0. It should be noted that the owners of FDM/FFF machines are not only companies that most often have developed a waste disposal and recycling system but also private owners, for whom recycling materials is a more complicated and often unprofitable process.

The value of the filament market in 2021 was estimated at €2.4 billion [11]. It is also forecast that the market for FDM/FFF printing materials only will exceed the €20 billion threshold in 2030. The materials most commonly used in FDM/FFF technology are polylactide (PLA) and acrylonitrile-butadiene-styrene (ABS) terpolymer. With recent shortages of PLA on the market, customers are increasingly opting to print with poly(ethylene terephthalate) (PET). PLA is a material of natural origin and biodegradable which does not endanger the environment. However, PET and ABS are plastics that need up to 1,000 years to decompose completely. Like most polymer products, parts produced using FDM/FFF methods can be successfully recycled, thus protecting the environment. Unfortunately, users, mainly individuals, lack awareness that a product made from recycled materials can be as good as one made from new raw materials. The paper aims to provide a scientific basis for such claims by comparing the mechanical properties of recycled polymer materials to reference materials made from new raw materials.

2. Materials and research methodology

Popular FDM filaments – new and recycled (R-) polylactide (PLA) and poly(ethylene terephthalate) with glycol (PETG) - for the research were selected. All tested materials were sourced from the same supplier guaranteeing the same origin of new raw materials and the purity of recycled materials from internal print farm circulation. Those raw materials were used in an additive manufacturing process using a modified 3D printer Ender 3 Pro by Creality, equipped with a direct-drive extruder with a 0.4 mm diameter nozzle and open printing parameters. The minimum thickness of the applied layer in the Z-axis is 0.08 mm. A standard layer thickness of 0.2 mm for the research was selected. The average printing temperature for each material on the producer's recommendations was based. It was determined experimentally, using a temperature tower, that at 10°C above and 10°C below the recommended temperature value, unacceptable printing artefacts occur or printing is no longer possible. Therefore, the experiments were performed at a temperature equal to ± 10°C, the average recommended one. Table 1 shows the temperature of the printhead used. The printer working platform was heated at 60°C for PLA and R-PLA and 85°C for PETG and R-PETG.

Table 1.
Printing temperature of tested materials

Material	Minimum temperature, °C	Average temperature*), °C	Maximum temperature, °C
PLA	210	220	230
R-PLA	210	220	230
PETG	225	235	245
R-PETG	225	235	245

*) Values recommended by the producer

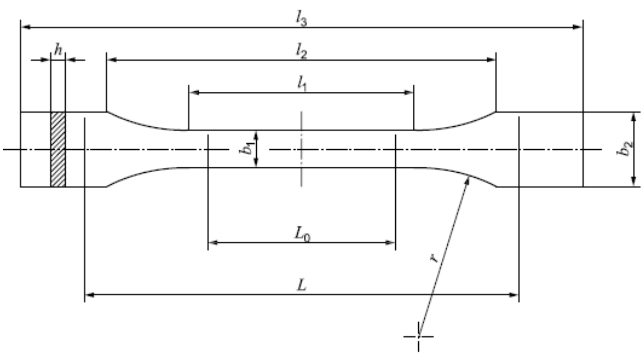

Fig. 1. B-type specimen for determination of tensile properties according to ISO-00527-2-2012 standard

The tensile strength of polymers using a testing machine Zwick Z050 was researched. Shaped specimens dedicated to these tests were manufactured. Using DSS Solidworks 2022 CAD software, a specimen model was prepared for tensile testing according to ISO-00527-2-2012 standard, as shown in Figure 1. The dimensions of the test sample correspond to the B-type specimen specified in the norm. Dimensions marked by symbols in Figure 1 are shown in Table 2.

Table 2.
B-type specimen dimensions according to ISO-00527-2-2012 standard

Symbol	Value, mm
l_3	150
l_2	108
l_1	60
r	60
b_2	20
b_1	10
h	4
L_0	50
L	115

During the tests, samples manufactured by two different methods of filament deposition were used. The methods were pre-selected in order to minimise their impact on the material strength properties. The filament vertically (Fig. 2) and horizontally (Fig. 3) was deposited. The machine code in version 2.3.57.9 of SuperSlicer was created, for each type of tested material and the method of filament deposition, respectively. The process parameters of sample manufacturing are shown in Table 3.

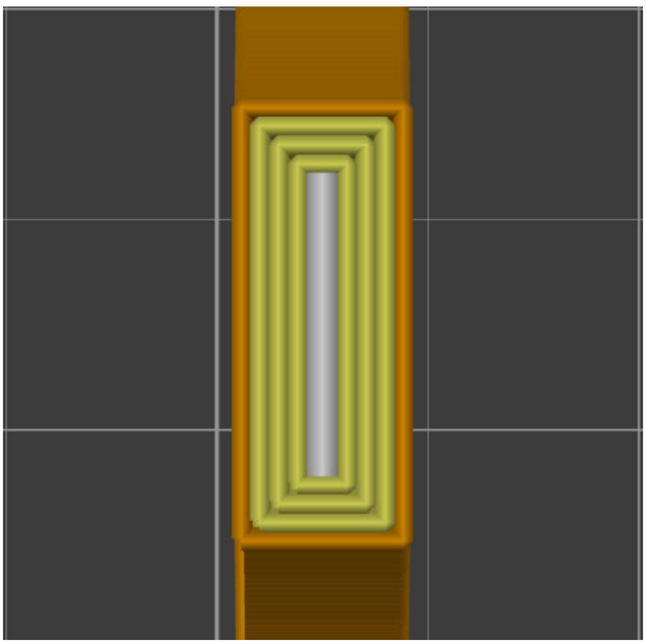

Fig. 2. Vertical filament deposition

Fig. 3. Horizontal filament deposition

In order to test microhardness and observe the material structure depending on manufacturing process temperature, additional cubes measuring 20x20x10 mm were prepared. The same process parameters as in the case of horizontal filament deposition were used for the strength tests. Microhardness by the Vickers method using microhardness testing machine Zwick ZHV10 with standard 136° diamond indenter and a load of 0.2 kg was researched. In order to prepare the specimens for microscopic investigations, the specimens using a machine, Presi Mecatech 250, were sanded. The structure of the materials using a Zeiss Observer AM1 inverted optical microscope was observed.

Table 3.
The process parameters of samples manufacturing

Parameter	Filament deposition method	
	vertical	horizontal
Layer height, mm	0.2	0.2
Print speed, mm/s	40	40
Infill pattern	concentric	linear
Sample infill, %	100	100
Minimum number of outer perimeters	4	3

3. Tensile strength results

Five shapes, made from each tested material by vertical and horizontal filament deposition, were subjected to a static tensile test at 50 mm/min elongation speed to failure. The results of the static tensile tests for materials with vertical filament deposition in Table 4, while for materials with horizontal filament deposition in Table 5, are shown. The average tensile strengths of the tested materials obtained by vertical and horizontal filament deposition at different temperatures are presented as histograms in Figures 4 and 5.

Table 4.
Tensile strength test results for materials with vertical filament deposition

Material	Process temperature, °C	Tensile strength, Rm, MPa						
		Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Average	Median
R-PLA	210	26.8	29.2	26.5	29.5	28.9	28.2	28.9
PLA	210	30.1	33.6	30.2	30.9	30.7	31.1	30.7
R-PLA	220	37.4	37.9	38.7	34.4	39.3	37.6	37.9
PLA	220	37.4	36.5	37.7	36.4	37.1	37.0	37.1
R-PLA	230	32.4	31.1	27.7	30.2	29.4	30.2	30.2
PLA	230	28.5	28.2	28.4	29.2	28.8	28.6	28.5
R-PETG	225	15.6	14.8	15.6	14.1	16.4	15.3	15.6
PETG	225	15.0	16.1	17.9	15.9	15.1	16.0	15.9
R-PETG	235	19.4	19.3	19.0	19.6	18.5	19.2	19.3
PETG	235	21.8	20.3	21.5	18.9	18.9	20.3	20.3
R-PETG	245	20.0	21.2	20.6	19.3	14.8	19.2	20.0
PETG	245	18.3	19.0	18.4	17.8	17.5	18.2	18.3

Table 5.
Tensile strength test results for materials with horizontal filament deposition

Material	Process temperature, °C	Tensile strength, Rm, MPa						
		Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Average	Median
R-PLA	210	66.2	67.7	68.3	67.1	64.0	66.6	67.1
PLA	210	67.0	68.4	66.3	67.1	70.3	67.8	67.1
R-PLA	220	66.6	66.8	66.3	66.8	66.3	66.6	66.6
PLA	220	66.8	66.3	67.1	66.3	67.1	66.7	66.8
R-PLA	230	66.6	68.2	67.7	68.3	65.5	67.2	67.7
PLA	230	66.8	66.2	67.0	67.4	66.4	66.8	66.8
R-PETG	225	57.0	57.0	56.6	56.6	57.4	56.9	57.0
PETG	225	57.5	56.2	57.3	56.1	57.3	56.9	57.3
R-PETG	235	59.0	59.1	57.2	56.8	54.0	57.2	57.2
PETG	235	57.5	59.1	58.2	58.5	58.5	58.4	58.5
R-PETG	245	57.2	55.6	56.4	55.3	56.8	56.3	56.4
PETG	245	56.3	58.0	58.2	55.9	57.0	57.1	57.0

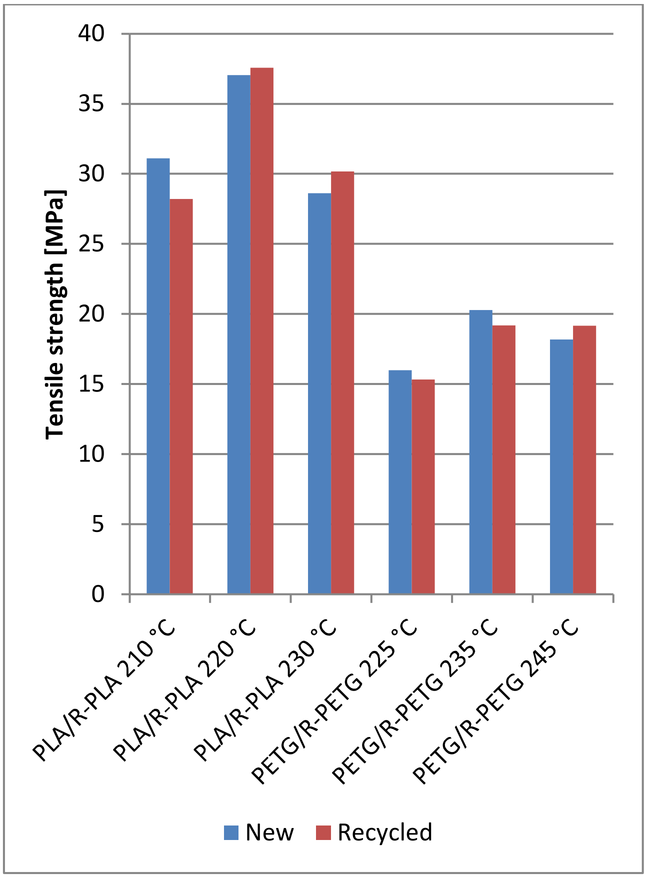

Fig. 4. Average tensile strength of materials with vertical filament deposition

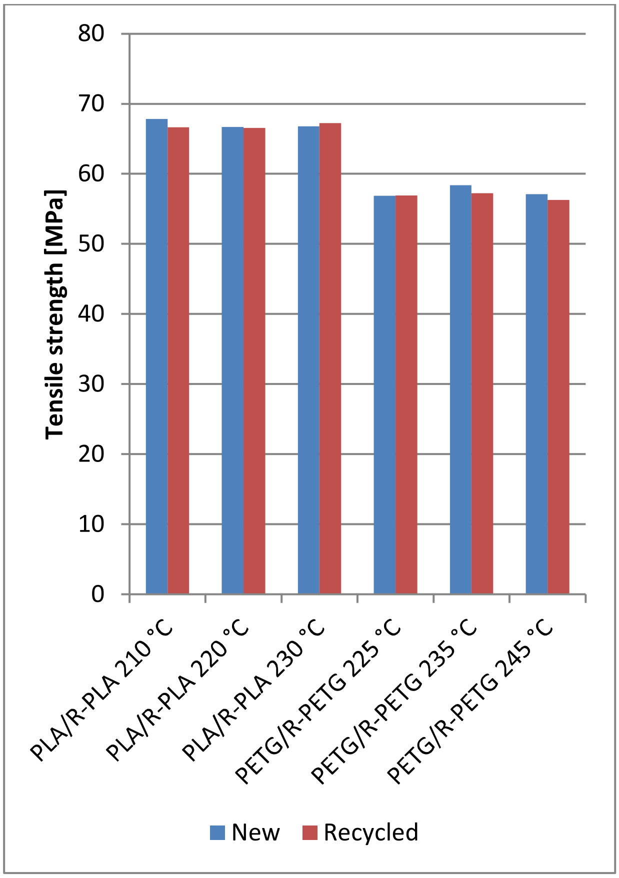

Fig. 5. Average tensile strength of materials with horizontal filament deposition

The test results regarding materials with vertical filament deposition show no significant tensile strength differences between materials formed from recycled and new row materials. In the material group, R-PLA manufactured at 220°C has the highest tensile strength, averaging 37.6 MPa, and R-PETG manufactured at 225°C has the lowest one, averaging 15.3 MPa.

In the case of materials with horizontal filament deposition, process temperature has little impact on tensile strength. There are no significant differences between the tensile strengths of materials produced from recycled raw materials and reference samples from new ones. PLA and R-PLA have higher tensile strengths than PETG and R-PETG. Among these materials, PLA manufactured at 210°C has the highest tensile strength, averaging 67.8 MPa. Within the material group, the lowest average value of 56.3 MPa for the R-PETG sample was obtained at 245°C.

The obtained tensile strength results of PLA and R-PLA for materials with vertical filament deposition are slightly worse than the literature data. Still, for horizontal filament deposition, they overlap or are better than the results already reported by other authors. Marsavina and co-authors [12] tensile strengths for both types of filament deposition, approx. 50 MPa have been obtained. However, Atakok et al. [13] and Krishna Upadhyay et al. [14] have recorded results for materials with horizontal filament deposition equal to, respectively: ca. 46 MPa and ca. 55 MPa. Bhandari et al. [15] and Khosravani et al. [16] have noted tensile strengths of 36.7 MPa and 53.5 MPa for PETG filament deposited horizontally. Szust and Adamski [17] obtained PETG tensile strength for vertical filament deposition of 15±2 MPa and horizontal one of 46±1 MPa.

4. Vickers microhardness results

Due to the much better results in terms of tensile strength, samples with horizontal filament deposition to the Vickers microhardness test were subjected. Microhardness at five random locations on the sample surface was measured. Table 6 presents the results obtained by measuring microhardness using the Vickers method at a load of 0.2 kg. Figure 6 graphically presents the average microhardness of the tested materials at different manufactured temperatures.

The results show that the hardest material is PLA printed at the temperature of 210°C with an average measurement result of 22 HV. Both PETG and R-PETG, regardless of process temperature, show approximately two times lower microhardness than PLA and R-PLA. Materials printed at 10°C lower than the average temperature recommended by the producer show minimally higher microhardness. Krishna Upadhyay et al. [14] obtained PLA microhardness of 30 HV using the Vickers method. In their research results, Rajesh et al. [18] show microhardness of 15.8 HV for PLA and 10 HV for PETG. Loskot et al. [19] have noted PETG microhardness of approx. 16 HV at identical printing speeds.

Table 6.
Vickers microhardness of materials with horizontal filament deposition

Material	Process temperature, °C	Vickers microhardness, HV						
		Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Average	Median
R-PLA	210	19	20	22	21	22	21	21
PLA	210	22	22	23	21	23	22	22
R-PLA	220	20	21	19	18	21	20	20
PLA	220	17	18	18	17	19	18	18
R-PLA	230	20	19	19	18	20	19	19
PLA	230	21	19	20	20	19	20	20
R-PETG	225	12	12	13	13	12	12	12
PETG	225	12	13	13	14	12	13	13
R-PETG	235	11	12	11	10	11	11	11
PETG	235	10	11	11	11	10	11	11
R-PETG	245	11	11	10	11	10	11	11
PETG	245	11	12	11	12	10	11	11

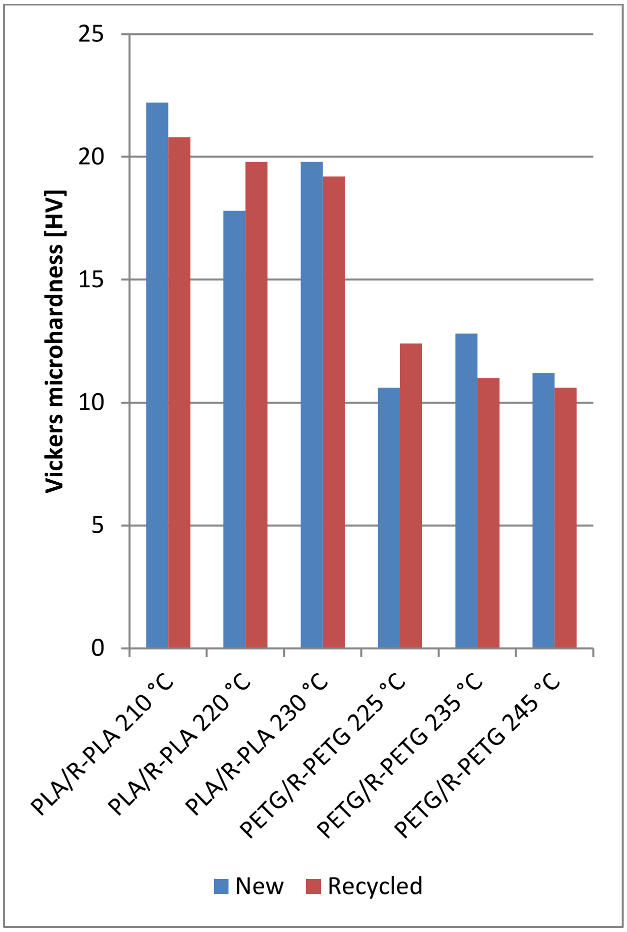

Fig. 6 Average Vickers microhardness

The results of the microhardness tests are compatible with the results of the tensile tests. PETG and R-PETG are materials with lower microhardness but higher ductility than PLA and R-PLA [18]. The hardest materials tested are non-recycled PLA printed at 210°C with an average microhardness of 22 HV. PLA, both made from new and recycled raw materials, has almost twice higher microhardness than of PETG and R-PETG. No significant differences between the microhardness of samples manufactured from new and recycled raw materials were observed, although each time, samples formed from recycled raw materials had insignificantly lower microhardness.

5. Microscopic investigations results

The chapter presents selected representative microscopic images (Figs. 7-12) of surfaces and fractures of the sample prepared from R-PLA and R-PETG.

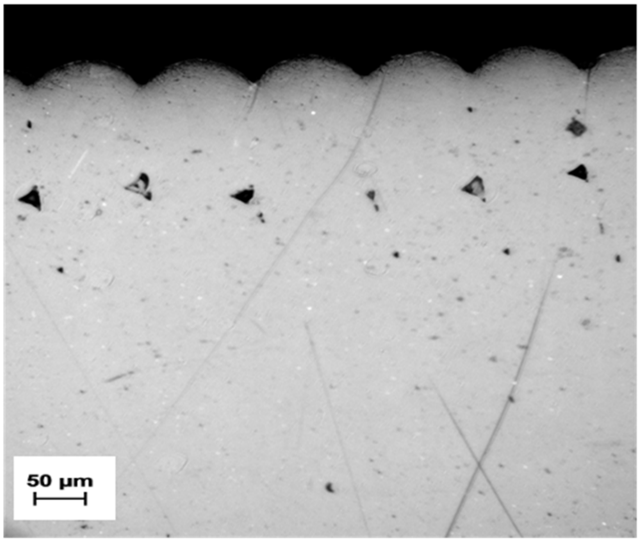

Fig. 7. The surface of R-PLA manufactured at the temperature of 230°C

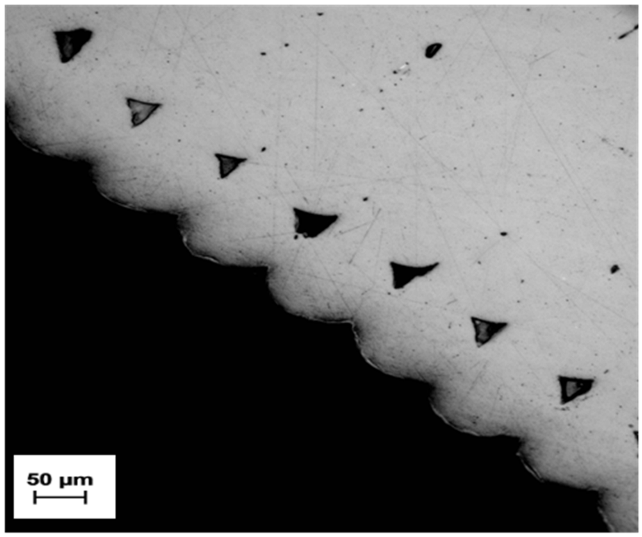

Fig. 8. The surface of R-PETG manufactured at the temperature of 235°C

The microscopic images of the material surface (Figs. 7,8) show the characteristic scale structure corresponding to the successive layers applied. Triangle-shaped holes typical for materials produced using extrusion techniques are also visible. In the case of R-PLA, the holes are smaller compared to R-PETG, suggesting better adhesion between successive material threads deposited. The sample fractures show the material structure typical for, respectively, vertical (Figs. 9,10) and horizontal (Figs. 11,12) deposition of the filament. Figures 9 and 10 show the printhead transitions.

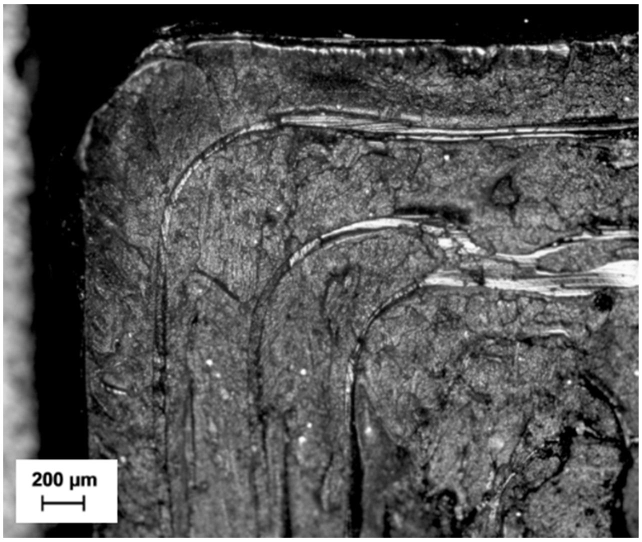

Fig. 9. Fracture of R-PLA manufactured by vertical filament deposition at the temperature of 220°C

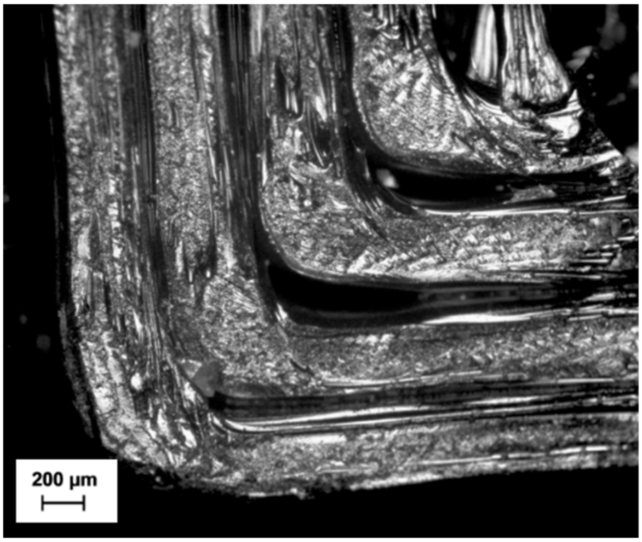

Fig.10. Fracture of R-PETG manufactured by vertical filament deposition at the temperature of 235°C

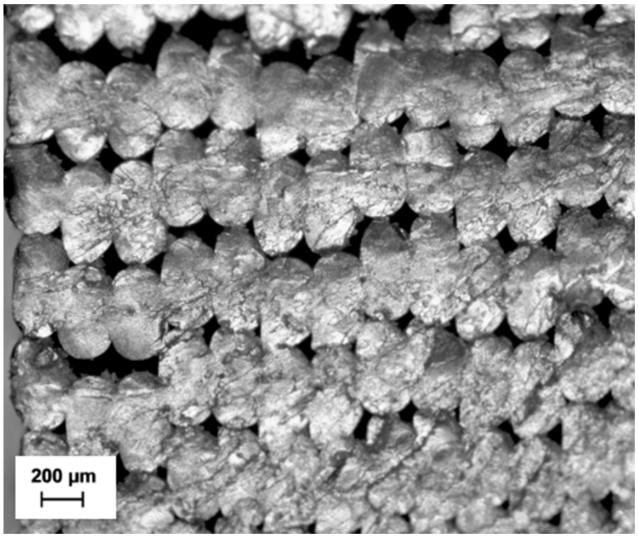

Fig.11. Fracture of R-PLA manufactured by horizontal filament deposition at the temperature of 220°C

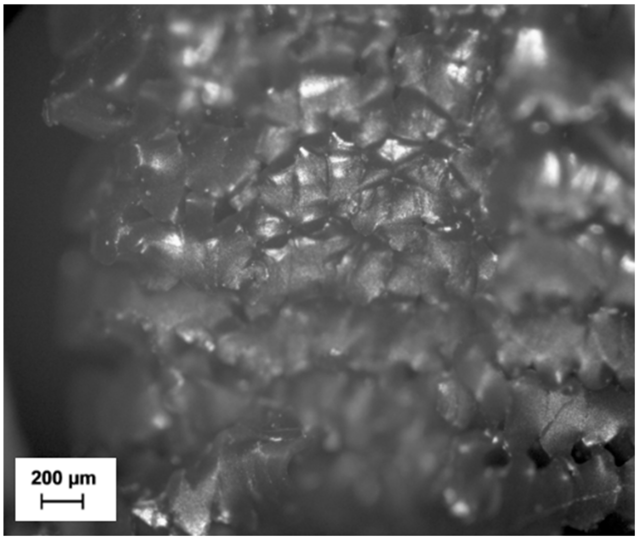

Fig. 12. Fracture of R-PETG manufactured by horizontal filament deposition at the temperature of 235°C

Empty spaces between successive passages in Figure 10 are visible. The phenomenon occurs as a result of too rapid material cooling. R-PETG requires a higher printing temperature. Probably, the separation of the threads is caused by cooling and heat convection, to which the material surface is the most exposed. For materials with horizontal filament deposition, joining successive threads is better, as the fractures presented in Figures 11 and 12 are visible. R-PETG fractures are more ductile than R-PLA ones. They are characterised by visible pieces of plasticised threads (Fig. 10). The scale formed on the R-PETG surface is large, pointed, uneven and stretched (Fig. 12). In the case of R-PLA (Fig. 11), a more flat surface with a circular shape and an even distribution of scales is visible.

6. Potential and attractiveness evaluation of tested materials

Because of the increasing share of additive technologies in everyday life and the current ecological and recycling

		PLA			R-PLA			PETG			R-PETG		
Potential criteria	Symbol	Weight	Expert score	Weighted score	Weight	Expert score	Weighted score	Weight	Expert score	Weighted score	Weight	Expert score	Weighted score
Material strength	P1	0.15	6	0.90	0.15	6	0.90	0.15	5	0.75	0.15	5	0.75
Material availability	P2	0.35	4	1.40	0.35	5	1.75	0.35	6	2.10	0.35	7	2.45
Length of the production chain	P3	0.25	5	1.25	0.25	3	0.75	0.25	8	2.00	0.25	4	1.00
Area of use	P4	0.25	5	1.25	0.25	5	1.25	0.25	7	1.75	0.25	7	1.75
Potential weighted average		1.00		4.80	1.00		4.65	1.00		6.60	1.00		5.95
		PLA			R-PLA			PETG			R-PETG		
Attractiveness criteria	Symbol	Weight	Expert score	Weighted score	Weight	Expert score	Weighted score	Weight	Expert score	Weighted score	Weight	Expert score	Weighted score
Price of the material	A1	0.30	7	2.10	0.30	8	2.40	0.30	6	1.80	0.30	7	2.10
Lifespan of the material	A2	0.25	5	1.25	0.25	3	0.75	0.25	9	2.25	0.25	8	2.00
Eco-friendliness	A3	0.15	8	1.20	0.15	7	1.05	0.15	4	0.60	0.15	7	1.05
Biodegradability	A4	0.30	7	2.10	0.30	7	2.10	0.30	4	1.20	0.30	4	1.20
Attractiveness weighted average		1.00		6.65	1.00		6.30	1.00		5.85	1.00		6.35

Fig. 13. Evaluation criteria of the researched materials' potential and attractiveness

trends, the potential use of the materials presented in the paper by a heuristic analysis using a dendrological matrix [20] was analysed. For this purpose, criteria for the attractiveness and technological potential of the materials by assigning appropriate weights to each characteristic were distinguished. The expert evaluation of each criterion ranges from 1 to 10, with 1 being the worst, the most difficult and/or least profitable option and 10 being the best, easiest and/or most profitable one. A summary of the selected potential and attractiveness criteria is presented in Figure 13.

The evaluation criteria for potential are as follows: material strength, availability, length of the production chain and area of use. The strength of the material on the basis of the static tensile test results presented in this publication and results obtained by other authors were assessed. It was assumed that they reflect the mechanical properties of the material. Material availability is the ease of obtaining the material and its amount in the environmental circuit. The length of the production chain refers to the complexity of creating the filament from the raw materials. Area of use refers to the products obtainable in both small and large series production.

Criteria for assessing attractiveness include the price of the material, the lifespan of the material, the eco-friendliness of the process and biodegradability. The price of a material is the market price per unit. The lifespan of a material determines how many cycles the material can survive in a closed loop. The eco-friendliness of a process determines the energy consumption and the amount of greenhouse gasses emitted to produce a material unit. Biodegradability determines whether and how quickly a material will decompose in the environment.

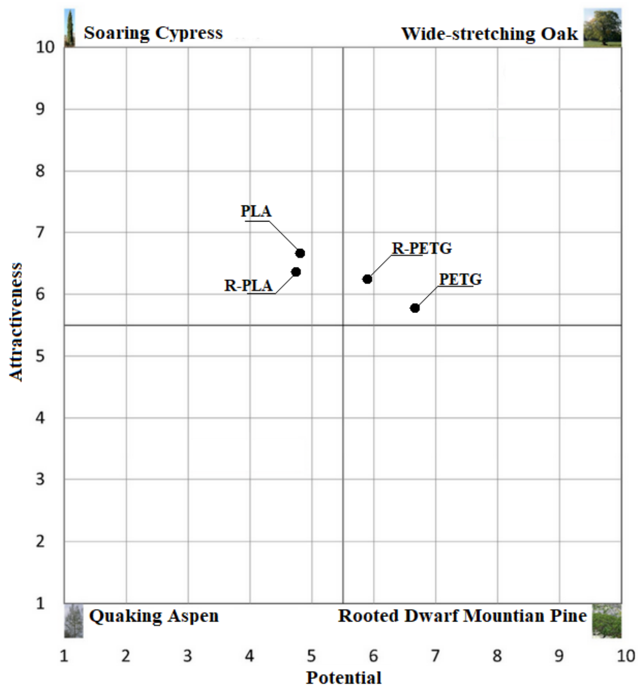

Fig. 14. Positioning of the tested materials using the dendrological matrix in terms of the criteria in Fig. 13 presented

The dendrological matrix (Fig. 14) shows that PETG and R-PETG are located in the quarter called "Wide-stretching oak". It indicates that they have the highest development potential and are attractive from the customer's point of view. Moreover, R-PETG is rated higher because of the environmental friendship of the manufacturing process. PLA and R-PLA in the quarter called "Soaring cypress" are included due to their high attractiveness but limited development potential, mainly during the limited lifespan of the material and availability.

7. Recapitulation

The researched R-PLA and R-PETG materials manufactured from recycled raw materials are qualitatively similar to their equivalents (PLA and PETG, respectively) created from new raw materials.

Among materials with vertical filament deposition, the highest tensile strength of 37.6 MPa has R-PLA manufactured at the temperature of 220°C. Its equivalent PLA manufactured from the new raw materials under the same process conditions has Rm slightly lower, i.e. equal to 37 MPa. For materials with horizontal filament deposition, the highest Rm of 67.8 MPa for PLA manufactured at 210°C was recorded. A value of 66.6 MPa for R-PLA manufactured under the same conditions was obtained. The tensile strength of the second tested material is lower. A maximum Rm of 20.3 MPa for PETG and 19.2 MPa for R-PETG deposited vertically at the temperature of 235°C were noted. Higher Rm values for materials with horizontal filament deposition were obtained. The best results at the temperature of 235°C with an Rm value of, respectively, 58.4 MPa for PETG and 57.2 MPa for R-PETG by the testing machine were noted.

The deposition method significantly determines the Rm value. In the case of PLA and R-PLA, horizontal filament deposition allows for obtaining a material that is almost twice tensile-resistant compared to vertical deposition. For PETG and R-PETG, the difference is even greater and is at almost three times. Differences in the process temperature, graded every 10°C, within the ranges close to the values recommended by the producer, generate changes in Rm up to 25% maximum. The smallest impact on the obtained Rm values was observed when comparing materials made from recycled raw materials (R-PLA and R-PETG) with their equivalents made from new raw materials, i.e., PLA and PETG. The observed differences usually equal a few per cent. The results of the microhardness tests are compatible with the results of the tensile tests, meaning that the harder, the more tensile-resistant material. The microscopic investigations revealed the structure specificity of materials with vertical and horizontal filament deposition. The observation of the sample fractures indicates the R-PETG ductility.

The carried out material science experiments and the heuristic analysis results of the potential and attractiveness of the compared materials show that, in both private and industrial applications, it is reasonable and purposeful to reduce the use of new raw materials in the manufacturing process using FDM/FFF technology in favour of replacing them with recycled raw materials. In future, it is planned to extend the carried-out research to other types of polymers and materials derived from several successive recycling cycles in order to test the feasibility of creating a closed loop of these materials.

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