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Production of biomedical filament and mechanical testing of samples produced by FFF additive technology

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Abstract: This scientific study, which brings completely new results, is characteristically divided into chapters where the authors deal with the extrusion mechanism, filament production, 3D printing of samples, and mechanical testing of samples. This research deals with a current topic in the field of production and testing of filaments composed of biodegradable polymers based on custom made PLA/PHB material. The authors of this study managed to produce and test a new type of biomaterial in the form of a filament called PLA BIOPOLYMER 20. The individual components forming this new type of material are described in the detailed statistics of this scientific article. After optimizing the parameters during single-screw extrusion on a filament maker, where the extrusion temperatures were set to 180° and the subsequent additive manufacturing of samples using FFF technology was started, where "dogbone type 5A" samples were printed. The authors managed to optimize the parameters of additive manufacturing and achieve significant results, which are also represented by individual printed samples, intended for subsequent mechanical testing, specifically for tensile testing. Important testing of materials for mechanical tensile tests was carried out according to generally applicable standards STN EN ISO 527-2, on the Inspekt table 5kN device. By mechanical testing, the individual stresses of the samples were determined. The average stress was 5.28 MPa. The authors compared the values obtained with samples printed from the new type of PLA BIOPOLYMER 20 material with the tension obtained with samples printed from pure PLA filament without admixture of other components. These tests are intended to determine the future application of the given material. This article brings new knowledge in the given field.

1 Introduction

The extrusion process can also be defined as a production technology in which metal or plastic materials are pushed through a fixed cross-sectional profile to create a continuous strip of shaped product (filament). The extrusion process begins with the introduction of material in the form of granules, pellets or powders from hoppers into the extruder zone. The melting process then begins through the heat generated by the mechanical energy generated by the rotating screws and heaters located along the head. The molten ones are then pressed into a matrix, which structures the materials into a hard pipe material during the cooling process. A single-screw extruder is the most common type of extruder and offers relatively low investment costs for companies involved in extruding materials intended for biodegradable purposes. If higher production and higher performance are required, twin-screw extruders are used. The easiest way to increase the throughput of the extruder is to increase the speed of the screw. This easy solution usually results in poor melt quality caused by exceeding the melting capacity of the screw design and degradation caused by high melt temperature. Using a screw with a smaller diameter can offer several advantages for achieving higher throughput at a higher screw speed. One of the important advantages of a smaller diameter extruder is better heat transfer characteristics. Higher output at higher screw speed can be achieved by using a smaller diameter extruder. This offers better heat transfer characteristics [1-6]. A well-designed, developed screw design improves product quality and reduces the time needed to design and optimize the extrusion process, resulting in lower costs. Figure 1 shows a type of single-screw extruder. In twin-screw extruders, clogging of the material can be prevented, but in single-screw extruders, the material is retained much longer than in twin-screw extruders. A twin-screw extruder has about three times the material output. Theoretically, the material flow process can be divided into four sections: (a) extruder filling, (b) mass transport, (c) flow through the die, and (d) exit from the die and subsequent processing. The material processing time in the extruder is called the material distribution time. The residence time distribution is an important parameter for product quality. Most commercial extruders provide a choice of screws or interchangeable sections that change the configuration of the feed, transition and metering zones.

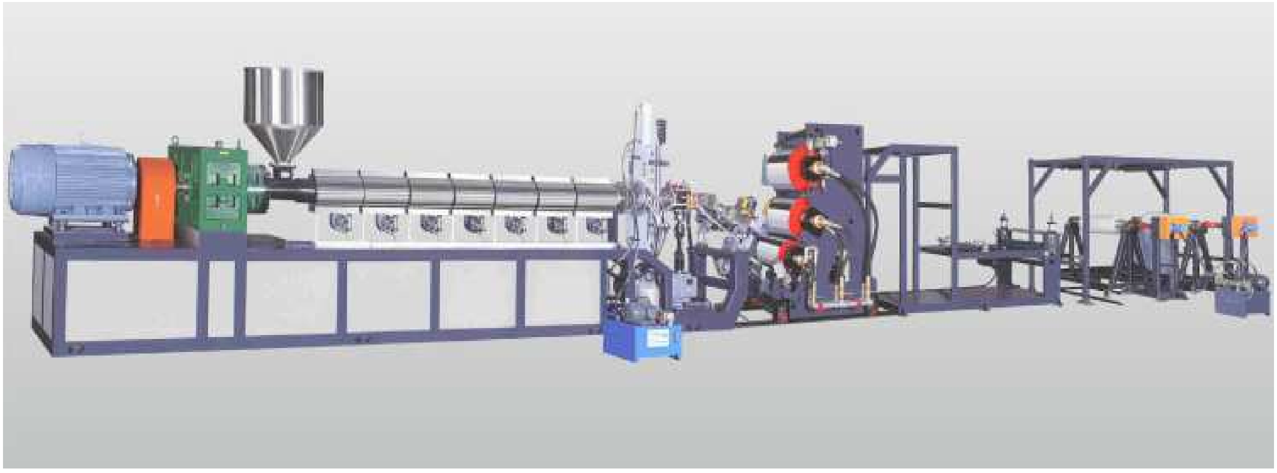

Figure 1 Single-thread industrial system for the production of filaments [1]

1.1 *Characteristics of materials*

Our filament consists of PLA/PHB polymers and added plasticizer with thermoplastic starch. The distribution and weight ratio of all materials is one to 25 percent. PLA material is easy to use and will ensure a relatively high-quality 3D model. It is biodegradable in nature and is made from corn starch. The thermal properties of PLA play a significant role in its performance during the 3D printing process. One of the key thermal properties of PLA is its glass transition temperature, which is the temperature at which the material changes from a hard, glassy state to a soft, rubbery state. For PLA it is usually around 60°C. Another important thermal property of PLA is its melting point. It is the temperature at which the material changes from a solid state to a liquid state. For PLA, the melting temperature is typically in the range of 130–180 °C. This relatively low melting point is one of the reasons why PLA is preferred in 3D printing. It allows easier extrusion through the printer nozzle and better control over the printing process. PLA is a thermoplastic polymer characterized by high mechanical resistance, suitable biocompatibility and bioresorbability. It is produced from renewable and non-toxic sources of raw materials. Lactic acid (LA) is converted to PLA by condensation polymerization. Due to the chiral nature of LA and the two asymmetric centers it has, it can form in a wide variety of forms and also has the following isomers: L, D and D, L isomers as well as the D isomer. Compared with other aliphatic polyesters, PLA has demonstrated many superior properties, such as high mechanical strength and modulus, biodegradability, biocompatibility, and easy processing. In Figure 2 we can see the pla structure [7-9].

PHB materials are mechanically stiff and brittle, with low thermal stability and a high degree of crystallinity. Many PHB plastics have properties that are similar to petroleum polymers - polypropylene (PP) and polyethylene (PE) [10]. Feedstocks for the production of PHB biopolymers include renewable and sustainable sources such as food waste. When exposed to designated active biological environments, these factors, combined with its biocompatibility and predisposition to biodegradation, make PHB a leading candidate as an alternative to synthetic polymers. Figure 3 shows us the structure of the PHB [11-14].

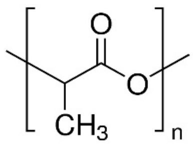

Figure 2 PLA structure [11]

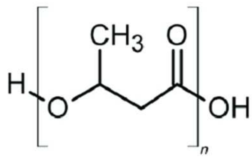

Figure 3 PHB structure [11]

2 Methodology of extrusion and 3D printing

2.1 *Filament extrusion*

Filament production is a multi-step process. We performed filament extrusion on a single-screw extruder from 3Devo. As an input material, we had medically certified pellets, which were composed, as mentioned above, from PLA/PHB materials with the addition of a plasticizer. The extrusion temperature was set to 180°C on all four extrusion bodies of the extruder. We set the screw rotation speed to 3.0 RPM. In Figure 4 we see the material flow. Figure 5 shows us the finally manufactured filament BIOPOLYMER 20.

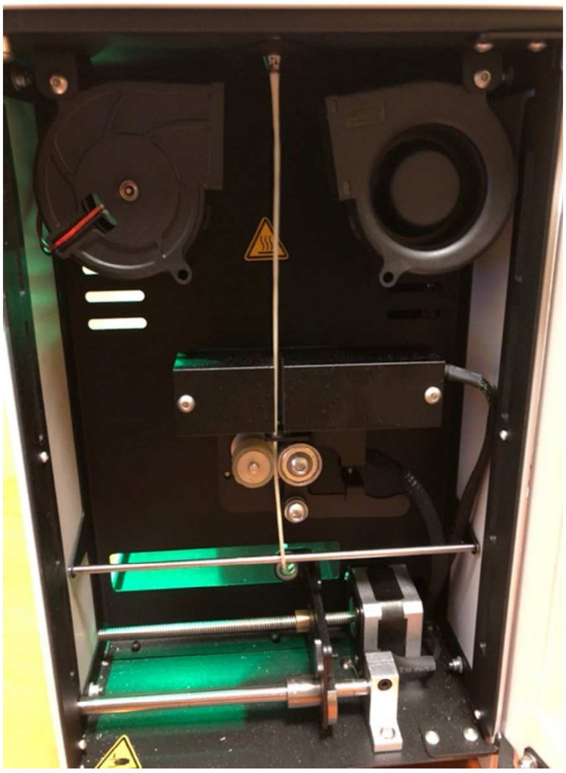
Figure 4 Material flow


Figure 5 Final filament BIOPOLYMER 20

2.2 3D printing of samples

Samples for mechanical testing were produced using additive FFF technology on a Trilab printer. These were dogbone 5A samples. We set the printing temperature to 175°C and the substrate temperature to 60°C. We set the print speed to 1200mm/s. The modeling of the samples was performed in the Symplify software (Figure 6). In total, more than 20 samples were printed (Figure 7).

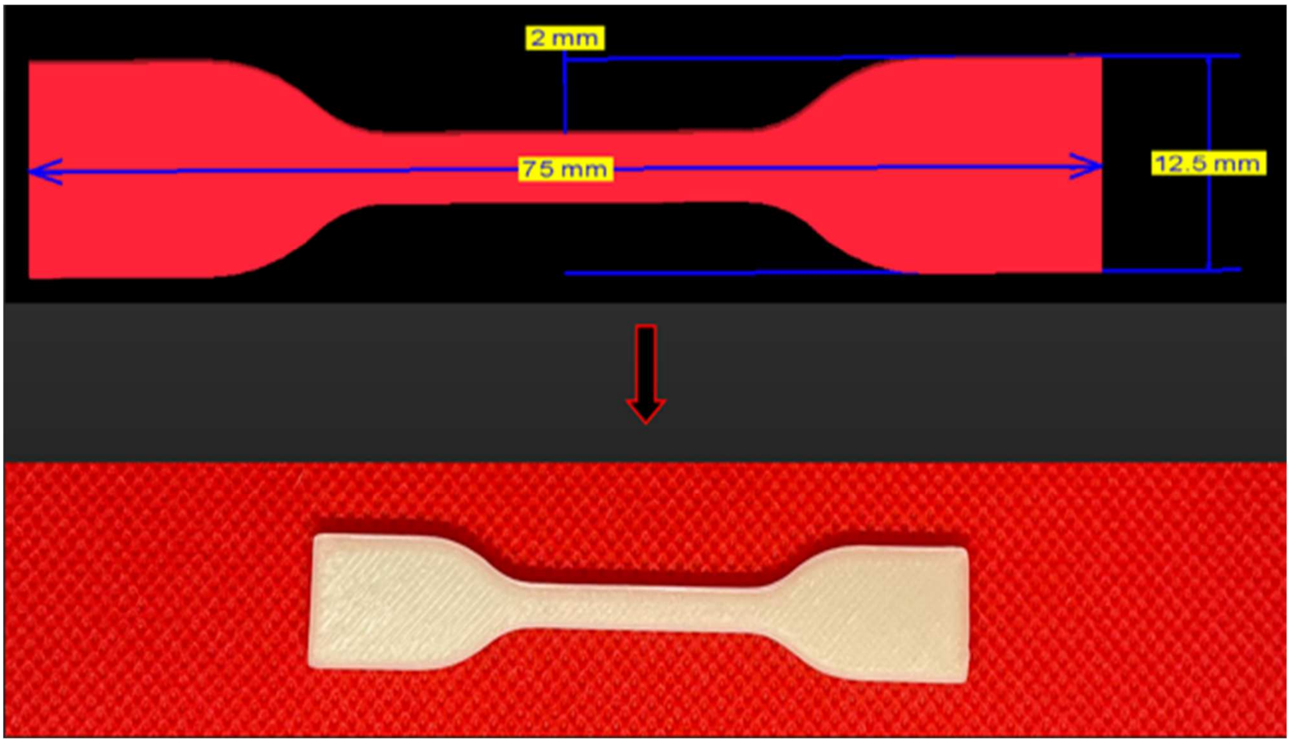

Figure 6 Type 5A dogbone sample model

Figure 7 Final printed samples using FFF technology

3 Mechanical testing characterisation

Important testing of materials for mechanical tensile tests was carried out according to generally applicable standards STN EN ISO 527-2, on the Inspekt table 5kN device. The load speed was set to 2 mm per minute. About 20 samples were mechanically tested, where Table 1 gives us the results of tension values with average of 5.28 MPa.

Table 1 Technical specifications

Tested sample	***PLA/PHB [MPa] Tension***
1	4.57
2	5.45
3	5.11
4	5.23
5	5.46
6	5.53
7	5.11
8	5.12
9	5.09
10	5.02
11	4.90
12	5.26
13	5.39
14	5.61
15	5.18
16	5.45
17	5.25
18	5.66
19	5.66
20	4.99
21	5.22
22	5.18
Average	**5.28**

4 Conclusion

The results in this study significantly contribute to the field of material production through extrusion on a filament maker device, 3D printing using FFF technology, and determine the future of use by mechanically determining the strength of the material. Printing biodegradable materials provides precision and flexibility in creating complex structures. Material based on PLA and PHB is biodegradable to some extent. This is what motivated us to implement and solve the problem of this topic.

The facts we have obtained can be of great benefit to the practice in the future. The biodegradable material created has the potential to influence the medical industry in the future. For example, for access to more personalized treatment procedures, or also the possible use of material in the human body for the regeneration of bone tissue. These findings open the door for further possible research into the evaluation of mechanical, biological and biocompatible properties.

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