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The Influence of Selected Parameters of Recycled Polyvinyl Butyral on the Sustainable Filament Extrusion Process

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Abstract: In recent years, sustainability has permeated nearly every aspect of our lives, and the manufacturing sector is no exception. The terms "sustainable manufacturing" and "zero-waste manufacturing" are now part of our everyday vocabulary. This study, which explores the influence of key parameters on the filament extrusion process using recycled polyvinyl butyral (PVB), which is an amorphous polymer commonly obtained from the glass recycling industry, has significant practical implications. By determining the optimal conditions for the extrusion process, we can enhance the mechanical properties of the produced PVB filament yarns and their printability. As a result of identifying errors, optimizing the process, and eliminating the resulting shortcomings, a fiber made of PVB material with a diameter of 1.75 mm (±0.06 mm) was created that can be used in most FDM devices. The length of the created fiber was approx. 20 m, and in the presentation of the results, it will be used for printing samples, adhesion tests to the printing mat, shrinkage tests, and tensile tests of the fiber. After removing all the shortcomings, the ideal extrusion temperature was at 155 °C. This temperature was verified using microscopic cross-sections, and deformations or changes were observed in their cross-sections. The deviation of the material currently undergoing testing for the adhesion of PVB to various types of print beds, which was found suitable for use in FFF devices, was 1.75 −0.25/+0.25. This, in turn, can significantly expand the application of these materials in additive manufacturing, thereby making a substantial contribution to the advancement of sustainable manufacturing.

Keywords: filament extrusion; additive manufacturing; industry

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Citation: Martiček, M.; Tauberová, R.; Kaščak, J.; Vandžura, R.; Sukić, E.; Knapčíková, L. The Influence of Selected Parameters of Recycled Polyvinyl Butyral on the Sustainable Filament Extrusion Process. *Appl. Sci.* **2024**, *14*, 9752. https://doi.org/10.3390/app14219752

Academic Editor: Dionysios E. Mouzakis

Received: 5 September 2024
Revised: 11 October 2024
Accepted: 16 October 2024
Published: 25 October 2024

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1. Introduction

Polyvinyl butyral (PVB) is a thermoplastic polymer composed of vinyl butyral and vinyl acetate monomeric units. PVB has outstanding properties, such as high tensile strength, impact resistance, optical clarity, strong adhesion properties, and flexibility [1]. PVB can be used for the production of structural adhesives, coating and as a binder to ceramics. Nowadays most common applications of PVB are in the automotive industry, in which PVB is used as an interlayer for car windshields, architecture, in which it has applications for skyscrapers as safety glass, and the solar industry for photovoltaic panels, in which PVB is used for the encapsulation of photovoltaic cells to protect them against mechanical damage [2]. As mentioned, recycled PVB can be obtained from car windshields,

which contain PVB foil. In general, the following two possible approaches for car windshield recovery can be distinguished: material recovery (recycling) and energy recovery. The appropriate form of recycling depends on the legislation of the specific country [3]. Since PVB is an essential component of each car, it is subject to Directive 2000/53/EC of the European Parliament and the Council of 18 September 2000 on end-of-life vehicles. According to Eurostat, in 2021, the total number of end-of-life vehicles was estimated at 5.7 million, an increase of 5.7% compared with the previous year, 2020 [4]. According to the dataset, it is possible to predict that the number of end-of-life vehicles will grow rapidly in the following years. At this point, it is crucial to recycle all raw materials coming from cars after their end-of-life cycle. As mentioned above, PVB has many applications in industry, mostly automotive, but additive manufacturing is another prospective area in which PVB can be applied. This emerging technology is well-known nowadays, and it can be stated that 3D printing is part of sustainable manufacturing. In comparison to traditional types of manufacturing, such as turning or milling, minimal waste is generated during the whole process of additive manufacturing. Filament is key to creating a physical object from a 3D model [5]. Currently, the use of materials in 3D printing is diverse. For the proposed study, PVB was chosen as the input material for the extrusion process. The extrusion process's advantage is the possibility of producing its filament from a wide range of materials. This device produces filament for additive manufacturing from raw plastic pellets. Recently, this instrument has become more popular in the consumer market. The utilization of this device allows users to produce their filament from peculiar materials. The production of filament with the utilization of an extruder reduces the final cost of the manufactured filament. The most commonly used extruders use the screw extrusion process for creating different composite filaments. As a feedstock to the extruder, it can be used in diverse materials, such as PLA, ABS, or polyvinyl butyral pellets. The section on related works offers a perspective on the literature review, which is closely related to the specific problem of the proposed manuscript. Regarding the experimental studies mentioned below, which are closely related to the proposed research, it can be concluded that their content and results of performed measurements influenced the proposed research. Seibert et al., in their research, have focused on extruding recycled PET bottle flakes into filament and have estimated several dimensions for the material extrusion (MEX) process [6]. Krolikowski et al., in their paper, discussed the recycling possibilities of PVB waste from vehicle windshields and building glass for reuse [7]. A recent study by Balint et al. contributed to the filament production area of research by offering a detailed description of the extrusion process and how selected parameters can affect optimal resulting filament quality [8]. Hartig et al. have employed the Taguchi method to optimize the filament production process, intending to reduce irregularities and improve the filament quality; applying the Taguchi method can help ensure better performance in diameter homogeneity [9]. Turku et al. have investigated several mechanical properties of fabricated filament from acrylonitrile butadiene (ABS) and polyvinyl chloride (PVC). The obtained results from the measurements (glass transition temperature, tensile properties, and pyrolysis scenario) were evaluated. The results show differences in virgin and recycled materials' melt flow indices (MFI). The MFI of recycled ABS is significantly lower than that of virgin ABS material [10]. Šooš et al., in their study, have provided a methodology for the recycling process of laminated glass, which consists of PVB interlayer film. The recycling methodology consists of several steps, such as mechanical separation, vibration, and chemical cleaning of the PVB film. Additionally, the study proposed the equipment design needed for the separation method [11]. Although the related studies peripherally address this specific area of research, no investigation deals with filament production using the extrusion method from recycled PVB.

Work Methodology and Research Gap Identification

As mentioned in the Introduction section, the possibility of the application of several divergent materials in additive manufacturing is remarkable [10]. Undoubtedly, the technology of additive manufacturing is ongoing nowadays, and not only the technology but

everything that comes together in general will definitely be ongoing in the future [11]. The cross-connection of this technology into almost every industry forms opportunities for diverse studies that can be conducted in this field [12]. The proposed research points out the connection between additive manufacturing technology and the application of specific materials, such as polyvinyl butyral (PVB). PVB has significant mechanical properties, which are the study's main subject of investigation. The proposed paper's overall methodical steps are depicted in Figure 1, in which decisive chronological steps are briefly outlined. The work's introduction focuses on the filament's current state and production process using the extrusion method. The filament production process consists of several steps that must be completed [13]. Therefore, the study points out the optimization of production. A further part of the manuscript is focused on thermovision diagnostic measurement using a thermal camera during the extrusion process and the basic definition of selected terminology in specific areas [14]. The following part of the work is applied to the practical section, in which the non-contact measurement of a particular electrical machine extruder is performed. The measurement can be evaluated in which way and how the temperature affects the extrusion process.

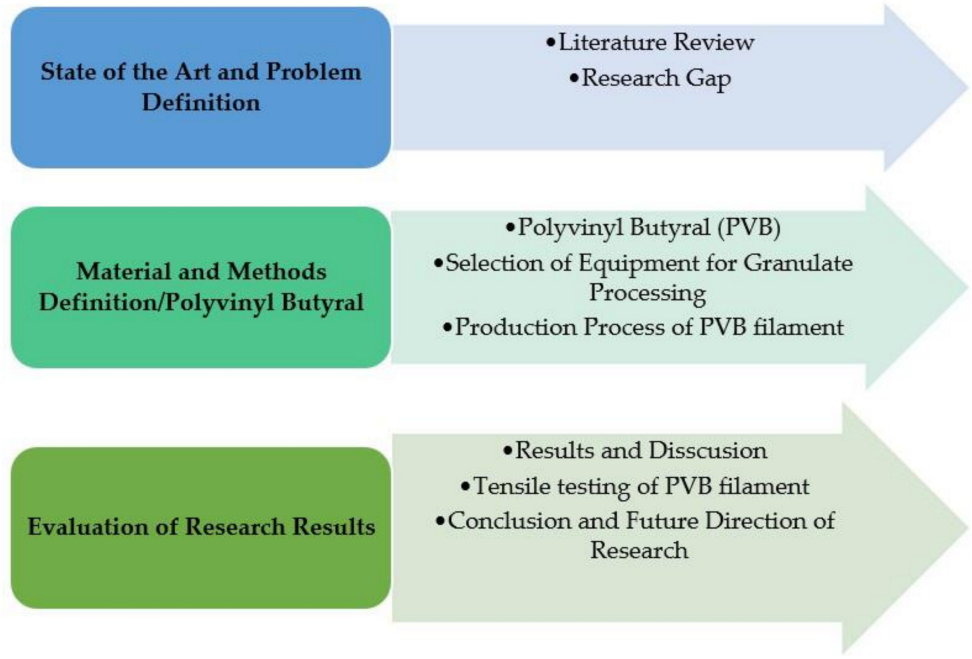


Figure 1. Methodological framework of the research.

The research focuses on determining the optimal conditions for the extrusion process to enhance the mechanical properties of produced PVB filament yarns, their printability, and their application in additive manufacturing.

2. Materials and Methods

Car windshields are composed of two types of bent sheet glass, between which a foil is glued for increased glass safety [14]. The foil that fulfills the safety function is polyvinyl butyral; it prevents fragments from getting inside the car in an accident and breaking the windshield, thereby not endangering the car crew. The PVB structure is built up of approximately 81% polyvinyl butyral, 18–23% polyvinyl alcohol, and less than 1% polyvinyl acetate. PVB film is characterized by extreme strength, adhesion, and flexibility [15]. The technological process of waste windshield processing begins with loading into the processing line through the loading part with a loader. With the help of conveyors, the material reaches the equipment, removing unwanted substances. Physical and mechanical processes gradually remove unwanted elements, such as dirt, from the shards [16]. The foil remains separated in this way. As mentioned above, glass without

foil is a 100% recyclable raw material. Glass granulate is an output raw material for the recovery of glass and is further used as an input raw material in the glass industry, replacing glass sand [17]. The raw materials created in this way form a significant part of the input raw materials that save the environment. Their environmental impact is minimal, so they currently play an important role in selecting suitable raw materials for input. Recycled PVB is obtained from car windshields by separating the PVB foil. The glass is then shredded using a cylindrical branch crusher [15]. Further size reduction occurs in a clad mill, in which the PVB foils are ground to a smaller size of approximately 20 cm^2. After separating the materials using vibrating machines, impurities are removed. Recycled PVB for consumers is available as PVB flakes, PVB granulate, PVB foil, and PVB filament [16]. The recyclability of polyvinyl butyral as a raw material upholds the fundamental principles of the circular economy. The following Figure 2 shows recycled PVB in four different forms.

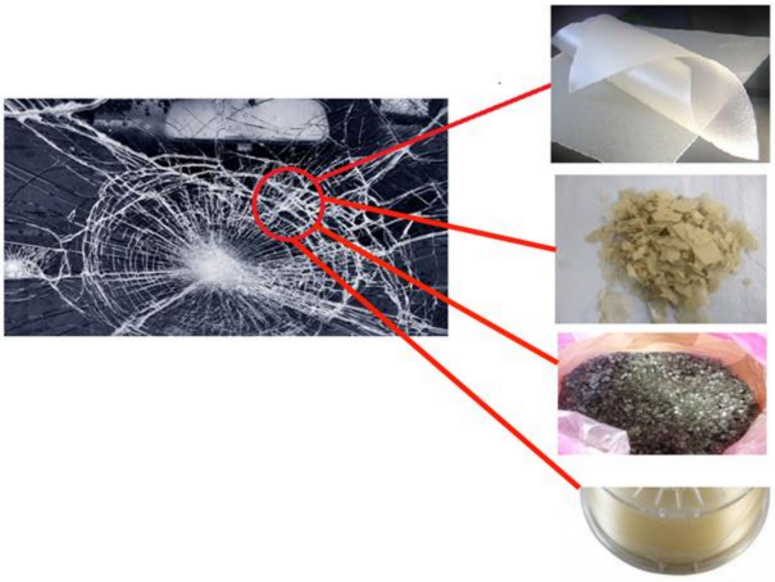


Figure 2. Recycled PVB and its forms (from up to down): foil, flakes, granulate, and filament.

The structure and properties of PVB profusely depend on the hydroxyl content. For instance, the hydroxyl content controls the adhesion to surfaces, influences cross-linking behavior, improves the properties of thermoset resin, and affects the miscibility and morphology of blends [17]. A terpolymer is a chemical compound from a polymer with a molecular structure built mostly or completely from many similar units bonded together (such as a complex resin). Results from the copolymerization of three different monomers are sometimes used to prevent corrosion. PVB is generally an amorphous polymer with very high VA content (higher than 63.3%). The melting temperature of PVB is between 171 °C and 218 °C (was made according to standard DIN 53015) [18]. Table 1 below represents PVB material characteristics.

Table 1. Material characteristics of polyvinyl butyral.

Material PVB—Polyvinyl Butyral, Recycled	
Form	Flakes
Melting temperature	130–170 °C
Color	Transparent
Viscosity	100–175 m Pa·s (DIN 53015)
Melt volume rate (MVR)	6–7 cm^3/10 min
Melt flow rate (MFR)	5–6 g/10 min

Preparing recycled polyvinyl butyral is an important step toward achieving the work's goal. The material itself naturally absorbs ambient moisture. Therefore, getting the material

to the most suitable state for processing is necessary before starting work. The preparation of polyvinyl butyral took place in laboratory conditions at the Faculty of Manufacturing Technologies of the Technical University of Košice at a room temperature of 22 °C and a humidity of 60%. The water content was determined using a gravimetric analysis, representing the water in the tested sample according to the standard DIN ISO 11465 [19]. Polyvinyl butyral (PVB) has gained attention as a filament material in 3D printing due to its unique flexibility, durability, and transparency [20]. Before testing, the material was dried at 60 °C for 24 h. The material is better adapted to similar materials, such as polylactic (PLA); its advantage over the mentioned material is its mechanical properties, which are at the level of materials such as glycol-modified polyethylene terephthalate (PETG) and co-polyester (CPE) [12,17]. Its other distinguishing feature, which is directly related to FDM support, is good adhesion to printing pads. On the other hand, its disadvantages include low heat resistance or poor adhesion in the print layers. The work's introduction focuses on the filament's current state and production process using the extrusion method [12,20]. The filament production process consists of several steps that need to be completed. Therefore, the study points out the optimization of production [20]. A further part of the manuscript is focused on thermovision diagnostic measurement using a thermal camera during the extrusion process and the basic definition of selected terminology in specific areas [20]. The following part of the work is applied to the practical section, in which the non-contact measurement of a particular electrical machine extruder is performed. The measurement can be evaluated by which way and how the temperature affects the extrusion process. This study focuses on determining the optimal temperature for the extrusion process of polyvinyl butyral.

The material is also hygroscopic and absorbs moisture comparably to PLA material (Table 2). Generally, the required filament diameter should be 1.75 mm. Still, a slight deviation may occur while producing the recycled filament, and the allowed deviation is 0.5 mm.

Table 2. Comparison of mechanical properties of PLA and PVB materials (tensile test DIN EN ISO 527-1) [21].

	PLA (z)	**PVB (z)**	**PLA (x,y)**	**PVB (x,y)**
Tensile Yield Strength [MPa]	51 ± 3	50 ± 5	59 ± 2	49 ± 5
Tensile Modulus [GPa]	2.3 ± 0.1	1.7 ± 0.1	2.4 ± 0.1	1.7 ± 0.1
Elongation at Yield Point [%]	2.9 ± 0.3	4.6 ± 0.7	3.2 ± 1	4.4 ± 0.7
Flexural Strength [MPa]	83 ± 6	72 ± 1	99 ± 1	73 ± 3
Flexural Modulus [GPa]	3.1 ± 0.1	2.2 ± 0.1	3.2 ± 0.1	2.3 ± 0.1
Deflection at Flexural Strength [mm]	7.5 ± 0.2	8.4 ± 0.4	8.3 ± 0.2	8.5 ± 0.3
Impact Strength Charpy [kJ/m^2]	13 ± 1	55 ± 7	14 ± 1	59 ± 12

As we can see in the table, PVB is very similar to the most commonly used material for FDM technology, PLA, with a few differences [22]. The compared samples were made in two variants. The first variant, in Table 2, which is marked with the adjective "z", was printed vertically, while the pattern "x,y" was printed horizontally. The only diametrically different parameter is the Charpy test, in which the PVB material has several times better results.

2.1. Differential Scanning Calorimetry

Information about the tested material's processing possibilities is obtained by determining the glass transition temperatures (Tg) and melting temperatures (Tm) for recycled polyvinyl butyral. The method used in the testing is differential scanning calorimetry (Netzsch 204 DSC, Selb, Germany). The samples were analyzed in laboratory conditions at a room temperature of 22 °C and a humidity of 60% using the DIN ISO 113 57 standard [23]. The test temperature ranged from −50 °C to 300 °C. The measurement process began with cooling the sample to −180 °C and heating it. The key temperature for starting the analysis

was −50 °C. The sample is heated only after reaching a temperature of +300 °C. Using differential scanning calorimetry, the sample is subjected to linear heating and thus to the rate of heat flow in the sample [24]. The heat flow is proportional to the instantaneous specific heat (Figure 3). Two symmetrical containers were placed inside the measuring device, whose internal temperature was equal to room temperature (i.e., ca. 20 °C).

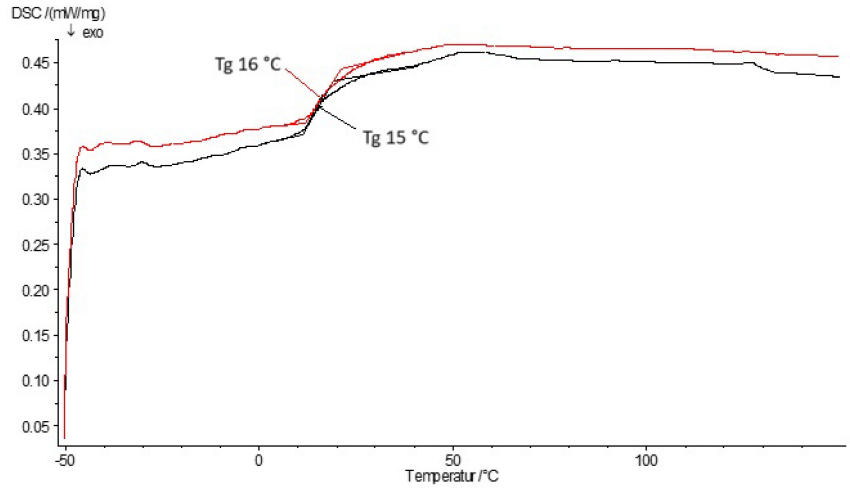


Figure 3. Graphic display of analysis of differential scanning calorimetry-1. heating.

The difference between the first and second heating (Figure 4) can be seen from the graphical representation obtained through the evaluation program NETZSCH Proteus, version 6.0, Germany. It means that the imprecise sample settles in the test aluminium container during the first warm-up. In the second case of heating, the final analysis of the material was obtained. The red color describes recycled polyvinyl butyral (1st heating), and the black colour is for recycled polyvinyl butyral (2nd heating).

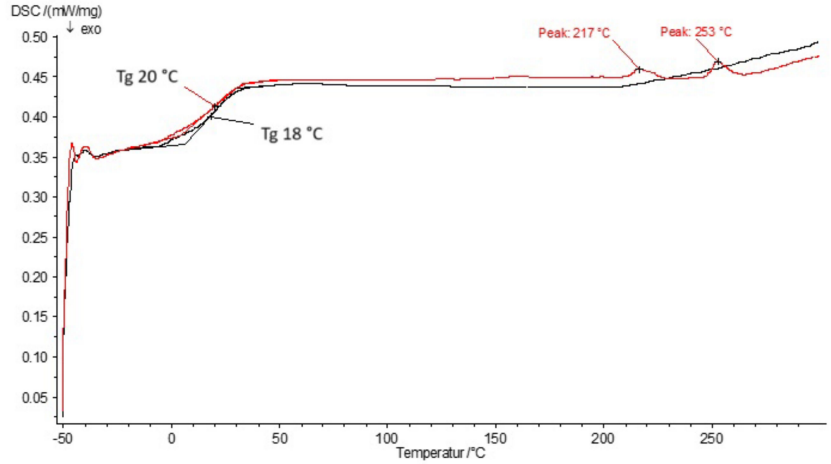


Figure 4. Graphic display of analysis of differential scanning calorimetry-2. heating.

Glass transition temperatures for recycled PVB material range from 18 °C to 20 °C. The melting temperature Tm is higher, between 217 and 253 °C.

2.2. Selection of Equipment for Granulate Processing

This section applies to selecting appropriate equipment for filament production and its basic attributes. As mentioned above, the extruder is chosen for filament production [23]. This electrical device is used for granulate processing to fabricate filament from recycled granulates. The filament production itself begins when the pellets are poured into the

pellet's hopper, and then the pellets are fed into the extrusion screw [24]. The pellet extruder has several heating zones; in the first part of the extrusion screw, the temperature is usually higher to ensure uniform material melting [25]. The operational rotational speed range of the device used for extrusion is 5–25 rpm. Considering the nature, properties, and potential deformations that may be caused by the discontinuous winding of PVB material, an initial/optimal speed of 15 rpm was determined. This rotational speed of the extrusion screw, along with the speeds of other device components, such as the feeder and winder, ensured the ideal tension of the filament during extrusion and minimised the occurrence of deformations in the cooling zones during the initial phase, specifically at the start of extrusion and the initial winding of the produced filament. It is important to note that the device is equipped with an optical diameter control system. This optical sensor/micrometer is located before the feeder and continuously monitors the filament diameter along one axis. In the event of a deviation greater than the calibrated value, automatic corrections to the extrusion speed, feeder, and winder are made. When pellets are melted into the desired liquid, the movement of the auger screw builds up the pressure at the end part of the extruder, where the melted plastic is pushed through the extruder nozzle. A FilaFab Pro EX350, D3D Innovations Limited, Poole, UK (Figure 5) extruder was chosen for the experimental part of the proposed study. The Filafab 350EX, D3D Innovations Limited, UK, extruder features a single zone where the filament is melting. Due to its design, the manuscript mentions shortcomings associated with the unintentional heat spread toward the hopper. This single heating zone is adequate for extrusion and filament production. However, for PVB material, the cooling of the extrudate proved to be more challenging, requiring cooling at multiple points. Rapid and abrupt cooling, such as in a standard water bath, caused deformations during extrusion, while slower cooling led to material deformation by the feeder. A multi-zone active cooling system was designed to extrude PVB material to address these issues. This approach prevented internal stresses, filament deformation in the neck area at the nozzle exit, deformation caused by the feeder, and other undesirable effects. As shown in the following figure, the maximum operating temperature of the extruder is 250 °C.



Figure 5. Extruder FilaFab Pro EX350.

These data may appear on the edge or even insufficient compared to the normal temperatures that the PVB material requires during printing [26]. The processing of granulate into filament is different in terms of temperatures; as the standard, temperatures that are an order of magnitude lower are used during filament production. For example, when extruding PLA material, the temperature during printing is 180–205 °C, but the temperature during production is in the range of 120–135 °C. Thus, the device is sufficiently efficient when it comes to the processing of PVB material granules. Generally, the filament production process can be seen as quite elementary, but some challenging issues may be present during the extrusion. For instance, optimizing laboratory conditions (right temperature) and proper granulate storage prevent the wetting of materials before extrusion starts. Based on the series of experiments, it can be stated that granulate must be properly dried for at least three hours in the dryer [26]. Another challenging issue is the determination of the right extrusion temperature. If the temperature is too high, it can

subsequently cause a small filament diameter or bubbles inside the filament; therefore, the resulting filament is useless. If the temperature is too low, we can observe the diameter of the filament is slowly getting bigger. Generally, the required filament diameter should be 1.75 mm, but while producing the recycled filament, slight deviation may occur, and the allowed deviation is 0.5 mm. The deviation of the material currently undergoing testing for the adhesion of PVB to various types of print beds, which was found suitable for use in FFF devices (both with direct and Bowden extruders), was 1.75 −0.25/+0.25. For the filament, this represents a relatively high deviation, which may lead to jamming in the print head, filament sensor, or the PTFE tube of a Bowden-type extruder head. It is important to note that these values represent the highest measured deviations, occurring in the areas where automatic correction of process parameters took place. Despite these being maximum deformations within a relatively small range, the authors decided not to exclude these data from the publication, given the informational nature of the work. The publication aims to thoroughly describe the preparation and challenges associated with processing PVB material for sample preparation and further testing.

Some other issues which can happen during extrusion are listed below.

- Nodules or particles in the fiber.

Unwanted particles are probably the most characteristic problem in this area. Particles of other polymers, waste, or pieces of metal from the body of the extruder are common impurities that we can deal with in this process.

- Insufficient diameter of the extruded fiber.

It is important to ensure sufficient material output for the required filament thickness when producing filament. The device has an IR sensor and an optical micrometer that monitors the process. This equipment oversees the entire process and performs the necessary corrections to ensure the continuous extrusion of filament with the appropriate diameter. There are several ways to identify this problem. One cause of low output capacity is an auger speed that is too low. If the auger speed is too slow, insufficient material is pushed through the drill, resulting in low output capacity [16].

- High speed of the feeder.

The feeder pulls the filament out of the extruder at excessively high velocity. This process narrows the neck of the extrudate in the part where the temperature is too high (180 °C).

- Low outlet temperature.

This can cause restricted flow. The extruded volume can change if the material does not melt or melts too quickly (120 °C). If the material is melted too early, sufficient pressure will not be created inside the output cylinder, which is necessary to develop a high-quality filament.

- High outlet temperature.

The melt flow index (MFI) is used to compare the viscosity of different materials. Each material has its own unique MFI value. Materials with a high MFI (>15 g/10 min) have a low viscosity, resulting in a very fluid output. The material's properties cannot be changed, so it is best to choose a material that will meet the needs of the given assignment [20]. For PVB, the relevant melting temperature is 130–170 °C. When the material has a suitable MFI, you can increase its viscosity by lowering its temperature. Of course, increasing the fan speed is the easiest way to cool the material. But the simplest way is only sometimes enough. In this case, the temperature of the heaters can be reduced. By lowering the temperature of the heaters, the material cools and thus increases the viscosity (130–170 °C). It is also possible to increase the speed of the drill, thereby reducing the time the material spends heating up. However, increasing the velocity increases the barrel friction, which could cause an increase in temperature rather than viscosity [27]. The material's roughness is important in 3D printing [3,17]. In printing, the recycled polyvinyl butyral (PVB) material is applied

layer by layer, each touching the previous and subsequent layers. Suppose the material's surface is too rough. In that case, it can lead to undesirable effects, such as increased friction between the layers, which can lead to abrasion or the deformation of the final product [23]. Scratches, burrs, or foreign material in the nozzle can cause surface defects in the die area. The fault causes a deepening of the fiber that remains in the same place over time. If foreign particles cause a problem on the edges of the nozzle or in the nozzle, the nozzle should be cleaned to eliminate the problem [4,9]. Any scratches in the nozzle area must be mechanically removed to eliminate the problem. Lines or streaks on the fiber can be caused by material build-up around the edges of the nozzle [25]. This can happen if the nozzle temperature is too low for the material used, especially for semi-crystalline polymers. If the material comes into contact with the cold nozzle, it can solidify and form lines on the printed object [27]. From this point of view, it is necessary to determine the optimal extrusion temperature for a specific material—PVB. For the determination of optimal extrusion temperature, thermovision diagnostics were applied.

3. Results and Discussion

To identify the optimal extrusion temperature for PVB material, we have chosen thermovision diagnostics. For measuring the temperature during the filament production, we have used Thermo camera Flir T 425, W-Technika group., Prague, Czech Republic, and its cooperating software, Flir Thermal Studio Suite, Flir, Prague, Czech Republic, are used to better understand infrared data and create reports. The thermocamera measures the filament temperature produced by the extruder nozzle. The process of thermovision diagnostics starts at 175 degrees and gradually decreases by 5 degrees. The measurement aims to designate the optimal temperature for the extrusion process of polyvinyl butyral. To identify the optimal extrusion temperature for PVB material, we have chosen thermovision diagnostics. For measuring the temperature during the filament production, we have used Thermo camera FLIR T 425, W-Technika group., Prague, Czech Republic, and its cooperating software, FLIR Thermal Studio Suite v. 1.9.40, FLIR, Prague, Czech Republic, is used to understand infrared data better and create reports. The thermocamera measures the filament temperature produced by the extruder nozzle. The process of thermographic diagnostics begins at 175 °C and gradually decreases at 5 °C intervals. The measurement aims to designate the optimal temperature for the extrusion process of polyvinyl butyral.

3.1. Extrusion of Filament from Polyvinyl Butyral Granulates

Before filament fabrication starts, it is necessary to ensure the proper extrudepreviously previously previously used. This step is crucial for the resulting quality of the produced filament. As was suggested before, proper material drying before extrusion is an essential part of the whole process. Based on the experiment, we recommend drying the material for at least three hours. If these steps are successfully performed, it is possible to start the extrusion process [28]. The extruder has to be heated up at the required temperature. Filament yarns were extruded at different temperatures to reveal the optimal extrusion temperature for the chosen material. The first group of specimens was made at a temperature of 175 °C for extrusion. Then subsequent groups were made at 160 °C, 155 °C, and 150 °C, and the last group of specimens was extruded at a temperature of 140 °C. Figure 6 represents the process of filament production and measurements taken using a thermocamera.

These measurements consisted of stabilizing the nozzle temperature at 175 °C and gradually lowering it, stabilizing it, and measuring it until the material breaks.

In this case, the temperature of 155 °C appeared ideal for extruding fibers from this material. The temperature of 140 °C was already too low, the diameter of the material was not constant, and there was clogging of the nozzle and even fiber breakage. The subsequent verification of the established temperature was carried out using microscopic images. For this purpose, two sets of samples were selected. The first series consists of the following extremes: the highest and lowest extrusion temperatures [22,27]. These were intended to demonstrate the additional negative impact observable in cases of negative

or positive temperature extremes. The second series of samples comprise temperatures of 160, 155, and 150 °C, which appeared ideal upon simple visual inspection during and after extrusion. Considering that the filament itself, with a diameter of 1.75 mm, cannot be processed to a level at which individual defects are observable at the required level, the samples were prepared in advance—preparation involved embedding the samples into a casting resin commonly used for materials microscopy [28]. Three types of casting resins were gradually selected. Two of them were excluded due to insufficient filament visibility or defects arising during casting and a chemical reaction with the PVB material, resulting in material dissolution before the solidification of the casting resin. Figure 6 shows the preparation process of PVB filament samples, which were cold-hardened into acrylic resin Durocit. Samples were cold-hardened for better manipulation and stability during the observation under the microscope (Figure 7).

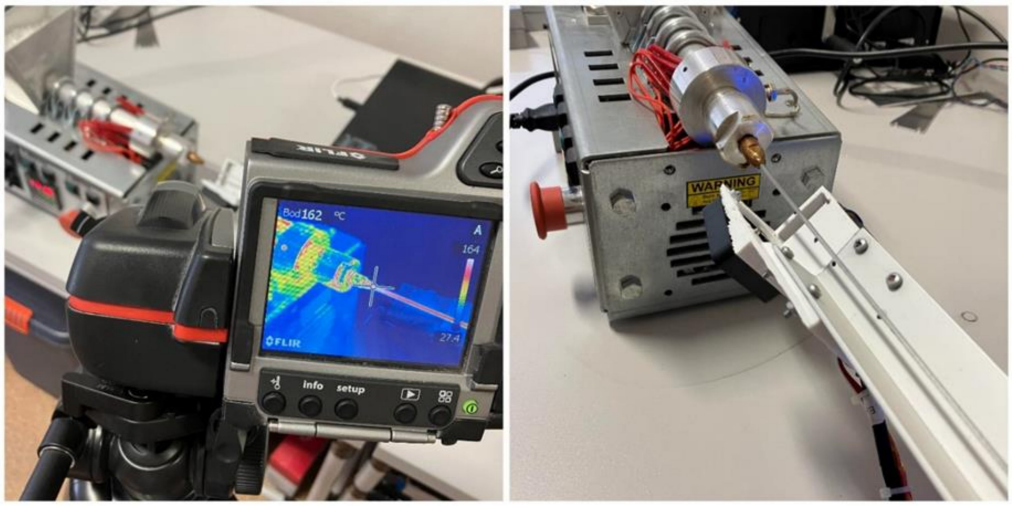


Figure 6. Thermovision diagnostics of filament extrusion.



Figure 7. Preparation of PVB filament samples embedded in DuroCit-3 material.

For a successful series of prepared samples, it is possible to declare the samples visible in Figure 8. The casting material DuroCit-3 was utilized for this purpose. The material did not chemically react with the material used, and sufficient color contrast was ensured between the observed sample and the casting material [27]. Given these results, the prepared samples were utilized to generate microscopic images. The Keyence VH-ZST device was employed to create microscopic images. As depicted in Figure 8, two images of the deformation of the extruded filament in the longitudinal direction were initially generated. These clearly illustrate two types of deformations in the case of incorrect extrusion temperature settings. Figure 8 displays a sample created at a temperature of

140 °C. Across the entire surface of the sample, visible irregularities are caused by the low extrusion temperature and discontinuous material feeding from the nozzle. The second image (Figure 8) represents a so-called "overcooked" sample extruded at a temperature of 170 °C. Its surface is notably smooth but contains small pores, which were created literally by the material overheating and the evaporation of the moisture contained within. These samples were deliberately produced at extreme temperatures to elucidate better the negative effect caused by incorrectly chosen temperatures.

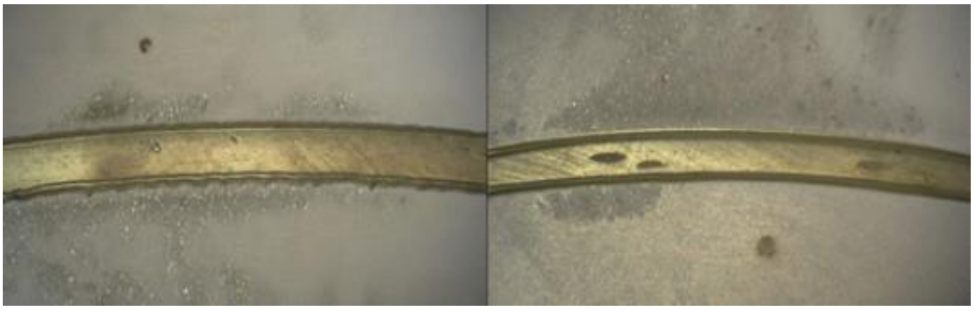


Figure 8. Microscopic images of filament produced at temperatures of 140 °C (left side) and 170 °C.

The next series of samples consists of cross-sections perpendicular to the extrusion axis. Essentially, this represents the previously mentioned verification of the phenomenon in which higher temperatures result in diameter deformation of the extruded filament. For this purpose, samples from the temperature range of 160 to 150 °C were employed. These samples appeared optimal during visual inspection and continuous diameter measurement during extrusion [27,28]. Microscopic images were utilized to finalize the ideal temperature, considering the filament's diameter deformation, as mentioned above. This deformation can be relatively easily identified in such images through a visible change in the shape of the fiber's cross-section from circular to elliptical. Through microscopy, the filament manufactured at a temperature of 150 °C was identified as ideal. As depicted in Figure 9, the filament exhibits an ideal circular cross-section, and the diameter (red line) of 1.75 mm (±0.06 mm) size corresponds to the commonly adopted diameter of fibers utilised in most FDM devices.

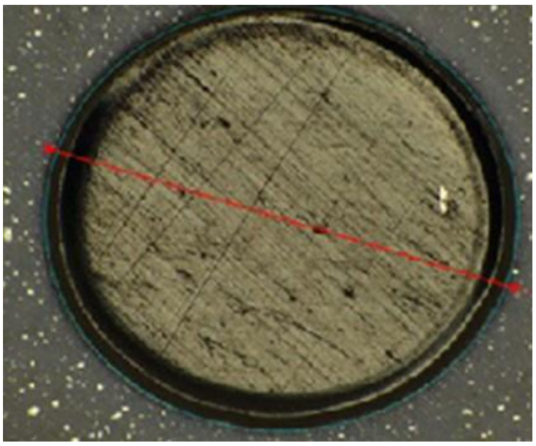


Figure 9. Microscopic image of the cross-section of PVB filament extruded at a temperature of 155 °C.

The cross-section contains only one defect; it is an impurity or a metal chip identified as part of a screw, which likely occurred due to the collision of another material with a high content of carbon fibers or shearing between the screw and the hopper wall. As this is a purely random and non-recurring error, which does not significantly impact the identification and verification of the optimal temperature of the PVB material, this defect is strictly incidental and irrelevant in this case.

3.2. Tensile Test of Extruded PVB Filament

Tensile testing was performed on a specific device, TestometricX350-5, Testometric Company Ltd., Oldham, UK according to standard DIN 527-1. The diameter of the produced filament yarns corresponds to 1.75 mm. A total of five groups of measurements were taken, and every group consisted of six specimens as stripes. Tensile testing was performed to specify which extrusion temperature is optimal for the best material characteristics of filament yarns [17,29]. The TestometricX350-5 device is a tabletop, two-column, computer-controlled universal testing machine for testing materials in tension, compression, bending, cyclic loading, adhesion, shear, hardness, and spring testing. A modern and functional design, the latest control electronics, and high-quality processing characterize the machine. The electronics are placed on silent blocks for protection against impact during tests. The machine is controlled using the modern Wintest Analysis software running on the Windows operating system [27]. The software is fully configurable and allows for the setup of simple and more complex multi-step test methods. WinTest Analysis software, EC 6.2.5 is a complex system working on the Windows operating system. It is fully integrated and fully customizable software that supports the creation of test programs according to ISO, EN, ASTM, ČSN, STN, DIN, BS, and other standards, including internal procedures and researchers' own specifications for testing materials [28,30]. With the help of this software, it is possible to perform all the aforementioned tests on the machine. The software allows for the setup of simple tests for normal material testing. For development or research needs, there is a possibility of setting up multi-stage loading. The used device, Testometric X350-S, measured mechanical properties more precisely for performing a mechanical tensile test [31]. The load capacity is 5 kN, the feed rate is from 0.00001 to 2000 mm/min, the height (without jaws) is 1100 mm, the width is 320 mm, and the measuring range is from 0.4% to 100% of the nominal value of the load [32]. The tested samples were made in a stabilized environment; each sample had a constant length of 160 mm and a diameter of 1.75 mm. Standard jaws equipped with preparation for the possibility of attaching the fiber were used for the tests [33]. A sample of PVB filament with a diameter of 1.75 mm at a temperature of 140 °C is clamped in the jaws of a Testometric X350-5 tearing machine (Figure 10). A tensile test was used to evaluate the material's tensile strength [34]. The test's principle stressed the test body until the sample ruptured. The test sample was clamped in the jaws of the tearing machine [35]. Due to the constant speed, it was stretched [29]. Along with the increasing deformation, the force needed to maintain a continuous speed of the displacement of the jaws of the tearing machine also increased [36]. Parts of the sample are still attached in the jaws of the tearing machine, which are taken out after loosening the screws that hold the sample.

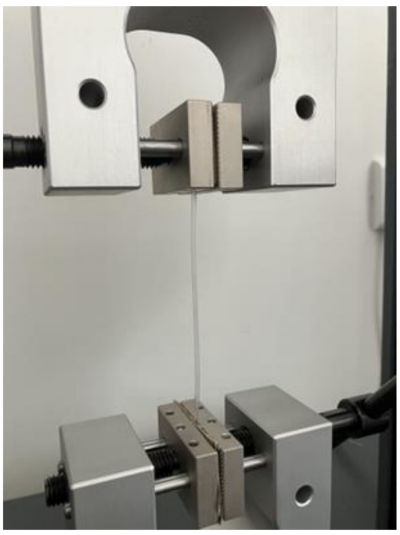


Figure 10. Clamping a 1.75 mm filament sample in the jaws of a Testometric X350-5 tearing machine.

The tensile test of the tested samples from recycled polyvinyl butyral (Table 3) took place at a room temperature of 23 °C and a humidity of 60%, according to DIN EN ISO 527-1.

Table 3. Mechanical properties (DIN EN ISO 527-1) [21] of the tested PVB filament with a diameter of 1.75 mm and a temperature of 140 °C.

Test No	Temperature	Force Peak (N)	Elongation at Break (%)	Elong. Peak (mm)	Strain Peak (%)	Tensile Stress (MPa)	Stress Yield (MPa)
1	140	104.53	43.459	12.327	12.327	0.020	43.454
2	140	108.19	44.98	25.187	25.187	0.021	44.959
3	140	100.08	41.608	5.165	5.165	0.025	41.588
4	140	104.05	43.259	4.197	4.197	0.017	43.255
5	140	128.27	53.328	3.328	3.328	0.021	53.312
6	140	118.54	49.283	8.549	8.549	0.037	49.221

The following graphic representation (Figure 11) shows the measurement progress of a PVB filament with a diameter of 1.75 mm. The tensile test works on the principle of stressing the test sample until it breaks [21]. The tested PVB filament sample is clamped in the jaws of the tearing machine and gradually stretched at a constant speed [37]. As the deformation increases, the strength increases. Elongation at break is measured in % (% of elongation vs. initial size when a break occurs) [38]. The maximum elongation at the break (Emax) is also called "strain to failure". The force is required to maintain a continuous displacement speed of the jaws of the shredding machine.

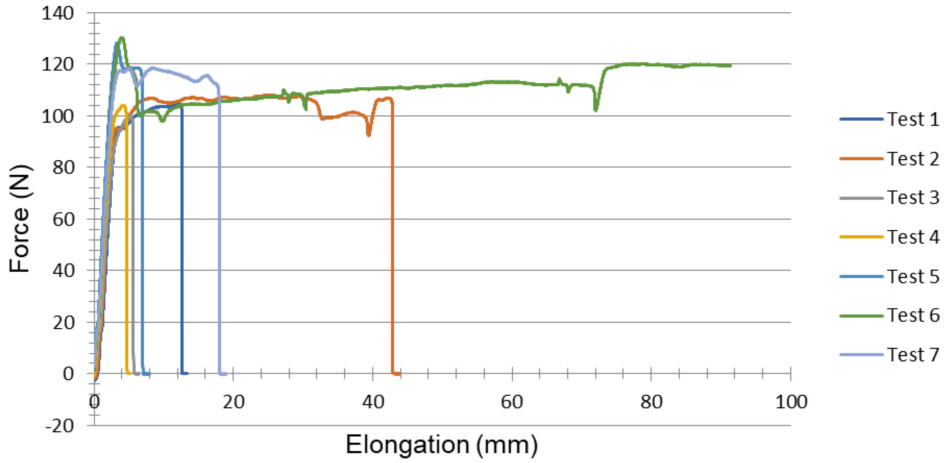


Figure 11. Graphic representation of a tensile test of a 1.75 mm filament with a temperature of 140 °C.

The following measurement (Table 4) occurred on a set of PVB filament samples with a diameter of 1.75 mm at a temperature of 150 °C and a speed of 50 mm.min^{-1}.

Table 4. Mechanical properties (DIN EN ISO 527-1) [21] of the tested PVB filament with a diameter of 1.75 mm and a temperature of 150 °C.

Test No	Temperature	Force Peak (N)	Elongation at Break (%)	Elong. Peak (mm)	Strain Peak (%)	Tensile Stress (MPa)	Stress Yield (MPa)
1	150	90.88	37.784	5.438	5.438	0.037	37.771
2	150	100.88	41.941	3.012	3.012	0.025	41.916

Table 4. *Cont.*

Test No	Temperature	Force Peak (N)	Elongation at Break (%)	Elong. Peak (mm)	Strain Peak (%)	Tensile Stress (MPa)	Stress Yield (MPa)
3	150	105.87	44.016	3.18	3.18	0.029	43.995
4	150	108.27	45.013	50.434	50.434	0.054	44.244
5	150	97.28	40.444	5.67	5.67	0.017	40.415
6	150	96.81	40.249	5.165	5.165	0.017	40.241

The tensile test evaluates the tensile strength of the material. The principle of the test is to stress the test body until the moment the sample breaks (Figure 12). In the tensile test, the test body is clamped in the jaws of the tearing machine and gradually stretched at a constant speed. With increasing deformation (Figure 12), the force required to maintain a continuous displacement speed of the jaws increases.

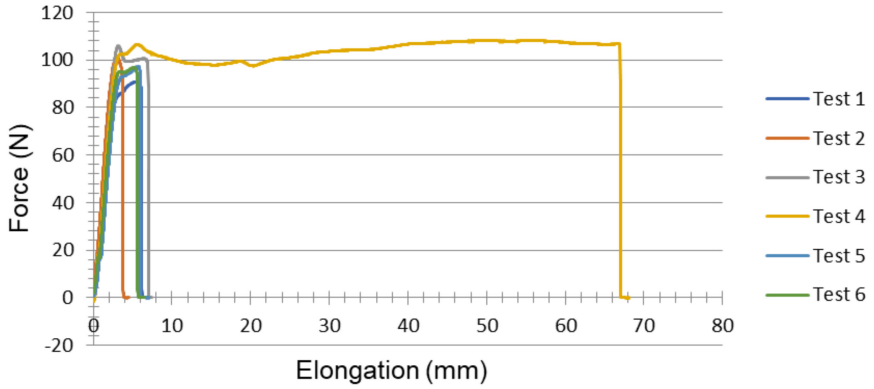


Figure 12. Graphical course of a tensile test of a PVB filament with a diameter of 1.75 mm and a temperature of 150 °C.

The dependence of stress on the amount of deformation is called the tensile curve, and its shape depends on the type of stressed material. Table 5 shows the results of the tensile test of the PVB material filament at a temperature of 155 °C. From the point of view of deformation behavior, the most frequently evaluated relative elongation of samples during the tensile test is expressed as a percentage of the original length of the working part of the test specimen. With small deformations of the sample (small values of relative elongation), changes in the cross-section of the sample can be neglected.

Table 5. Statistical processing of tensile test (DIN EN ISO 527-1) [21] data of PVB filament with a diameter of 1.75 mm and a temperature of 155 °C.

Test No	Temperature	Force Peak (N)	Elongation at Break (%)	Elong. Peak (mm)	Strain Peak (%)	Tensile Stress (MPa)	Stress Yield (MPa)
1	155	111.3	46.273	3.096	3.096	0.158	46.257
2	155	100.03	41.588	6.104	6.104	0.187	41.579
3	155	98.56	40.976	16.725	16.725	0.158	40.336
4	155	115.68	48.094	3.362	3.362	0.025	48.073
5	155	111.96	46.548	5.56	5.56	0.029	46.539
6	155	106.71	44.365	7.659	7.659	0.008	44.357

Figure 13 shows a tensile test of PVB filament at a temperature of 155 °C. The tested samples were subjected to a force stress of 50 mm.min^{-1}. In principle, the permissible load of the measuring head for force sensing must meet the range for the type of material being tested, which, in the case of polymers, represents a range from approximately 500 N for rubbers to 25 kN for very strong materials [39]. A visible change is in the relative elongation, which, at a temperature of 155 °C, shows values from 3.096% to 16.725%.

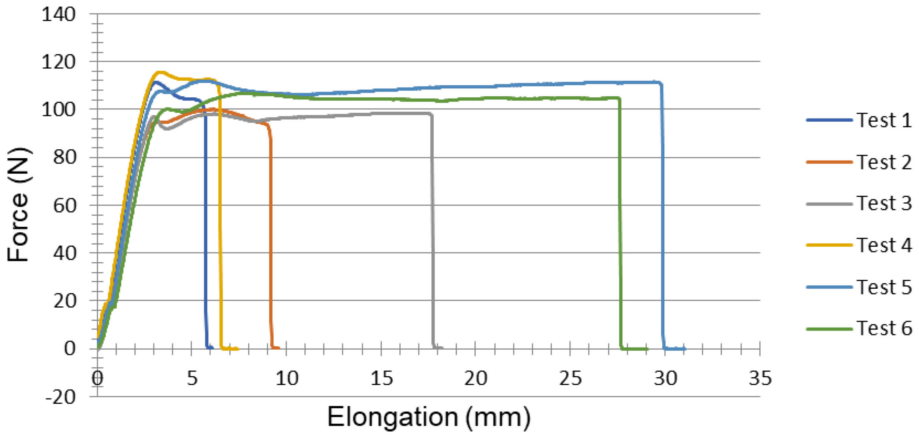


Figure 13. Graphic representation of a tensile test of a PVB filament with a diameter of 1.75 mm and a temperature of 155 °C.

The standard specifies a jaw displacement speed of 1 mm/min to calculate the modulus of elasticity in tension (Table 6). At the same time, the stress at elongations of 0.05 and 0.25% is subtracted from the tensile curve.

Table 6. Statistical processing of tensile (DIN EN ISO 527-1) [21] test data of PVB filament with a diameter of 1.75 mm and a temperature of 160 °C.

Test No	Temperature	Force Peak (N)	Elongation at Break (%)	Elong Peak (mm)	Strain Peak (%)	Tensile Stress (MPa)	Stress Yield (MPa)
1	160	96.88	40.278	7.576	7.576	0.025	40.27
2	160	104.18	43.313	6.94	6.94	0.021	43.301
3	160	98.98	41.151	7.752	7.752	0.071	40.719
4	160	116.1	48.269	7.747	7.747	0.017	48.26
5	160	115.83	48.157	5.155	5.155	0.008	48.14
6	160	111.18	46.223	5.9	5.9	0.008	46.207

Exclusion of a value from a set of parallel measurements is permitted by the standard only in the following cases:

- if the body breaks outside the marked working part;
- a tear occurs within 10 mm of the jaw.

Figure 14 shows the testing of samples from PVB material at 160 °C. It shows that all the tested samples broke within the working part. A temperature of 160 °C for the PVB material indicates a change in the relative elongation (elongation at peak) from 5.9% to 7.576%, compared to the previous values shown in the graphic.

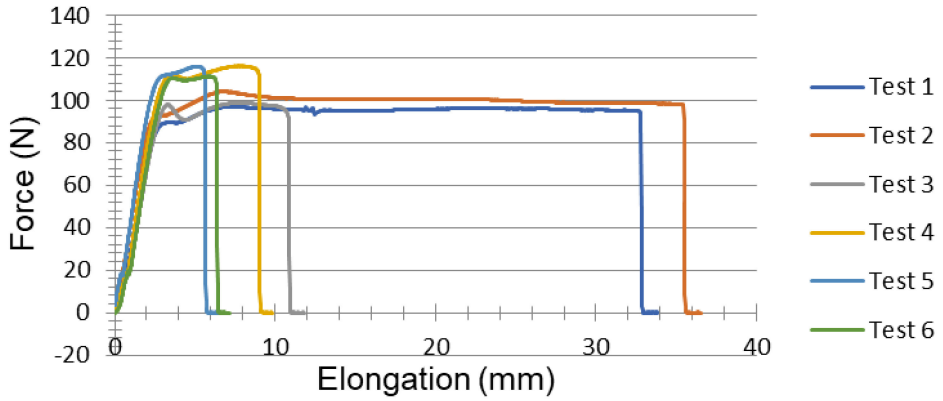


Figure 14. Graphic representation of a tensile test of a PVB filament with a diameter of 1.75 mm and a temperature of 160 °C.

The filament of PVB material at a temperature of 175 °C and the statistical processing of the tensile test are shown in the following table. At this temperature, the "Elongation at break" value for all six test samples ranged from 32.179% to 38.095% (Table 7).

Table 7. Statistical processing of tensile test (DIN EN ISO 527-1) data of PVB filament with a diameter of 1.75 mm and a temperature of 175 °C.

Test No	Temperature	Force Peak (N)	Elongation at Break (%)	Elong Peak (mm)	Strain Peak (%)	Tensile Stress (MPa)	Stress Yield (MPa)
1	175	83.4	34.674	5.552	5.552	0.183	34.657
2	175	80.14	33.318	3.353	3.353	0.146	33.314
3	175	77.4	32.179	4.879	4.879	0.017	32.009
4	175	78.6	32.678	5.525	5.525	0.582	32.67
5	175	86.81	36.091	7.905	7.905	0.021	36.079
6	175	91.63	38.095	3.212	3.212	0.033	38.075
7	175	88.08	36.619	5.31	5.31	0.008	36.615

The graphic display (Figure 15) of the tensile test draws attention to the proportional elongation of the tested material, which ranges from 3.212% to 7.905%. The force with which the test sample was stretched up to the moment of rupture tensile stress ranged from 0.033 MPa to 0.183 MPa.

Based on the tensile test data for PVB material with a diameter of 1.75 mm and a temperature range from 140 °C to 175 °C, we can conclude that the differences in the results of the tensile test are caused by the influence of external factors influencing [40] the course of the tensile test, as follows:

- frequency and load status of the tested recycled polyvinyl butyral (PVB);
- the magnitude of the mean stress when loading the polyvinyl butyral;
- state of tension of the recycled polyvinyl butyral;
- aggressiveness and temperature of the environment in which the measurement takes place;
- the shape tested from recycled polyvinyl butyral;
- test sample recycled polyvinyl butyral;
- the existence or assumption of cracks in materials (PVB recycled);
- surface properties of the recycled PVB;
- method of preparing the tested sample (use of material processing technology).

Internal factors influencing the course of the tensile test are as follows:

- chemical composition of the recycled PVB;

- structural changes in the recycled PVB;
- method of recycled PVB processing (use of material processing technology, as in the case of external factors).

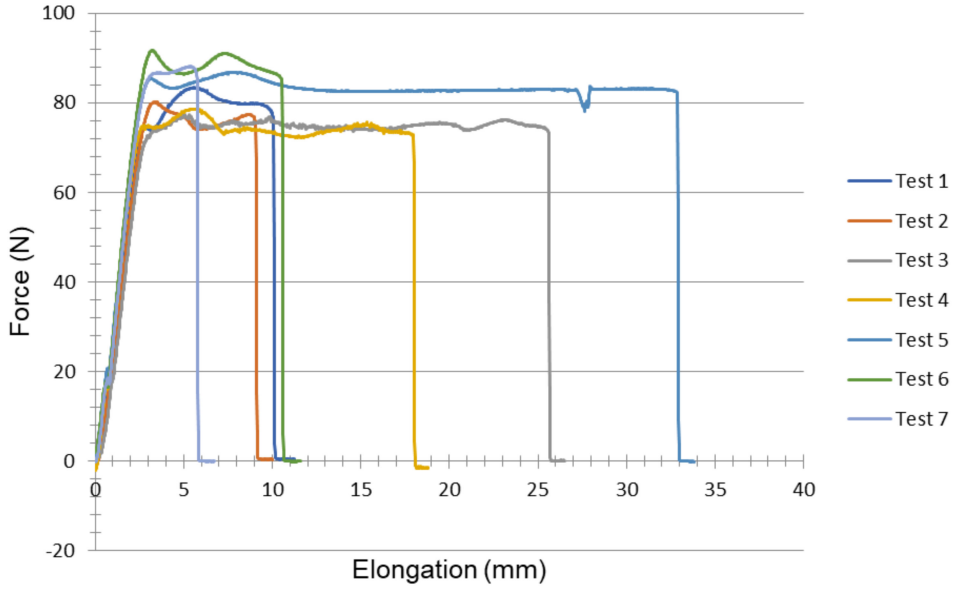


Figure 15. Graphic representation of a tensile test of a PVB filament with a diameter of 1.75 mm at 175 °C.

4. Conclusions

The presented research aims to underscore the current significant deficiencies in preparing filaments for FDM devices from recycled polyvinyl butyral and the absence of standards for producing and preparing fibers or samples for various mechanical tests. Through gradual changes and the optimization of the entire process, the goal is to create a plan to develop a unified and reproducible procedure for preparing recycled polyvinyl butyral. The advantages of the presented research are optimizing the process and eliminating the resulting shortcomings; a fiber made of PVB material with a diameter of 1.75 mm ($\pm$0.06 mm) was created, which can be used in most FDM devices. The length of the created fiber was approx. 20 m. These will subsequently be utilized for in-depth research into their mechanical properties that are directly related to the issues of FDM technology and the subsequent preparation of samples for the still absent mechanical properties of samples produced by layering thermoplastic material.

However, achieving this stable temperature in the conditions under which the device was placed is debatable. For that reason, we suggest the following possibilities for the further optimization of the process in the future:

- Create a stable environment or one with a constant temperature. Ideally, the location and design of the extrusion equipment should be changed;
- Using a different arrangement of cooling unit fans and increasing their performance or alternative cooling methods;
- Support of circular economy in the manufacturing process.

Author Contributions: Conceptualization, J.K.; methodology, J.K. and R.T.; software, J.K. and R.T.; validation, R.T. and M.M.; formal analysis, L.K.; investigation, J.K. and R.V.; resources, R.T. and R.V.; data curation, E.S. and M.M., writing—original draft preparation, J.K.; writing—review and editing, L.K.; visualization, R.T.; supervision, L.K.; project administration, L.K. and E.S.; funding acquisition, L.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by project VEGA 1/0268/22, which was granted by the Ministry of Education, Science, Research and Sport of the Slovak Republic.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article; further inquiries can be directed to the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

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