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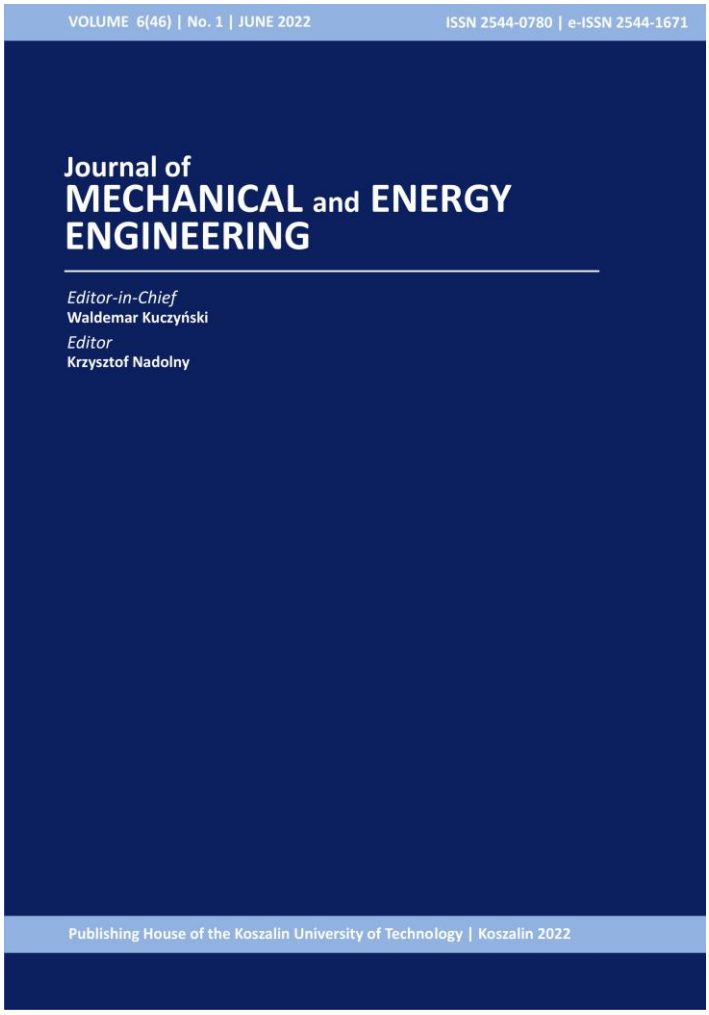

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MATERIAL INVESTIGATIONS ON POLY(ETHYLENE GLYCOL) DIACRYLATE-BASED HYDROGELS FOR ADDITIVE MANUFACTURING CONSIDERING DIFFERENT MOLECULAR WEIGHTS

Sven. F. KLIMASCHEWSKI[1*], Janine KÜPPERBUSCH[2], Annika KUNZE[2], Mark VEHSE[2]

[1*] Faculty of Mechanical Engineering, University of Applied Sciences Stralsund, Zur Schwedenschanze 15, Stralsund, 18435, Germany, sven.klimaschewski@hochschule-stralsund.de
[2] Faculty of Mechanical Engineering, University of Applied Sciences Stralsund, Zur Schwedenschanze 15, Stralsund, 18435, Germany

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Abstract: The aim of this case study is to generate several poly(ethylene glycol) diacrylate-based hydrogels using additive manufacturing processes. The interest here is in determining different material properties. The test specimens are produced using a commercial stereolithography system. For this purpose, three formulations are prepared. The basis in each case is PEGDA with average molecular weights of 700 M_n, 575 M_n and 250 M_n. A photoinitiator and a UV absorber are added to ensure spatial and temporal cross-linking. Furthermore, the formulations are tested for their material properties according to ISO standards using tensile, compression and hardness tests. An equivalence can be found in the tensile and compression tests. The results with the molecular weights of 700 M_n and 575 M_n show values close to each other. However, the results of the material tests with the molecular weight of 250 M_n are ten times higher. The Shore A hardness values also correlate with the previous tests. These results between molecular weight and material property are particularly striking. A novel aspect of this method could be that the properties determined of these tailor-made high-performance polymers can be applied to different areas of application in an organism.

Keywords: additive manufacturing, stereolithography, PEGDA, molecular weights, material properties, biocompatible hydrogel

1. INTRODUCTION

In medicine, 3D printing can be used to develop and manufacture individual products adapted to the human organism, such as for surgical applications [1], orthosis [2], drug delivery systems [3] and implants [4]. For medical applications, it is essential that the materials used for this purpose have defined mechanical properties as well as biocompatibility [5, 6]. One of these materials are polymers. These are already found in many medical fields and can serve as carriers of drugs or life processes, among other things [3, 7]. Due to this, research is being conducted on tailor-made high-performance polymers as they have a broad property profile and they could thus fulfill many requirements [8-12].

In this regard, great interest lies in the material known as poly(ethylene glycol) diacrylate (PEGDA). Due to its acrylate groups, the polymer is crosslinkable and therefore capable of forming a three-dimensional (3D) network. In combination with a photoinitiator, polymerization is particularly versatile for tissue engineering [13]. Thus, spatially and temporally defined and highly complex structures can be 3D-printed from PEGDA to form 3D objects [14, 15]. Furthermore, this material offers a wide range of different molecular weights that influence the material properties, such as stiffness, strength, swelling and diffusivity [15-17].

The present study addresses the cited results by investigating the dependencies of different molecular weights of PEGDA with its material properties. The following questions are considered: the mixing ratio of PEGDA 700 M_n from previous research is verified [14] and two new formulations with PEGDA 575 M_n and

250 M_n are developed for use on a commercial stereolithography (SLA) system. It is important to adjust the mixing ratio of the PEGDA, the photoinitiator and the UV absorber to achieve a good print quality. In addition, the mechanical properties will be investigated by producing test specimens according to ISO standards and testing them for their material properties in tension, compression and hardness. From this, dependencies of molecular weight and material properties can be derived and thus possible areas of application of the polymers can be identified.

2. MATERIALS AND METHODS

2.1. Stereolithography (SLA)

The 3D planned objects will be produced with the commercial (SLA) *Nobel 1.0* from *XYZ-Printing, Inc., Taiwan*. The SLA operates with a laser light wavelength of 405 nm. A laser intensity of 55 mW and a layer height of 0.50 mm are selected for generating the 3D objects, as this corresponds to the standard parameters of the SLA for transparent liquid resins.

To create the design specimens, the SLA must first be reduced in size. This decreases the chamber volume from approx. 90 ml to 10 ml. Thus, a considerable amounts of material for the required specimens can be saved. Figure 1 (A & B) shows the modification of the system. The connector (1) is replaced, this serves to hold the platform built (2). Furthermore, the chamber (3) is replaced in order to reduce the filling volume considerably. Furthermore, the chamber (3) is replaced and fixed by a 3D-printed special unit (4). A tension lock (5) is used to fix the chamber. A tension lock (5) is used for fixation. A detailed description with schematic drawings of the printer used can be found in the previous article [18].

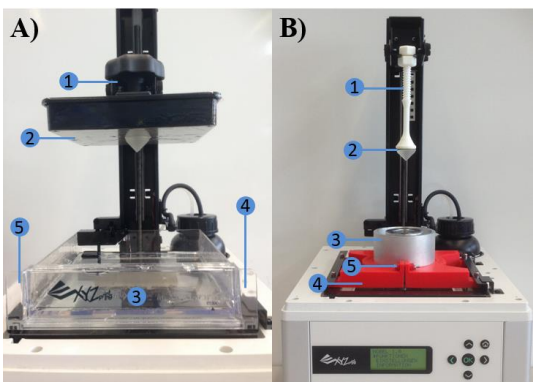

Fig. 1. Modification of the SLA for small test specimens; A) original illustration of the SLA; B) SLA with reduced building space [18]

Finding the appropriate formulation initially requires only small sample quantities. It is determined that the amount of liquid resin for exploring the specimens should be between 3 to 5 ml. Therefore, the SLA is initially modified for these investigations (for more details, see [12, 16]).

To avoid falsification, all 3D objects are generated individually. This means that if several 3D objects were generated in one manufacturing process, it would not be possible to ensure the focus of the laser beam.

2.2. Post-Processing

In post-processing, the specimens are cleaned with *2-propanol* (≥99.5%, Ph.Eur., purest) from *Carl Roth GmbH + Co. KG, Germany*, for 9 to 15 seconds so that no polymer resin remains on the 3D object. Subsequently, they are post-cured for about 15 to 20 minutes in a UV chamber (*Form Cure, 3Dmensionals / PONTIALIS GmbH & Co. KG, Germany*) with a wavelength of 405 nm. The temperature of the curing chamber is 0°C.

2.3. Synthesis of the resin

Three formulations are mixed, each with poly(ethylene glycol) diacrylate (PEGDA) with average molecular weights of 700 M_n, 575 M_n and 250 M_n from *Sigma Aldrich, Inc., USA*. Each of these is blended with a portion of the photoinitiator (PI) bis(2,4,6-trimethylbenzoyl)-phenyl- phosphine oxide / 2-hydroxy-2-methyl-1-phenyl-propane-1-one (Omnirad 2022) from *IGM Resins B.V.* and a portion of the ultraviolet radiation absorber (UV-AB) (2,2'-dihydroxy-4,4'-dimethoxybenzophenone) from *TCI Deutschland GmbH*. To determine the best mixing ratio on the SLA, 5 ml samples are first mixed at 40°C for 12 h and 1000 rpm. Then, the formulations are allowed to rest for one hour at a room temperature of 23°C. After a successful adjustment of the resin on the SLA, three formulations with 270 ml are carried out. Table 1 shows the mixing ratios used. Due to the higher mass, the stirring time is extended to 168 h, the temperature is 40°C and 900 rpm. The formulations rest for 5 h.

Tab. 1. Mixing ratios of the three formulations

Average molecular weight	PI, g/ml	UV-AB, g/ml
PEGDA 700 M_n	0.00500	0.006
PEGDA 575 M_n	0.00850	0.007
PEGDA 250 M_n	0.01078	0.004

2.4. Design of the test specimens

For the determination of the suitable formulations in interaction with the SLA, a stepped double cone is constructed with the program *SolidWorks 2017* of the company *Dassault Systèmes SolidWorks Corp., France*. Figure 2 shows the constructed stepped double cone in a 90° view A), in an isometric view B) and the conditions of the step description C). These are the visual requirements for the printed image. It is a reference of the criteria to be investigated for all the three formulations. These are polymerization, the layer

generation, the shape, the design, dimensions, contours, defects and angles.

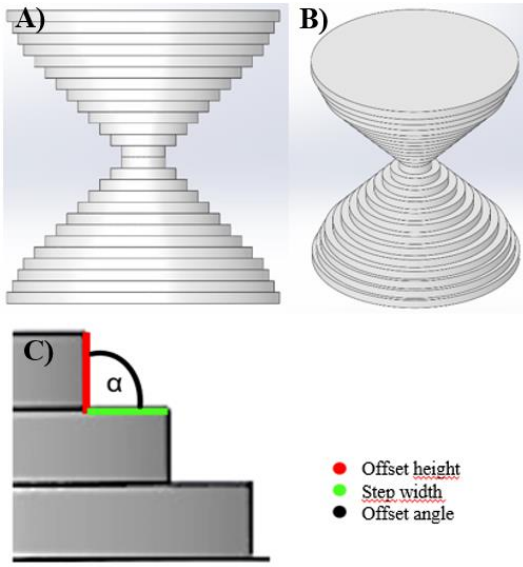

Fig. 2. Stepped double cone created from CAD data in the views, 90° A) and 45° B), terms of the step description C)

2.5. Procedure for material characterization

To determine the material properties, test specimens are constructed according to ISO standards and tested for tension, compression and hardness. The influence of the molecular weights on the material properties is determined.

For the tensile specimens, the test specimen type 1BA is designed according to the EN ISO 527-2 standard, as shown in the technical drawing in Figure 3 A). At least ten specimens are generated B) and fixed between two clamps according to the standard and pulled to failure at a constant test speed of 1mm/min. The tensile and compressive properties are determined using the *TIRAtest 2705* testing machine from *TIRA Maschinenbau GmbH, Germany*. This is equipped with a load cell of 5 kN. The load cell type KAP-S from the company *A.S.T. Angewandte System Technik GmbH, Germany* was used.

The SLA software does not offer an alignment of the layer trajectories, these layer trajectories follow the shortest path layer by layer. Figure 4 shows this alignment for the tensile specimen.

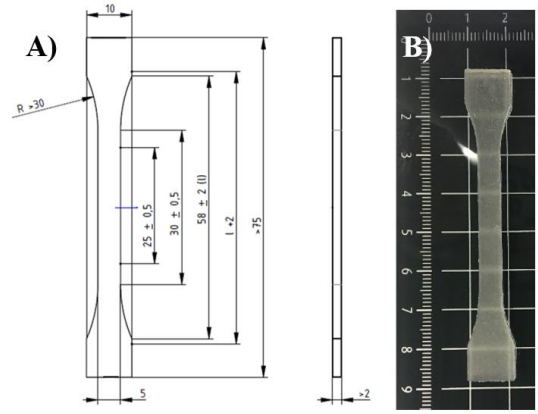

Fig. 3. Technical drawings of the tensile specimen A), test specimen generated according to ISO standard B)

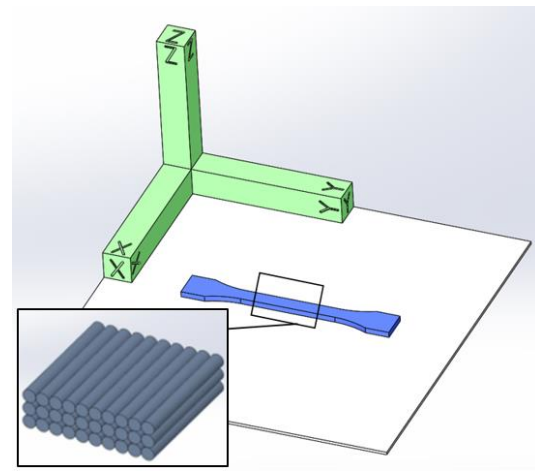

Fig. 4. Alignment of the layers during 3D printing on the SLA

To determine the compressive properties, test specimen type A is constructed in accordance with standard EN ISO 604. This is shown in the technical drawing A) in Figure 5. Ten of each are subjected to the pressure procedure. A force is applied to the test specimen until it is destroyed, at a test speed of 1mm/min. Part B) shows the 3D-printed test specimen. For the determination of the compressive properties of PEGDA 250 M_n, the testing machine *TIRAtest 28100 E58* with a 100 kN load cell must be used, as higher compressive strengths σ_M occurred and the load cell of 5 kN is not sufficient. The testing machine is also from *TIRA Maschinenbau GmbH, Germany*.

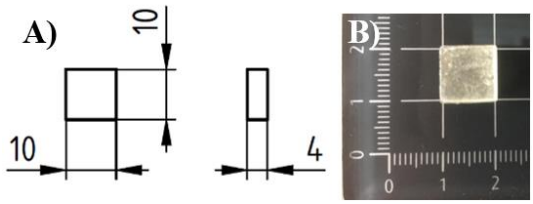

Fig. 5. Technical drawings of the compression specimen A), test specimen generated according to ISO standard B)

The hardness is determined by the Shore A method according to DIN ISO 7619-1. Figure 6 shows the technical drawing of the test specimens A). For each formulation, ten penetration tests are run on a 50×50×7 mm test specimen B) to determine its resistance behaviour. The measuring points must have a distance of 5 mm from each other and 13 mm from the edge. The hardness testing machine used is from the company *ZORN INSTRUMENTS GmbH & Co. KG, Germany*.

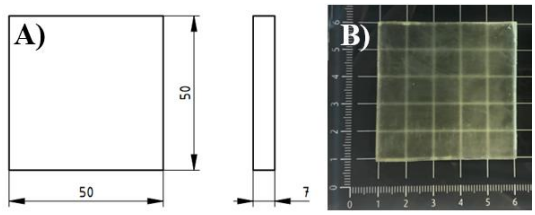

Fig. 6. Technical drawings of the hardness specimen A), test specimen generated according to ISO standard B)

3. RESULTS AND DISCUSSION

In this chapter, an assessment of the macroscopic and microscopic evaluation of the three formulations will be made on the basis of the stepped double cones. Furthermore, an assessment of the material properties will take place. The room temperature during the tests is 22 ± 1.50 °C and the humidity is 49.90 ± 2 %.

3.1. Evaluation stepped double cone

The test specimens will be used to explore the best mixing ratios (PI and UV-AB) on the SLA. Figure 7 shows an example in the upper image 1) where the mixing ratio needs to be further adjusted. The hydrogel was not sufficiently polymerized, resulting in voids (A) in close-up 2). Also, the dimensions defined are not achieved. The diameter of the uppermost step (B) and the step in the smallest cross-section (C) did not meet the specifications. The circular geometry of the steps is not achieved everywhere (example (D)). A step offset including the defined angles is not achieved (example (E)).

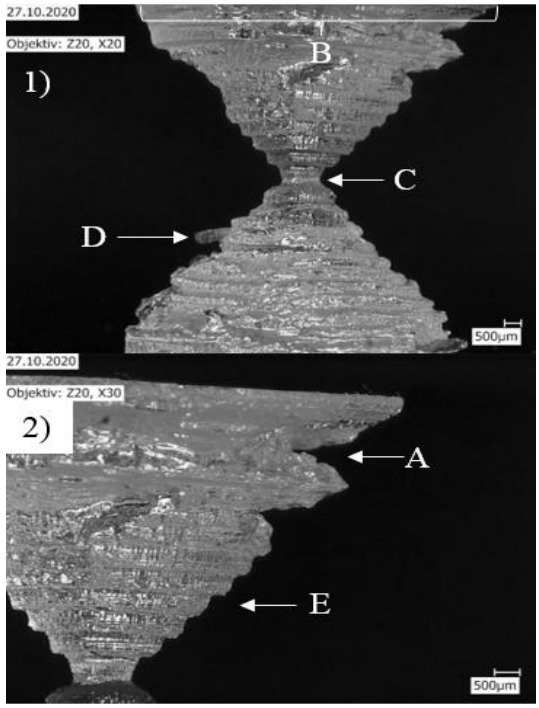

Fig. 7. Negative example of a printed stepped double cone in 90° view 1) and in close-up 2)

The reason for the poor result is the insufficient proportion of PI in the formulation. It follows that not enough radicals are available, and spatial and temporal crosslinking was not possible.

PEGDA 700 M_n

The formulation PEGDA with an average molecular weight of 700 M_n showed the best printing result in the former studies and it is repeated to verify it for the further steps. In Figure 8, the upper image 1) shows the stepped double cone of the repeated formulation. In the upper cone, all the steps are present (A). The step contours are slightly washed out (B), indicating that the cone came into contact with light during extraction from the space built. Due to the surface tension, this may have led to post-crosslinking of the liquid polymer residues in the corners. In the lower image, right side 3) shows that at the points where the cone was protected from the influence of light, the individual layers are clearly visible (C). In the close-up, the lower image left 2), the steps of the upper cone have a relatively good offset angle of 90° (D). Furthermore, the step at the smallest cross-section is well visible, but post-crosslinking occurred here, as well (E). The lower cone, in image 1) shows insufficient quality. The steps are only vaguely visible (F). It is almost the same appearance as the cone from the previous results in the former studies [17]. Based on this, the formulation is verified and used for the experiments.

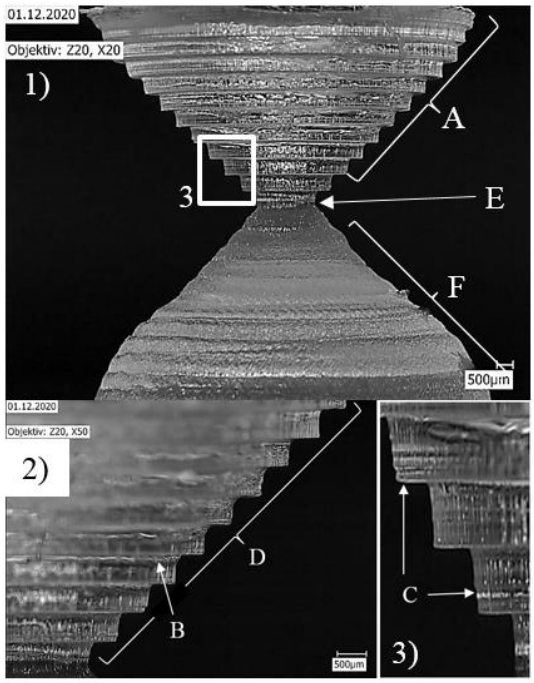

Fig. 8. Stepped double cone with PEGDA 700 M_n in 90° view 1), close-up 2) and further close-up from 3)

PEGDA 575 M_n

Figure 9 shows in image 1) the stepped double cone of PEGDA with an average molecular weight of 575 M_n. All the steps in the upper cone can be seen, even the step at the smallest cross-section is well formed (A). The individual layered steps are clearly visible in the close-up 2) (B). The steps in the upper cone have good contours and an offset angle of approximately 90° (C). In some steps, there is polymerized residual resin (D). Since this effect is not seen in all the stages, there is an assumption that this is due to surface tension. The claim is further supported by the fact that there are no layered or clear edges, but rather fillets are visible in the offset angles. Thus, this effect can be attributed to post-processing. The reason for this may be that the cleaning was not thorough. In the lower cone, the individual beginnings of the steps are clearly visible. The result obtained provides a good appearance and can be used in the further course of the investigations.

PEGDA 250 M_n

Regarding the polymerization process, it must be mentioned that PEGDA 250 M_n requires special safety measures. A test in advance showed that there is a significant increase in temperature when irradiated with UV light. Smoke is generated and parts splinter off from the polymerized material. One reason for this could be that shorter polymer chain lengths provide more reactants, which trigger more reactions during the polymerization process. Nevertheless, a generation of the stepped double cone takes place. For these double cones, it was found that the proportion of PI must be increased and the UV-AB decreased. It can be seen in Figure 10 in image 1) that all the stages of the upper cone are present. The step in the smallest cross section can be seen well and it shows a good transition to the smallest step of the lower cone (A). In the lower cone, the step contours can be seen suggestively (B). Furthermore, no defects or material breakouts are visible. Moreover, the individual layers are visible, and an offset angle of about 90° is also achieved. In the close-up image 2), the individual layer paths can be seen with a 30x magnification (example C). However, there is again an adhesion of polymerized residual resin in the offset angles of the steps (example D). The formulation is suitable for further tests.

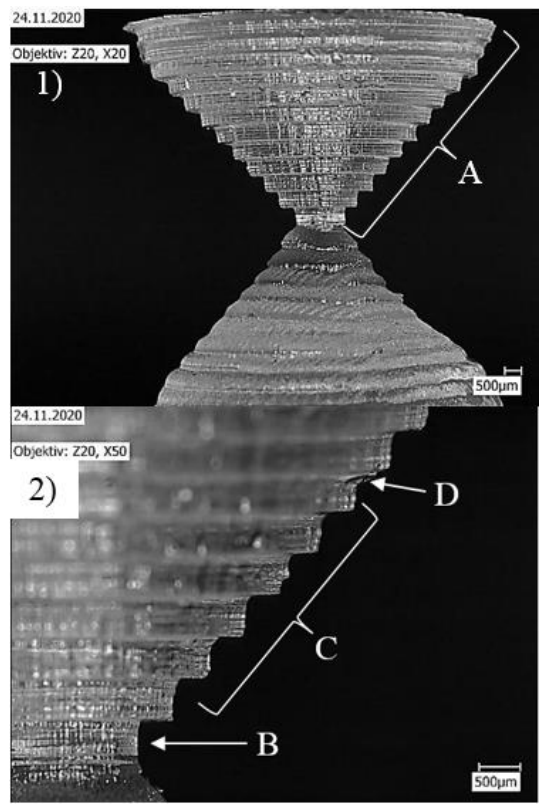

Fig. 9. Stepped double cone with PEGDA 575 M_n in 90° view 1), close-up 2)

3.2. Investigations of the material properties

Tensile test EN ISO 527-2

The stress-displacement plots in Figure 11 show the averaged curves of the formulations. PEGDA 700 does not exhibit any yield stress (0.00 MPa). In contrast, the PEGDA 575 formulation exhibits an insertion stress of 0.95 ± 1.10 MPa as determined by the testing machine. This may have been due to numerical calculations by the software of the tensile machine or to slippage of the test specimens from the restraint. The PEGDA 250 formulation shows a stronger or stiffer behavior than PEGDA 700 and PEGDA 575, with the value determined by the testing machine increasing to the

yield stress of 19.00 ± 6.27 MPa. For tensile strength, PEGDA 700 reaches a value of 1.90 ± 0.30 MPa. For PEGDA 575, the value drops to 1.68 ± 0.65 MPa.

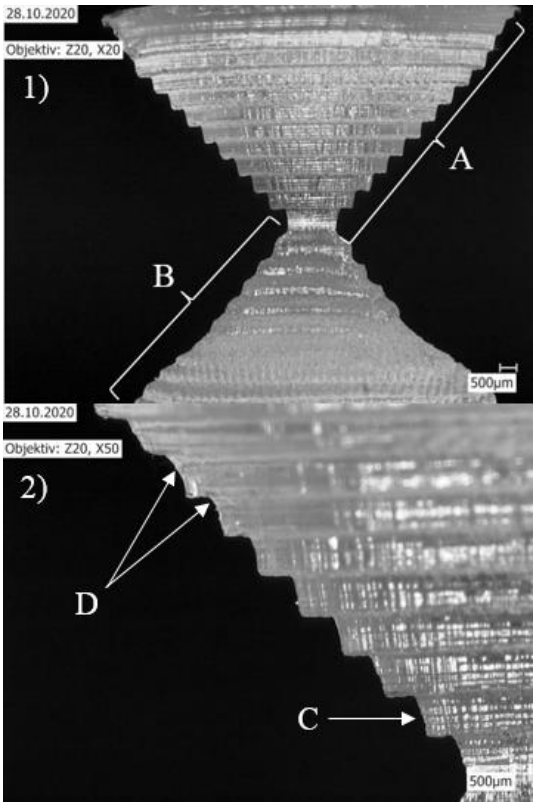

Fig. 10. Stepped double cone with PEGDA 250 M_n in 90° view 1), close-up 2)

For the PEGDA 250, the value of tensile strength increases significantly, which is 20.45 ± 3.31 MPa. Table 2 shows an overview of the values determined. The PEGDA 700 and 575 show a significant brittle fracture behavior in the course of the curves as well as on the fracture surfaces. In addition to the brittle fracture, the course of the curve of PEGDA 250 also shows an elastic-plastic behavior, which indicates a certain degree of toughness.

Tab. 2. Results of the tensile test

	Yield stress, MPa	Tensile strength, MPa
PEGDA 700 M_n	0.00 ± 0.00	1.90 ± 0.30
PEGDA 575 M_n	0.95 ± 1.10	1.68 ± 0.65
PEGDA 250 M_n	19.00 ± 6.27	20.45 ± 3.31

Compression test: EN ISO 604 Standard

The force-displacement curves in Figure 12 show a comparison of the averaged curves with the different molecular weights of the three formulations. PEGDA 700 again shows a brittle fracture behavior, the average compression strength is 20.92 ± 2.65 [MPa]. For PEGDA 575, the compression strength decreases and it reaches a value of 15.67 ± 2.03 [MPa]. The brittle fracture behavior also occurred with this formulation. In the compression test with PEGDA 250, the compression strength increases significantly, reaching a value of 233.55 ± 19.15 [MPa]. The material exhibits brittle fracture but the results show a ductile-elastic-plastic behavior, which indicates a yield stress. Table 3 shows an overview of the values determined.

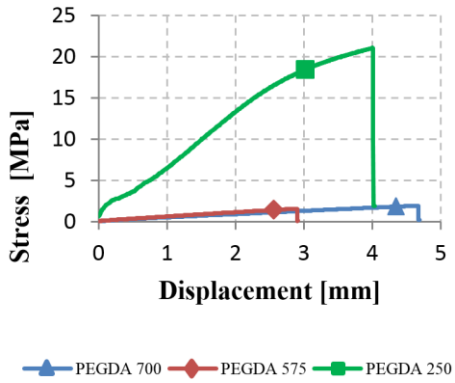

Fig. 11. Stress-displacement curves of PEGDA 700 M_n, PEGDA 575 M_n and PEGDA 250 M_n

Tab. 3. Results of the compression tests

	Compression strength, MPa
PEGDA 700 M_n	20.92 ± 2.65
PEGDA 575 M_n	15.67 ± 2.03
PEGDA 250 M_n	233.55 ± 19.15

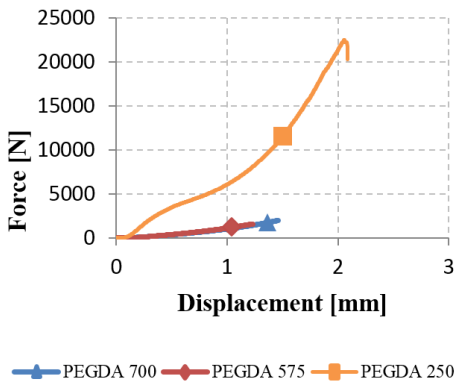

Fig. 12. Force-displacement curves of PEGDA 700 M_n, PEGDA 575 M_n and PEGDA 250 M_n

Hardness test DIN ISO 7619-1

The measurement by Shore A hardness shows that PEGDA 700 has a mean value of 70.90 and a deviation of ± 0.94. PEGDA 575 has a slightly lower hardness value of 66.20 ± 0.75. PEGDA 250 shows an increase

to Shore A hardness of 76.50 ±0.81. The hardness values of the formulations are compared in Figure 13 for visualization.

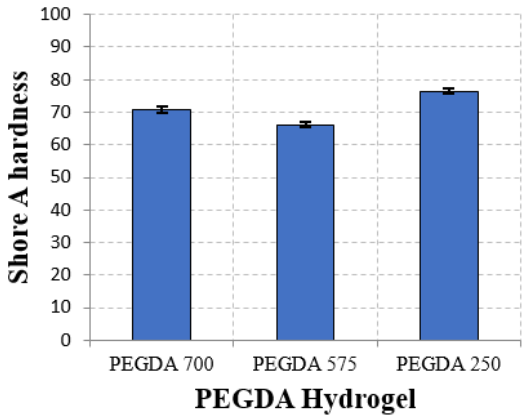

Fig. 13. Comparison of the hardness values of formulations: PEGDA 700 M_n, PEGDA 575 M_n and PEGDA 250 M_n

Again, PEGDA 700 and PEGDA 575 show a similar behavior based on similar values. PEGDA 250, on the other hand, repeatedly shows a higher value. The evaluation of the Shore A hardness measurements clearly shows that the values correlate with the previous material characterization tests.

4. CONCLUSIONS AND OUTLOOK

The aim of this study was to produce hydrogels based on poly(ethylene glycol) diacrylate (PEGDA) with three different molecular weights of 700 M_n, 575 M_n and 250 M_n. Subsequently, the mechanical behaviour of the samples printed was compared in order to clarify how they later could be used, for example, as implant material.

First of all, it should be mentioned that it was successfully proven that it is possible to create chemical low-molecular compounds. These could be spatially and temporally cross-linked by means of photoinitiation and highly complex geometries could be printed on a stereolithography machine.

Secondly, the static material tests, using tensile, compression and hardness tests according to ISO standards, demonstrated different properties depending on the molecular weights. These tests took place without swelling absorption. The tensile tests demonstrated that PEGDA 700 and PEGDA 575 exhibit a brittle fracture behaviour. The results of the yield stress and tensile strength of these two materials did not show any significant differences between each other. In the compression tests, the behaviour of the formulations with PEGDA 700 and 575 reflected the results of the tensile tests. Again, PEGDA 700 and 575 showed brittle fracture behaviours. The results of the Shore A hardness tests correlated with the previous investigations, too. The samples cured with PEGDA 250 showed a significantly different material behaviour compared to the other two formulations. Thus, the material properties in terms of tensile, compressive and hardness behaviour improved considerably. A slightly tough-elastic behaviour was accompanied by a significantly increased force resistance, e.g. in tensile and compression tests by a factor of 10 times higher.

With regard to potential medical applications in the human body, such as tissue engineering, the aforementioned differences have a significant impact. Due to the ability of PEGDA to support cell cultures, it has in principle an enormous potential for biomedicine, taking into account, depending on the application, the material properties identified here. For example, a custom scaffold can be fabricated to serve as a support for 3D cell culture and tissue engineering [19]. This raises the prospect of a wide range of in vivo applications in medicine, such as reconstructive surgery or cartilage replacement. It would thus be possible to adapt a PEGDA-based hydrogel to a selected tissue type in the human body based on its specifically selected material properties. In detail: for example, the ear cartilage or the nasal septum can be imitated due to the difference in hardness or an adjustable bending behaviour. In addition, the additive manufacturing process enables an individual geometric adaptation to the organism. In summary, the field of the applications of PEGDA-based hydrogels becomes more clearly definable and adaptable to the target application.

Finally, the investigations have shown that further test procedures should be carried out to obtain more detailed results on the material properties. Further investigations should include long-term loading, dynamic and cyclic material testing. Based on the results, all the formulations should be subjected to differential scanning calorimetry (DSC) to determine melting (T_{mS}) and glass transition (T_{gS}) temperatures, crystallinities and enthalpy of fusion (ΔH_{mS}). In addition, gel permeation chromatography (GPC) testing is recommended. The elongation at yield and elongation at break as well as the Young's modulus should be part of the next study. In this way, further necessary characteristic values are determined which, from the perspective of the materials science, provide more information about the material's behaviour for tissue engineering.

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would also like to thank Normen Fuchs for his support in interpreting the data.

Nomenclature

Symbols

T_{mS} – Determine melting
T_{gS} – Glass transition temperatures
ΔH_{mS} – Crystallinities and enthalpy of fusion

Acronyms

AM – Additive Manufacturing
CAD – Computer aided design
DSC – Differential scanning calorimetry
GPC – Gel permeation chromatography
ISO – International Organization for Standardization
M_n – Molecular weight
PEGDA – Poly(ethylene glycol) diacrylate
PI – Photoinitiator
rpm – rounds per minute
SLA – Stereolithography
UV-AB – Ultraviolet radiation absorber

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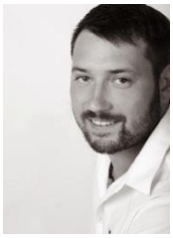
Biographical notes



Sven Frank Klimaschewski received his B.Eng. degree in Mechanical Engineering in 2019. He is an undergraduate assistant at the Lab of Engineering Design and Product Development at HOST Hochschule Stralsund, Germany. He is responsible for PEGDA based research

Annika Kunze received her B.Arts. degree in Business Administration in 2020. As part of the master's program in industrial engineering, she completed an internship in the laboratory for "Digital Product Development & Additive Manufacturing".

Janine Küpperbusch received her B.Arts. degree in Business Administration in 2020. As part of the master's program in industrial engineering, she completed an internship in the laboratory for "Digital Product Development & Additive Manufacturing".

Mark Vehse is a full professor for Engineering Design and Product Development at HOST Hochschule Stralsund, Germany and also the group leader of the two groups *Digital Product Development & Additive Manufacturing* and *Digital Product Development & Autonomous Robotics*.