journal of the mechanical behavior of biomedical materials 119 (2021) 104494

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: http://www.elsevier.com/locate/jmbbm

The effect of time on mechanical properties of biocompatible photopolymer resins used for fabrication of clear dental aligners

Aleksa Milovanović [a,*], Aleksandar Sedmak [b], Zorana Golubović [b], Ksenija Zelić Mihajlović [c], Aleksandar Žurkić [d], Isaak Trajković [a], Miloš Milošević [a]

[a] University of Belgrade, Innovation Center of the Faculty of Mechanical Engineering, Belgrade, Serbia
[b] University of Belgrade, Faculty of Mechanical Engineering, Belgrade, Serbia
[c] University of Belgrade, School of Medicine, Institute of Anatomy, Laboratory for Anthropology Belgrade, Serbia
[d] Shimadzu Corp. (Kyoto, Japan), Branch Belgrade, Serbia

ARTICLE INFO

Keywords:
Time Effect
Additive manufacturing
Biocompatible polymers
Dental aligners
Mechanical properties.

ABSTRACT

Clear dental aligners are used for treating orthodontic anomalies (misaligned teeth, inappropriate contact between upper and lower teeth etc.), minor irregularities and bruxism. Using additive manufacturing technologies clear dental aligners are made of biocompatible photopolymer, using a vat photopolymerization technology. One of problems in application is the change of aligner material properties after production, including strength and elongation at failure. This can cause different sequence of tooth displacement which will not correspond to the planned therapy. In this paper three types of material testing are conducted i.e., tensile, compressive and three-point bending testing on specimens of 1 (24 h), 3 (72 h), 5 (120 h) and 7 (168 h) days old. Mechanical properties, such as tensile, compressive and flexural strength and strain at failure are monitored in order to show the effect of time on biocompatible photopolymer resin.

1. Introduction

Development of 3D printing technology in recent years expanded and advanced significantly. Because of the fact that 3D technologies are commercially available and offer broad range of possibilities, their applications in medicine and dentistry provide good solutions in production and prototyping. The 3D printing offers advantages in engineering process when comparing to conventional techniques in dental every-day practice, for example subtractive computer numeric controlled methods.

Generally, 3D printing represents an additive manufacturing process where solid 3D objects are created by continuous adding of material in layer-by-layer manner. Till today, 3D printing was mostly used for rapid prototyping purposes, but nowadays this technology has high perspective in functional part manufacturing field (ASTM F2792-12a, 2013).

Several 3D printing mechanisms, such as fused deposition modelling (FDM), stereolithography (SLA), selective laser sintering (SLS), inkjet-based system, power bed fusion and digital light processing are used for creating and printing 3D objects, as well as for rapid prototyping. Each method offers some advantages depending on the type of products which should be created. For the research presented here, the SLA printer, Formlabs Form 2 (Formlabs, Sommerville, MA, USA) was used to produce specimens.

Stereolithography is important tool in dental practice because it offers exceptional dimensional accuracy which is of great significance in dentistry. The SLA printing works as deposition of material in layer-by-layer manner, using concentrated beam of UV light to polymerize and solidify liquid photopolymer resin placed in a vat, and create 3D complex shapes (Wang et al., 2017; Ligon et al., 2017). The 3D part is produced by projecting the 2D slice on the surface of resin for determined period of exposure, build platform is then lowered for determined thickness, and the process is repeated until 3D structure is completed (Uzcategui et al., 2018). The SLA uses thermosetting acrylates, or epoxy photopolymers in their liquid state, or polymers that become solids after vat photo-curing. Dimensional inaccuracy in this process can occur when more layers are used to build a model, because if error appears at any layer, it will influence following layers, and notable dimensional mismatch will be created.

Mechanical strength of SLA produced parts is limited due to viscosity restrictions of resins. Also, there is the advantage of faster curing of acrylate monomers in comparison with analogous methacrylate's,

* Corresponding author.
E-mail address: amilovanovic@mas.bg.ac.rs (A. Milovanović).

https://doi.org/10.1016/j.jmbbm.2021.104494
Received 30 October 2020; Received in revised form 25 February 2021; Accepted 22 March 2021
Available online 26 March 2021
1751-6161/

which are anyhow giving the higher strength of polymers and better biocompatibility than acrylates (Stansbury and Idacavage, 2016). Nevertheless, since each new layer is closely connected with prior layer during photopolymerization high strength and minimal anisotropy in the structure of printed parts is obtained (Stansbury and Idacavage, 2016). The SLA 3D printing technology is the most investigated in dentistry applications, and it is found that it can achieve the best accuracy in full arch dental models (Etemand-Shahidi et al., 2020).

One should also keep in mind that the post-polymerization plays an important role in the UV curing and it can lead to polymerization shrinkage (Tanasic et al., 2015; Milosevic, 2016). To measure shrinkage 3D optical deformation analysis was used for liquid resin-based composites (Lezaja et al., 2015; Tanasic et al., 2012). Recent research has shown that there is no significant material shrinkage of photopolymer resin used in SLA 3D printing technology in comparison with PLA thermoplastic material (Milovanovic et al., 2018).

Digital dentistry is rapidly becoming more and more used in everyday practice. Dentists are now able to use 3D scanners to record state of soft and hard tissues, which can be followed with 3D printing of dental models or supporting structures, such as surgical guides and aligners. One of the best examples of digital dentistry advantages is the introduction of dental aligners in 1997 (Machado, 2020). In the scope of clear aligner orthodontic therapy dental aligners (shown in Fig. 1) are used to change position, angulation and rotation of teeth, improving all parameters needed for a proper and healthy occlusion and articulation (Moshiri et al., 2017; Ke et al., 2019; Ryu et al., 2017).

Preparation for 3D printing begins with computerized technology and scanning of the patient's teeth with an intraoral scanner, or scanning the cast model obtained from a dental impression, combining laser and optical scanning to create a digital model. Then series of incremental changes are made on the digital model, followed by matching series of stereolithographic casts for aligner fabrication (Buschang et al., 2015; Zhang et al., 2015; Boyd et al., 2001; Kravitz et al., 2009). Each aligner is worn for at least 22 h a day for approximately one month. Then, patient moves to the next aligner. During this period the aligner exhibits force on the teeth and if patient is dedicated enough good results can be expected. This system can, in many occasions, replace dental braces that are still the gold standard of orthodontic treatment today. One of the biggest advantages and the main reason why patients choose aligners is the possibility to remove it when needed (Cooper-Kazaz et al., 2013).

During the treatment many different forces are applied on teeth in order to place them in proper position. It is very important that the aligner material can stand the stresses caused by this process. Furthermore, the most important material properties, such as tensile, compressive and flexural strength, must be stable from the beginning of the therapy. Namely, if the properties are changing when the aligner is already in position, the dentist will not have control of the force applied on teeth, as well as of the placement of teeth. Therefore, it is very important to know the best moment after 3D printing to place the aligner.

Fig. 1. Clear dental aligner placed on dental model (Formlabs.com, 2020).

The force transmission mechanism of the clear aligner orthodontic therapy is specific and completely different than the mechanism used in conventional orthodontic therapy (Machado, 2020; Cortona et al., 2020). In the conventional therapy forces originating from the metal wire are transmitted to the bracket which is bonded to the tooth surface. This is causing displacement of the tooth. However, with clear aligner therapy, the tooth displacement is caused by a mismatch between the teeth and the aligner. The goal is to position teeth as predetermined by the aligner design. The aligner is fitted on dental arch but, because of this intentional mismatch, significant forces are generated. Although these forces are needed to cause the tooth displacement into desired position, they also cause significant stress generation in the aligner itself (Cortona et al., 2020).

The 3D printing process demands control of various parameters. Mechanical and physical properties of printed part are influenced by several significant parameters: choice of material, manufacturing method, thickness of printed layers, shrinkage between layers, direction of 3D printing, process of polymerization and post-polymerization (Chantarapanich et al., 2013; Zhang et al., 2019; Kim et al., 2020; Shim et al., 2020; Reymus, Stawarczyk, 2020). Complete understanding of different 3D printing parameters is crucial, because they are affecting mechanical strength and quality of printed part (Puebla et al., 2012).

This study is focused on the effect of time on mechanical properties of aligners, with an aim to define the optimal moment of 3D printed aligner placement. Experiments performed here included tensile, compressive and three-point bending testing of the biocompatible Dental LT Clear V1 (Formlabs) material.

2. Materials and methods

2.1. Materials

Material examined in this research is biocompatible Dental LT Clear V1 (Formlabs) polymer, used for manufacturing of splints and aligners. Dental LT Clear V1 resin is a monomer based on acrylic ester, Class IIa i. e., long-term biocompatible resin according to EN-ISO 10993–1:2009/AC:2010 (ISO Standard, 2009). This polymer has a high resistance to fracture, which implies that it is a good solution for production of hard splints, retainers and other direct-printed long--termorthodontic appliances (Zguris, 2016).

This material satisfies crucial requirements and terms of the Medical Device Directive 2007/47/EC (Formlabs.com, 2017b; Formlabs.com, 2017c) and Council Directive 93/42/EEC. It also corresponds to series of specific standards including, EEN ISO 1641:20092 (BS EN 1641:2009, 2009), EN-ISO 10993–3:2009 (ISO, 2009 - Formlabs Application Guide, 2017), EN-ISO 10993–5:2009 (ISO, 2009 - Formlabs Application Guide, 2017) and EN 908:2008 (ISO, 2009 - Formlabs Application Guide, 2017).

According to all these standards, it is necessary to follow the precise procedures for ensuring strict control of biocompatibility and preparation of samples, as well as printing and post-processing (Formlabs.com, 2017d). Formlabs Form 2 3D printer was used to print all the samples from Dental LT Clear V1 resin in order to conduct *in vitro* testing.

2.2. Preparation of specimens

Specimens are produced according to International Organization for Standardization (ISO) declaration for particular shape characteristics and dimensions. Testing of particular plastics is performed using ISO 527-2 standard for tensile tests (ISO 527–2:2012), to evaluate tensile strength, yield strength, modulus of elasticity, elongation at yield and at failure. Compressive tests were performed according to ISO 604 standard (ISO 604:2002) and three-point bending tests were performed according to ISO 178 standard (ISO 178:2019).

For this research five specimens were made for each of the three types of testing. All specimens were designed according to standards using SolidWorks (Dassault Systemes, France), then converted to.stl file format used for preparation of models for 3D printing. Specimen for tensile testing modelled in SolidWorks is shown in Fig. 2.

Specimen dimensions for compressive testing were 10x10 × 20 mm, shown in Fig. 3. All deviations from defined dimensions were measured using Vernier caliper and registered in TrapeziumX software (Shimadzu Corp., Kyoto, Japan), used for software support of tensile, compression and bending testing on particular machine.

For three-point bending testing specimen dimensions were defined according to ISO 178 standard (ISO 178:2019), and specimen is modelled in SolidWorks software with listed dimensions, shown in Fig. 4. Also, in Fig. 4 bending specimen is shown positioned between supporting pins and loading pin, which are components of bending test fixture. Specimen length, height and thickness were 80 mm, 4 mm and 10 mm, respectively. According to standard, for presented specimen dimensions recommended loading and supporting pin radius is 5 mm.

After 3D printing specimens were rinsed from any liquid resin pasted to finished parts in 99,5% iso-propyl alcohol, for two sessions of 10 min; afterwards the parts were dried at room temperature for 40 min, followed by UV post-curing to raise conversion of the photopolymer, according to the demands of Formlabs company (Formlabs.com). The finished parts generally had low surface roughness, so the final surface finishing was performed by hand sanding with fine sandpaper.

2.3. Tensile, compressive and three-point bending testing

Tensile, compressive and three-point bending tests were performed on Shimadzu AGS-X universal testing machine (Shimadzu Corp., Kyoto, Japan). Load cell capacity of this particular machine is 100 kN, with measurement inconsistency below 200 N. Machine can perform tensile, compressive and bending testing depending on the mounted adaptors. For tensile testing required adaptors are grips intended for tensile testing and extensometer for strain measurement (Fig. 5 – left). Compressive testing requires two plan-parallel plates, in order to adequately apply compressive load to tested specimen (Fig. 5 – middle). Mounted adaptors for three-point bending are shown in Fig. 5 - right.

For three-point bending the specimen was placed on two supporting pins, distanced apart. In this testing supporting pins were at 64 mm fixed distance. For force measurement different load cell was applied due to low measured forces in particular test, using load cell with capacity of 5 kN, which resulted in precise measurements. All specimens were made with identical printing parameters.

2.4. Effect of time on mechanical properties

Observation and examination of time effect influence on behavior and properties of polymers is important for different reasons, namely usability, lifetime and storage. In order to control degradation of polymer materials it is necessary to understand different chemical mechanisms behind structural changes in polymer macromolecules, reaction pathways of polymer additives, influences of polymer morphology, and

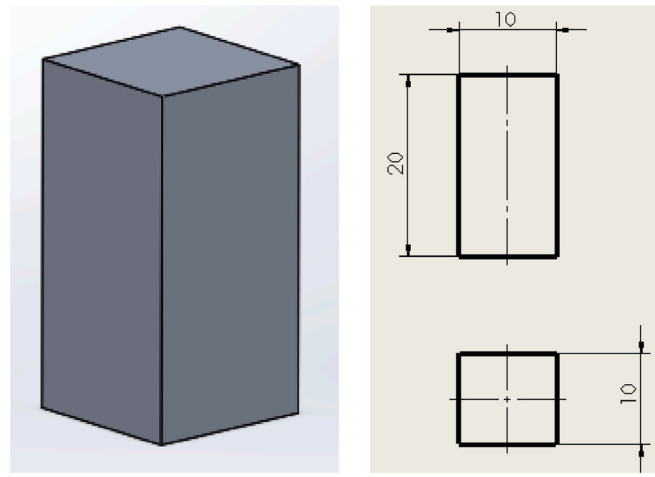

Fig. 3. Left - Specimen for compressive testing in SolidWorks software; Right - Specimen dimensions in millimeters.

complex process of oxidation chemistry. In dental practice it is not uncommon that some aligners arrive few days after fabrication, meaning that patient will have aligners with different mechanical properties than expected.

Post-curing processes using UV light source were applied to promote the polymerization of material, thus increasing its stability and strength. Depending on the used resin material, geometry and size of the model, post-curing processes are defined. Since Dental LT Clear V1 resin is fairly new material, there are only few studies dealing with its post-curing and post-polymerization properties in terms of different periods of time (Jindal et al., 2020; Maserpo, Tartaglia, 2020).

Mansour et al. investigated 24 days aging effect on tensile properties of epoxy resins. Results have shown that tensile strength, stiffness and flexural modulus increased. On the other side, because of the aging material became more brittle resulting in the decrease of impact resistance and elongation at failure (Mansour et al., 2007). Other group investigated aging and heat treatment influence on the mechanical properties of PLA filament printed parts. Their conclusions suggest that mechanical properties of the PLA parts degrade more when the printing resolution is lower i.e., with higher layer height (Hasan et al., 2020).

Our research included monitoring of effect of time on mechanical properties of biocompatible thermoset plastics, used in SLA 3D printing technology for printing the dental aligners. Four sets of specimens were made for all three mentioned tests. Specimens were observed in time period of 1 day (24 h), 3 days (72 h), 5 days (120 h) and 7 days (168 h) after printing. All specimens were stored under the same conditions, in darkened room to reduce environment effects, at room temperature (24 °C) and constant humidity (55–58%).

3. Results

Universal testing machine Shimadzu AGS-X (Shimadzu Corp., Kyoto, Japan), with which tensile, compressive and three-point bending tests

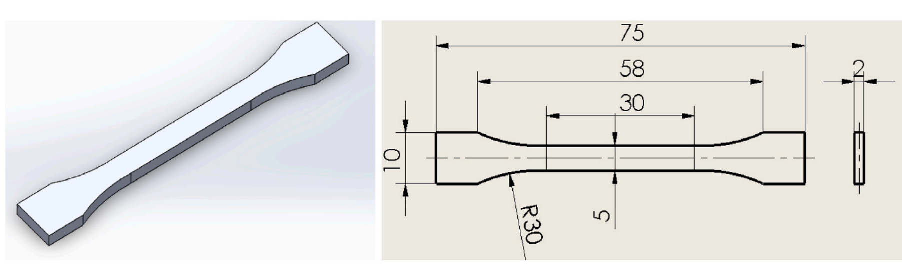

Fig. 2. Left - Tensile test specimen in SolidWorks software; Right - Specimen dimensions in millimeters.

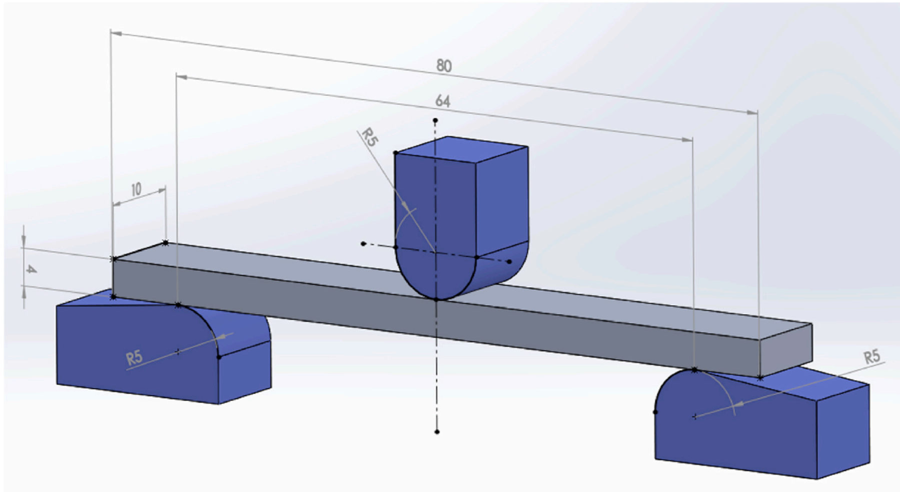

Fig. 4. Specimen for three-point bending test, placed on a bending test fixture, with dimensions in millimeters.

Fig. 5. Shimadzu AGS-X universal testing machine: Left - with loaded tensile testing grips and applied extensometer to tested specimen; Middle - with mounted tools which mimic two plan-parallel surfaces; Right - with mounted adaptors for three-point bending test.

were performed, consists of a load cell, vertically mobile crosshead, upper and lower grips - which hold the specimen and constrain any movement of the placed specimen in the Universal testing machine in the case of tensile testing (Fig. 5 - left). For the purposes of Elastic modulus evaluation gauge measurement i.e., extensometer, was used (Epsilon Technology Corp., Jackson, WY, USA). Gauge measurement in tensile testing is preferred, due to more localized area of measurement which will eventually result in greater precision of measured values. One should notice that the specimen was not loaded only axially during tensile test, but also with lateral loads due to relatively heavy extensometer, which resulted in specimen failure at locations which are by standard defined as non-valid for tensile testing. Hence, only 1/5 specimens were successfully tested with extensometer showing that there is a 29.7% difference in elastic modulus value obtained directly from the universal testing machine and the value obtained from extensometer. Considering that the strain measuring range of machine is the distance between grips, in this case 60 mm, while gauge length of extensometer was 25 mm, differences in measured values between machine and extensometer were expected. Nominal Stress – Nominal Strain diagrams attained from both the machine and extensometer are displayed on Fig. 6. Curves on the diagram in Fig. 6 are collected from the TrapeziumX software, and if they are not distinguishable to the reader, then an additional explanation is needed: the curve representing data collected from the extensometer is on the left-hand side in the diagram, opposite to the curve collected from the machine data.

In all the following elastic modulus results are measured only according to universal testing machine's readings. Tensile testing is performed by placing the specimen between upper and lower grips, which fix the specimen's upper and lower side, thus constraining specimen's movement. Specimen must be appropriately placed and adequately tightened between the grips. If the specimen is not properly tightened it will slip from the fixing grips during tensile testing, causing

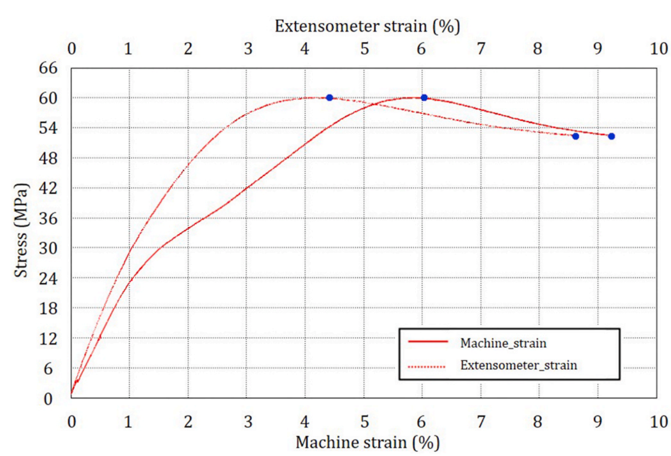

Fig. 6. Nominal Stress - Nominal Strain diagrams acquired from both the machine and extensometer.

testing failure. If the specimen is overtightened it will likely fracture at the site near the fixing grip, upper or lower one, which is a fracture site that is inappropriate for tensile tests. Tensile tests are performed according to ISO 527-2 standard (ISO, 2012).

Defining a testing method in TrapeziumX software (Shimadzu Corp., Kyoto, Japan) implies definition of strain rate, specimen dimensions and required output. Strain rate was set at 1 mm/min. Dimensional mismatches after 3D printing were within tolerance limits. Specimen thickness and width were measured in three points, and the mean values were used for all three types of testing. Elastic modulus, yield stress, elongation at yield, failure stress and elongation at failure were measured and average values of five specimens are presented in Table 1, together with standard deviation, whereas corresponding nominal stress – nominal strain diagrams are shown in Fig. 7.

Considering that a material in dental practice is most often loaded in pressure, the compressive strength is of utmost importance. Compressive tests are performed according to ISO 604 standard with strain rate set at 2 mm/min, for five specimens per batch. Compressive yield stress, compressive elongation at yield, ultimate compressive strength (UCS), elongation at failure and compression modulus were measured and average values are presented in Table 2, together with standard deviation. Corresponding nominal stress - nominal strain diagrams are shown in Fig. 8, indicating excellent repeatability.

Three-point bending tests were performed according to ISO 178 standard (ISO 178:2019,), with five specimens per batch. Flexural strength, elongation at flexural strength, failure stress, elongation at failure and flexural modulus were measured and values are presented in Table 3 with corresponding diagrams shown in Fig. 9, indicating sufficient repeatability. Somewhat higher standard deviation is noticed for failure stress and elongation at failure, due to amorphous nature of tested material.

Elastic modulus, compression modulus and flexural modulus of all conducted tests were estimated according to stress/strain ratio in the elastic region on tensile, compression and three-point bending test diagram, respectively.

Strain in flexural testing is measured according to ISO 178 standard using formula (1):

$$\varepsilon = \left(\frac{6Sh}{L^2}\right)\cdot 100\% \tag{1}$$

where h is the specimen height, and L is the distance between supporting pins, being 4 mm and 64 mm, respectively, and S is deflection (ISO 178:2019,) measured as the displacement of the top specimen surface middle point. In three-point bending tests this point is coincident with the loading pin tip. Position of the loading pin in these tests is controlled by the machine and represents the measure of the top surface middle point displacement, required in the formula (1). The flexural stresses were evaluated according to formula (2):

$$\sigma = \frac{3FL}{2bh^2} \tag{2}$$

where b is the thickness of the specimen i.e., $b = 10$ mm (Fig. 4) and F is

Table 1
Mechanical properties from tensile test.

	Yield stress	Elongation at yield	Failure stress	Elongation at failure	Elastic modulus
Unit	MPa	%	MPa	%	GPa
Specimen_1	37.82	4.1	35.17	15.1	1.69
Specimen_2	33.78	4.3	31.35	13.0	1.50
Specimen_3	32.91	4.5	33.02	18.0	1.40
Specimen_4	37.33	4.4	34.83	16.7	1.51
Specimen_5	36.68	4.3	34.28	16.8	1.58
Average	35.70	4.3	33.73	15.9	1.54
Standard Deviation	2.21259	0.14832	1.56162	1.93054	0.10738

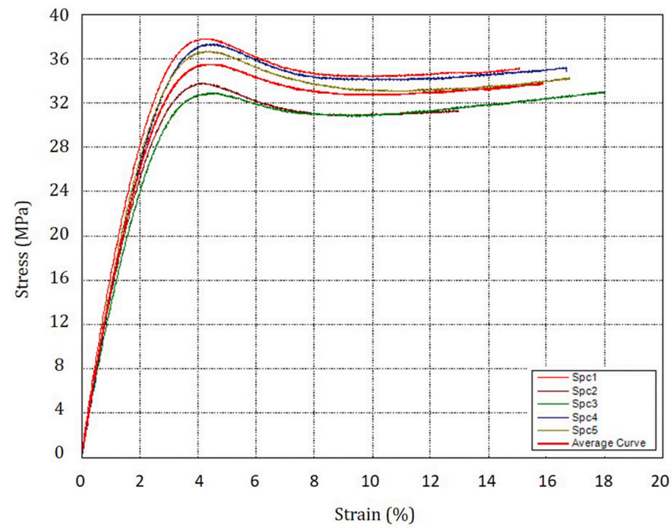

Fig. 7. Nominal stress – nominal strain diagrams of one tensile test batch.

measured force value from the load cell of universal testing machine.

Time effect on tensile properties of Dental LT Clear V1 resin material was observed on specimens after 1 day (24 h), 3 days (72 h), 5 days (120 h) and 7 days (268 h). Average results per each day for tensile testing are shown in Table 4.

Results show that ultimate tensile strength and failure stress increase with time, whereas elongation decreases. Between days 3 and 5 there is no clear distinction in both elongation at yield and elongation at failure, but the differences in values are obvious between days 1 and 7. The highest increase in both ultimate tensile strength and failure stress is between days 3 and 5 i.e., 12.8% and 9.6% respectively. Elastic modulus increases with time, with no clear distinction between days 3 and 5.

Time effect on average values of compressive yield stress, ultimate compressive strength (UCS), compressive elongation at yield, elongation at UCS and compression modulus is shown in Table 5.

Compressive tests show that until day 5 specimen's compressive yield stress and ultimate compressive strength increases, with decrease in overall strain. Highest increase in compressive yield stress and ultimate compressive strength is between days 3 and 5 i.e., 15% and 11.7%, respectively. In day 7, there is a decrease in compressive yield stress and ultimate compressive strength with increase in elongation at yield value. Elongation at UCS has identical value between days 5 and 7, with assumption that after day 5 compressive properties of material stabilize. Compression modulus has the highest value at days 3 and 5 i.e., 0.26 GPa. Lowest compression modulus is present in specimens from day 1.

Time effect on average values of flexural strength, elongation at flexural strength, failure stress, elongation at failure and flexural modulus for three-point bending are shown in Table 6.

Results for three-point bending tests show highest values for flexural strength and failure stress for specimens from day 7. Only mismatch from the increase trend are specimens from day 5, since flexural strength and failure stress decrease 15% and 9.9%, respectively, in comparison with their values from day 3. Lowest values for elongation at flexural strength are present in specimens from day 7. Elongation at failure has the lowest values in specimens from day 5, only lower by 0.2% from day 7 specimens. There is no clear difference between elongations from days 3 and 5. Flexural modulus has an increase trend from day 3, with highest increase between days 5 and 7.

Examined Dental LT Clear V1 resin is an older version of recently introduced Dental LT Clear V2 resin, but still more used material in dental practice. Only few material properties data for Dental LT Clear V1 resin is available on Formlabs website (Formlabs Dental LT Clear,): minimal ultimate flexural strength, minimal flexural modulus, Shore D hardness range, minimal stress intensity factor and total fracture work i.

Table 2
Mechanical properties from one compressive test.

	Compressive yield stress	Elongation at yield	Ultimate compressive strength	Elongation at failure	Compression modulus
Unit	MPa	%	MPa	%	GPa
Specimen_1	66.39	6.5	199.62	47.9	0.39
Specimen_2	65.14	7.2	185.57	50.0	0.16
Specimen_3	67.03	7.3	191.89	47.9	0.18
Specimen_4	65.95	7.0	204.66	48.6	0.23
Specimen_5	65.12	6.9	224.00	50.2	0.36
Average	65.93	7.0	201.15	48.9	0.26
Standard Deviation	0.822	0.311	14.707	1.117	0.08

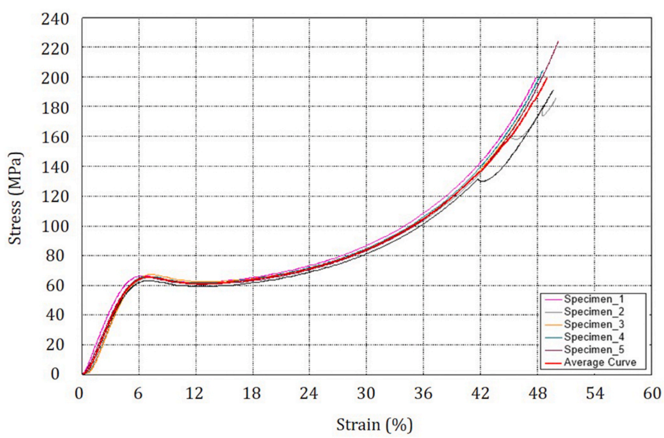

Fig. 8. Nominal stress – nominal strain diagrams of one batch of compressive tests.

Table 3
Mechanical properties from one three-point bending test.

	Flexural strength	Elongation at flexural strength	Failure stress	Elongation at failure	Flexural modulus
Unit	MPa	%	MPa	%	GPa
Specimen_1	61.33	6.5	37.21	15.0	1.36
Specimen_2	59.18	7.6	37.58	14.5	1.4
Specimen_3	58.86	6.1	20.26	15.3	1.47
Specimen_4	58.85	6.7	53.08	9.7	1.31
Specimen_5	56.01	6.8	43.64	12.7	1.24
Average	58.85	6.7	38.35	13.4	1.36
Standard Deviation	1.892	0.550	11.980	2.321	0.08

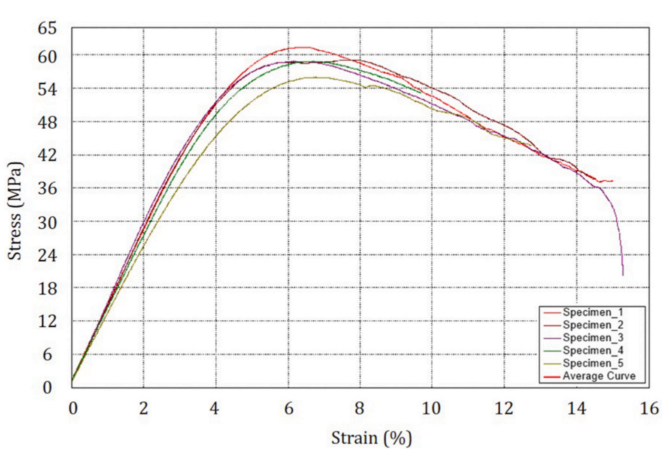

Fig. 9. Nominal stress – nominal strain diagrams of one batch of three-point bending tests.

Table 4
Tensile testing results for Dental LT Clear V1 resin material.

Day	Ultimate tensile strength [MPa]	Failure stress [MPa]	Elongation at yield [%]	Elongation at failure [%]	Elastic modulus [GPa]
1	35.7	33.7	4.3	15.6	1.54
3	40.7	35.5	4.5	12.1	1.9
5	45.9	38.9	4.1	12.9	1.8
7	48.8	40.33	3.9	11.5	2.2

Table 5
Compressive testing results for Dental LT Clear V1 resin material.

Day	Compressive yield stress [MPa]	UCS [MPa]	Elongation at yield [%]	Elongation at UCS [%]	Compression modulus [GPa]
1	59.2	173.5	7.1	46.3	0.14
3	65.9	201.1	7	48.9	0.26
5	75.8	224.7	6.6	49.6	0.26
7	71.7	207.5	7.1	49.6	0.22

Table 6
Three-point bending testing results for Dental LT Clear V1 resin material.

Day	Flexural strength [MPa]	Failure stress [MPa]	Elongation at flexural strength [%]	Elongation at failure [%]	Flexural modulus [GPa]
1	58.8	38.3	6.7	13.4	1.41
3	65.8	50.3	7.1	13.1	1.32
5	57.1	45.8	6.7	11.7	1.37
7	70.7	53.6	6.1	11.9	1.66

e., toughness. In all flexural tests in this research flexural strength and flexural modulus values for post-cured resin are over 50 MPa and 1.2 GPa, respectively. These values are defined as minimal values by the Formlabs (Formlabs Inc., Sommerville, MA, USA), thus proving the eligibility of results from this research.

In general, results from all three types of testing show that time has a significant effect on mechanical properties of tested material. Older samples can, in most cases, sustain higher stresses at the expense of elongation.

4. Discussion

Changes in mechanical characteristics of printed part are result of the fact that polymerization continues even after all curing steps are done. Polymerization as such implies chemical reactions in which unsaturated monomer molecules are connecting and propagating to a chain network. Process of photo-polymerization further implies polymerization in which monomers are connecting in polymer chains, changing mechanical properties at the same time (Fouasier et al., 2014; Sangermano et al., 2018; Carve, Wlodkowic, 2018).

In previous research it was found that strength and dimensional accuracy of printed parts can be affected by post-curing process, so that different post-curing mechanism result in different characteristics (Huettig et al., 2017; Reymus et al., 2019a; Reymus et al., 2019b).

Kim et al. confirmed that increase in the degree of polymerization does not occur evenly across the entire cross-section of a sample, which is then leading to further questions about effects on strength (Kim et al., 2020).

Post-polymerization period in this study was limited to 7 days, but concerning the fact that mechanical properties are changing and increasing till day 7, it can be expected that changes are happening even afterwards. It should be also taken into account that environmental conditions can influence mechanical properties of aligners.

Temperature, humidity and salivary enzymes affect the aligner and its mechanical properties in intraoral environment (Maspero, Tartaglia, 2020; Ryokawa et al., 2006). However, this research is limited on time dependent changes before aligner placement. In dental practice large dental clinics often can offer application of aligners 1 day after printing. But, in most cases, smaller ones do not have this option and because of that period of application of aligners in patients can be 5 to 7 days after printing. Time boundaries in this study (1, 3, 5 and 7 days after printing the aligners) are set based on these most common situations. Future research would be directed towards time dependent changes happening in an intraoral environment.

Orthodontic appliances displace teeth by delivering force to them. The force must be able to displace the tooth without any damage (for example apical root resorption). The orthodontic force delivered to the tooth was found to be between 0.18 and 2.91 N which is similar with the force delivered by Nickel–Titanium wire in conventional therapy (Kohda et al., 2013). In another study where the intrusion and extrusion forces were measured the maximal force was found to be 0.92 N and -0.52 N (Liu, 2018). Elkholy and coworkers analyzed whether the intensity of the force can be reduced by changing the thickness of the aligner. The force intensity reduction is important because higher forces can provoke root resorption (Elkholy et al., 2016). Further, intentional mismatch of the aligner and teeth position will cause significant deformation of the aligner, which must be reversible. The best material for an aligner should have highest possible stiffness and yield strength sufficient to ensure that the applied force causes only elastic strain. In that manner forces in an aligner are safe and efficient (Jindal et al., 2019). It is also important to avoid any possible problem that can occur due to unawareness of the changes of these properties caused by time passing. Effect of the aligner on teeth could be compromised causing loss of the control on orthodontic tooth displacement which could make serious and irreversible damage, eventually causing aligner failure. In other words, testing of these changes can easily give the information what is the right time to apply the aligner. Although compressive stress is generated in the aligner during its function, much stronger compressive forces are formed between the two dental arches. Namely, the success of the clear aligner therapy is expected if the patient wears the aligners 22 h per day. Although patients are recommended not to take food and chew with aligners on teeth, occlusal forces can still be generated due to unwilling clenching and grinding of teeth. Thus, aligner must resist compressive forces that can be 700 N or even higher. In our study compressive stress at yield was between 59.2 MPa and 75.8 MPa, and considering specimen size, forces applied at yield were one order of magnitude higher than 700 N, as defined for aligner material. Hence, material can sustain much higher compressive loads without any geometry change.

During the use of aligners complex loadings are present. If the goal is to make the rotation of teeth on the model of aligner higher strains appear. If molar tooth should be rotated model will be loaded in flexion. During night in patients with bruxism aligner is under tension and bending loads.

Although mismatch between teeth position and the aligner is small (about 0.25 mm and less than 3° for rotation), stresses and displacements can be high. In a FEA study the stress inside the aligner has been investigated (among other parameters), and its intensity was found to be greater in the cases with higher aligner activation for tooth rotation and in cases with attachments (up to 3.7 MPa). In the same cases deformation of the aligner was the greatest (up to 0.29 mm) (Cortona et al., 2020). In results shown in Tables 4–6 even lowest values are one order of magnitude higher than in previously mentioned case.

In clear aligner therapy significant tensile stresses are expected. Tested Dental LT Clear V1 resin material has yield stress between 35.7 and 48.8 MPa, depending on the time which has passed, with highest values for older samples. Also, deformation ranged between 3.9 and 4.3 mm with lower values for older samples. Thus, effect of time on particular material shows promises for future application in dental practice.

High flexural strength is needed in cases when a tooth in arch is missing so the antagonistic teeth are making pressure on the area of aligner which is not supported with tooth underneath it. Flexural strength of Dental LT Clear V1 resin material in our results ranges from 58.8 MPa to 70.7 MPa. Oldest tested samples (7 days), showed highest flexural strength and lowest elongation. Our research of Dental LT Clear V1 resin material also indicates benefits from time passing in bending properties.

This research confirmed that 3D printed aligners can fulfill mechanical demands of a device used for orthodontic tooth displacement as previously stated (Jindal et al., 2019, 2020). As already mentioned, 3D printed aligners can provide much more reliable geometry which is of crucial importance for control of tooth displacement and force intensity (Jindal et al., 2020). Also, it is important to avoid any dimensional inaccuracies which could be significant when aligners are made on 3D printed models. Above all, production and post-processing of the aligners is shorter and less demanding using 3D printing technology in comparison with conventional fabrication technologies.

5. Conclusions

Impact of 3D printing technologies in dentistry is significant with very promising future development of new approaches and manufacturing procedures of dental restorations. Different factors influence the mechanical properties of 3D printed objects, including specification of material which is used, 3D technology, printing parameters such as: percentage of infill, orientation of printing, resolution i.e., layer height, and post-processing procedure. Mechanical behavior of 3D printed part affected with certain loading condition is difficult to predict. Hence, there is a need to take as many printing parameters into account as possible to get better insight into setting of the procedure.

Effect of time on biocompatible photopolymer resin shows promising results in enhancement of mechanical properties on material used for clear dental aligners. Forces transmitted on aligners during exploitation can be as high as 700 N in compression. Conducted tensile, compressive and three-point bending tests indicate one order of magnitude higher values than maximal recorded forces transmitted on teeth. Concerning time effect, all specimens in all three types of testing show promising results, with tensile, compressive and flexural strength increasing, and elongation at failure slightly decreasing.

However, in order to get better control of the tooth movement it can be suggested that the best performance of the aligner can be expected if the device is handed over after the day 7.

Author Contributions

Aleksa Milovanović: Writing - Original Draft, Review & Editing, Data curation, Investigation

Aleksandar Sedmak: Conceptualization, Supervision, Writing - Review & Editing, Methodology

Zorana Golubović: Writing - Original Draft, Review & Editing, Visualization, Investigation

Ksenija Zelić Mihajlović: Writing

Aleksandar Žurkić: Software, Validation
Isaak Trajković: Investigation
Miloš Milošević: Resources, Project Administration, Funding

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This research is financially supported by European Union's Horizon 2020 research and innovation program ''SIRAMM'', under grant agreement No. 857124.

References

ASTM F2792-12a, 2013. Standard Terminology for Additive Manufacturing Technologies. ASTM International, West Conshohocken, PA. www.astm.org.
Boyd, R.L., Waskalic, V., 2001. Three-dimensional diagnosis and orthodontic treatment of complex malocclusions with the invisalign appliance. Semin. Orthod. 7, 274–293. https://doi.org/10.1053/sodo.2001.25414.
BS EN 1641:2009, 2009. Dentistry. Medical devices for dentistry. Materials. British Standards Institution [BSI]. www.bsigroup.com.
Buschang, P.H., Ross, M., Shaw, S.G., Crosby, D., Campbell, P.M., 2015. Predicted and actual end-of-treatment occlusion produced with aligner therapy. Angle Orthod. 85, 723–727. https://doi.org/10.2319/043014-311.1.
Carve, M., Wlodkowic, D., 2018. 3D-Printed chips: compatibility of additive manufacturing photopolymeric substrata with biological applications. Micromachines 9, 91. https://doi.org/10.3390/mi9020091.
Chantarapanich, N., Puttawibul, P., Sitthiseripratip, K., Sucharitpwatskul, S., Chantaweroad, S., 2013. Study of the mechanical properties of photo-cured epoxy resin fabricated by stereolithograpy. J. Sci. Technol. 35 (1), 91–98.
Cooper-Kazaz, R., Ivgi, I., Canetti, L., Bachar, E., Tsur, B., Chaushu, S., Shalish, M., 2013. The impact of personality on adult patients' adjustability to orthodontic appliances. Angle Orthod. 83 (1), 76–82.
Cortona, A., Rossini, G., Parrini, S., Deregibus, A., Castroflorio, T., 2020. Clear aligner orthodontic therapy of rotated mandibular round-shaped teeth: a finite element study. Angle Orthod. 90 (2), 247–254.
Elkholy, F., Schmidt, F., Jäger, R., Lapatki, B.G., 2016. Forces and moments delivered by novel, thinner PET-G aligners during labiopalatal bodily movement of a maxillary central incisor: an in vitro study. Angle Orthod. 86 (6), 883–890.
Etemad-Shahidi, Y., Qallandar, O.B., Evenden, J., Alifui-Segbaya, F., Ahmed, K.E., 2020. Accuracy of 3-dimensionally printed full-arch dental models: a systematic review. J. Clin. Med. 9, 3357, 10.3390/jcm9103357.
Formlabs.com. https://dental.formlabs.com/materials/#dental-lt-clear-v2. (Accessed 19 October 2020).
Formlabs.com, 2017d. Instructions for use: dental LT clear. available at: https://formlabs.com/media/upload/DentalLTClear-Instructions-for-use-webpdf.pdf. . (Accessed 19 October 2020).
Formlabs Application Guide, 2017. 3D Printing Splints with the Form 2. "Formlabs application guide: 3D printing splints with the form 2", available at: https://formlabs.com/media/upload/AppGuide-Splints-Retainers-EN.pdf. (Accessed 19 October 2020).
Formlabs Dental LT Clear. Material Data Sheet (2017e), '' Formlabs Dental LT Clear: material Data Sheet''. available at: https://dental-media.formlabs.com/datasheets/Dental_LT_Clear_Technical.pdf. (Accessed 17 February 2021).
Formlabs.com, 2017b. EU declaration of conformity. available at: https://formlabs.com/media/upload/Declaration_of_Conformity_Dental_LT_Clear_DoC-FLOC-2017-01-UK.pdf. . (Accessed 19 October 2020).
Formlabs.com, 2017c. Dental LT Clear: Biocompatible Photopolymer Resin for Form 2 [Internet], available at: https://formlabs.com/media/upload/DentalLTClear-DataSheet-EN.pdf/. (Accessed 19 October 2020).
Fouassier, J.P., Lalevée, J., 2014. Photochemical production of interpenetrating polymer networks; simultaneous initiation of radical and cationic polymerization reactions. Polymers 6, 2588–2610.
Hasan, M.S., Ivanov, T., Vorkapic, M., Simonovic, A., Daou, D., Kovacevic, A., Milovanovic, A., 2020. Impact of aging effect and heat treatment on the tensile properties of PLA (poly lactic acid) printed parts. Mater. Plast. 57 (3), 147–159. https://doi.org/10.37358/MP.20.3.5389.
Huettig, F., Kustermann, A., Kuscu, E., Geis-Gerstorfer, J., Spintzyk, S., 2017. Polishability and wear resistance of splint material for oral appliances produced with conventional, subtractive, and additive manufacturing. J. Mech. Behav. Biomed. Mater. 75, 175–179.
International Organization for Standardization ISO 10993-1: 2009. Biological Evaluation of Medical Devices–Part 1 ISO 10993-1, 2009. Evaluation and Testing within a Risk Management Process; International Organization for Standardization ISO. Geneva, Switzerland, 2009.
ISO 178:2019, 2019. Plastics — Determination of Flexural Properties. International Organization for Standardization ISO: Geneva, Switzerland.
ISO, ISO 527-2:2012, ISO/TC 61/SC 2 Mechanical behavior, 2012.
ISO 604:2002, 2017. Plastics — Determination of Compressive Properties. International Organization for Standardization ISO, Geneva, Switzerland.
Jindal, P., Juneja, M., Siena, F.L., Bajaj, D., Breedon, P., 2019. Mechanical and geometric properties of thermoformed and 3D printed clear dental aligners. Am. J. Orthod. Dentofacial Orthop. 156 (5), 694–697, 01.
Jindal, P., Worcester, F., Siena, F.L., Forbes, C., Juneja, M., Breedon, 2020. Mechanical behavior of 3D printed vs thermoformed clear dental aligner materials under non-linear compressive loading using FEM. J. Mech.Behav.Biomed.Mater 21 (112), 104045.
Ke, Y., Zhu, Y., Zhu, M., 2019. A comparison of treatment effectiveness between clear aligner and fixed appliance therapies. BMC Oral Health 19 (1), 24.
Kim, D., Shim, J.-S., Lee, D., Shin, S.-H., Na-Eun Nam, N.-E., Park, K.-H., Shim, J.-S., Kim, J.-E., 2020. Polymers 12, 2762. https://doi.org/10.3390/polym12112762.
Kohda, N., Iijima, M., Muguruma, T., Brantley, W.A., Ahluwalia, K.S., Mizoguchi, I., 2013. Effects of mechanical properties of thermoplastic materials on the initial force of thermoplastic appliances. Angle Orthod. 83 (3), 476–483.
Kravitz, N.D., Kusnoto, B., BeGole, E., Obrez, A., Agran, B., 2009. How well does Invisalign work? A prospective clinical study evaluating the efficacy of tooth movement with Invisalign. Am. J. Orthod. Dentofacial Orthop. 135, 27–35. https://doi.org/10.1016/j.ajodo.2007.05.018.
Lezaja, M., Veljovic, Dj, Manojlovic, D., Milosevic, M., Mitrovic, N., Janackovic, Dj, Miletic, V., 2015. Dental Materials, Publisher. Bond Strength of Restorative Materials to Hydroxyapatite Inserts and Dimensional Changes of Insert-Containing Restorations during Polymerization, vol. 31. Elsevier Inc Volume, 01095641. https://doi.org/10.1016/j.dental.2014.11.017. Issue 2, Pages 171–181.
Ligon, S.C., Liska, R., Stampfl, J., Gurr, M., Mulhaupt, R., 2017. Polymers for 3D printing and customized additive manufacturing. Chem. Rev. 10212–10290, 10.1021/acs.chemrev.7b00074.
Liu, Y., Hu, W., 2018. Force changes associated with different intrusion strategies for deep-bite correction by clear aligners. Angle Orthod. 88 (6), 771–778.
Machado, R.M., 2020. Space closure using aligners, 25. Dental Press J Orthod, pp. 85–100. https://doi.org/10.1590/2177-6709.25.4.085-100.sar, 4.
Mansour, S., Gilbert, M., Hague, R., 2007. A study on the impact of short-term aging on the machanical properties of a stereolithography resin. Materials Science and Engineering: A 447 (1–2), 277–284. https://doi.org/10.1016/j.msea.2006.10.007.
Maspero, C., Tartaglia, G.M., 2020. 3D printing of clear orthodontic aligners: where we are and where we are going. Materials 13, 5204. https://doi.org/10.3390/ma13225204.
Milosevic, M., June 2016. Polymerization mechanics of dental composites-advantages and disadvantages. In: International Conference on Manufacturing Engineering and Materials, ume 149. ICMEM 2016; Novy Smokovec, Slovakia, pp. 313–320.
Milovanovic, A., Milosevic, M., Mladenovic, G., Likozar, B., Colic, K., Mitrovic, N., 2018. Experimental dimensional accuracy analysis of reformer prototype model produced by FDM and SLA 3D printing technology. In: Experimental and Numerical Investigations in Materials Science and Engineering, vol. 54. Springer, pp. 84–95. https://doi.org/10.1007/978-3-319-99620-2_7.
Moshiri, S., Araújo, E.A., McCray, J.F., Thiesen, G., Kim, K.B., 2017. Cephalometric evaluation of adult anterior open bite non-extraction treatment with Invisalign. Dental Press J Orthod 22 (5), 30–38.
Puebla, K., Arcaute, K., Quintana, R., Wicker, R.B., 2012. Eeffects of environmental conditions, aging, and build orientations on the mechanical properties of ASTM type I specimens manufactured via stereolithography. Rapid Prototyp. J. 18, 374–388.
Reymus, M., Stawarczyk, B., 2020. In Vitro Study on the Influence of Postpolymerization and Aging on the Martens Parameters of 3D-Printed Occlusal Devices, the Journal of Prosthetic Dentistry. https://doi.org/10.1016/j.prosdent.2019.12.026. In Press, Corrected Proof.
Reymus, M., Fabritius, R., Keßler, A., Hickel, R., Edelho, D., Stawarczyk, B., 2019a. Fracture load of 3D-printed fixed dental prostheses compared with milled and conventionally fabricated ones: the impact of resin material, build direction, post-curing, and artificial aging—an in vitro study. Clin. Oral Invest. 24, 701–710.
Reymus, M., Lümkemann, N., Stawarczyk, B., 2019b. 3D-printed material for temporary restorations: impact of print layer thickness and post-curing method on degree of conversion. Int. J. Comput. Dent. 22, 231–237.
Ryokawa, H., Miyazaki, Y., Fujishima, A., Miyazaki, T., Maki, K., 2006. The mechanical properties of dental thermoplastic materials in a simulated intraoral environment. Orthod. Waves 65 (2), 64–72.
Ryu, J.H., Kwon, J.S., Jiang, H.B., Cha, J.Y., Kim, K.M., 2018. Effects of thermoforming on the physical and mechanical properties of thermoplastic materials for transparent orthodontic aligners. Korean J Orthod 48 (5), 316–325.
Sangermano, M., Roppolo, I., Chiappone, A., 2018. New horizons in cationic photopolymerization. Polymers 10 (2). https://doi.org/10.3390/polym10020136.
Shim, J.S., Kim, J.-E., Jeong, S.H., Choi, Y.J., Ryu, J.J., 2020. Printing accuracy, mechanical properties, surface characteristics, and microbial adhesion of 3D-printed resins with various printing orientations. J. Prosthet. Dent 124, 468–475.
Stansbury, J., Idacavage, M., 2016. 3D printing with polymers: challenges among expanding options and opportunities. Dental Materials 32 (1), 54–64. https://doi.org/10.1016/j.dental.2015.09.018.
Tanasic, I., Tihacek-Sojic, Lj, Milic Lemic, A., Djuric, M., Mitrovic, N., Milosevic, M., Sedmak, A., 2012. Optical aspect of deformities analysis in the bone-denture complex. Coll. Antropol. 36 1, 173–178. ISSN: 0350-6134.
Tanasic, I., Tihacek-Sojic, Lj, Mitrovic, N., Milic-Lemic, A., Vukadinovic, M., Markovic, A., Milosevic, M., 2015. An attempt to create a standardized (reference) model for experimental investigations on implant's sample. Measurement 72, 37–42. ISSN: 0263-2241.

Uzcategui, A.C., Muralidharan, A., Ferguson, V.L., Bryant, S.J., McLeod, R.R., 2018. Understanding and improving mechanical properties in 3D printed parts using a dual-cure acrylate-based resin for stereolithography. Adv. Eng. Mater. 20, 1800876. https://doi.org/10.1002/adem.201800876.

Wang, X., Jiang, M., Zhou, Z.W., Gou, J.H., Hui, D., 2017. 3D printing of polymer matrix composites: a review and prospective. Compos. B Eng. 110, 442–458, 10.1016/j.compositesb. 2016.11.034.

Zguris, Z., 2016. How Mechanical Properties of Stereolithography 3D Prints Are Affected by UV Curing. Formlabs Inc., Somerville, MA, 2017, (accessed Mar, 7).

Zhang, X.J., He, L., Guo, H.M., Tian, J., Bai, Y.X., Li, S., 2015. Integrated three-dimensional digital assessment of accuracy of anterior tooth movement using clear aligners. Korean J Orthod 45, 275–281. https://doi.org/10.4041/kjod.2015.45.6.275.

Zhang, Z.-C., Li, P.-L., Chu, F.-T., Shen, G., 2019. Influence of the three-dimensional printing technique and printing layer thickness on model accuracy. J. Orofac. Orthop. Fortschr. Kieferorthop 80, 194–204.

Update

Journal of the Mechanical Behavior of Biomedical Materials
Volume 120, Issue , August 2021, Page

DOI: https://doi.org/10.1016/j.jmbbm.2021.104581 journal of the mechanical behavior of biomedical materials 120 (2021) 104581

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: www.elsevier.com/locate/jmbbm

Corrigendum to "The effect of time on mechanical properties of biocompatible photopolymer resins used for fabrication of clear dental aligners" [J. Mech. Behav. Biomed. Mater. Vol 119 (2021) 1–9/104494]

Aleksa Milovanović [a,*], Aleksandar Sedmak [b], Zorana Golubović [b], Ksenija Zelić Mihajlović [c], Aleksandar Žurkić [d], Isaak Trajković [a], Miloš Milošević [a]

[a] *University of Belgrade, Innovation Center of the Faculty of Mechanical Engineering, Belgrade, Serbia*
[b] *University of Belgrade, Faculty of Mechanical Engineering, Belgrade, Serbia*
[c] *University of Belgrade, School of Medicine, Institute of Anatomy, Laboratory for Anthropology Belgrade, Serbia*
[d] *Shimadzu Corp. (Kyoto, Japan), Branch Belgrade, Serbia*

The authors regret that the current Acknowledgments, on page 8 in our paper, is not appropriate for the referencing of the project from which we received financial support, and the Acknowledgements should look like this:

The authors would like to thank the support from European Union's Horizon 2020 research and innovation program (H2020-WIDESPREAD-2018, SIRAMM) under grant agreement No 857124.

The authors would like to apologise for any inconvenience caused.

DOI of original article: https://doi.org/10.1016/j.jmbbm.2021.104494.

* Corresponding author.
E-mail address: amilovanovic@mas.bg.ac.rs (A. Milovanović).

https://doi.org/10.1016/j.jmbbm.2021.104581

Available online 24 May 2021
1751-6161/