Influence of post-polymerization processing on the mechanical characteristics of 3D-printed occlusal splints

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Abstract. Occlusal splints are the most common devices for preventing bruxism and its consequences. Their application significantly reduces the pathologies of the dental and periodontal structures. Apart from the well-known technologies of manufacturing occlusal splints – thermo-vacuum forming, wax elimination and CAD/CAM machining, three-dimensional (3D) printing is a novel promising technique. It makes use of stereolithography, whereby the splint is built layer-by-layer through consecutive polymerization of a liquid photosensitive polymer by ultraviolet laser light. A very important stage in stereolithography following the 3D-printing is the post-polymerization processing. It is a key moment in achieving biocompatibility and the final mechanical parameters – hardness, elasticity modulus, and strength. The process comprises two steps – washing with isopropyl alcohol (IPA) followed by post-curing in a polymerization device with an ultraviolet light source. The aim of the present study is determining the influence of the polymerization device’s light source on the mechanical characteristics of the occlusal splint.

1. Introduction

In the glossary of terms of the American Academy of Orofacial Pain, bruxism is defined as a “total parafunctional daily or nightly activity that includes grinding, gnashing or clenching of the teeth. It takes place in the absence of subjective consciousness and it can be diagnosed by the presence of tooth wear facets which have not resulted of the chewing function”.

The term “bruxism” originates from the Greek expression *brychein odontas* that means grinding the teeth. Bruxism can be classified according to the type and timing of the activity. The etiology of the parafunction is still a controversial subject. It is very difficult to accurately determine the prevalence of bruxism in the general population – the percentages reported range from 6% to 91%. These wide differences can be attributed to the methodology for diagnosis, the characteristics of the studied population and the types of bruxism. No significant differences regarding sex have been found. [1].

Bruxism may result in abnormal tooth wear, mobility, fracture, intrusion, opening of contacts, drifting, erosion, or pulp pathology [2]. Amongst the effects of bruxism on the dentition are pathologic tooth migration, bone alterations, temporomandibular joint disorders (TMD) and pain.

The use of occlusal splints for the management of parafunction has been advocated for many years. Limited evidence is available on the effectiveness of occlusal appliances in stopping completely occlusal attrition. For parafunctional patients, the primary role of the occlusal splint is the protection

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of tooth tissue by preventing tooth-to-tooth contact. The material of the appliance is expendable and easily replaced, whilst enamel and dentine are not [3]. Two main types of splints are known – soft and hard occlusal splints [4]. In terms of manufacturing, the known methods of occlusal splint production are: thermo-vacuum forming (for soft splints only), lost-wax technique [5], CAD/CAM milling [6] and three dimensional (3D) printing.

3D printing has been hailed as a disruptive technology which will change manufacturing. The process has a particular resonance with dentistry, and with advances in 3D imaging and modeling technologies such as intraoral scanning and the relatively long history of the use of CAD/CAM technologies in dentistry, it is becoming of increasing importance [7]. By combining oral or laboratory scanning, computer aided design (CAD) and 3D printing, dental labs can accurately and rapidly produce dental models, temporary crowns, dentures, a range of orthodontic appliances and aligners, surgical guides and occlusal splints [8].

The term 3D printing is generally used to describe a manufacturing approach that builds objects one layer at a time, adding multiple layers to form an object. This process is more correctly described as additive manufacturing, and is also referred to as rapid prototyping. Many different printing technologies exist, each with their own advantages and disadvantages [7]. For the aim of this study, stereolitography (STL, SL) was used.

A stereolithographic 3D printer uses a scanning ultraviolet laser beam to harden (cure) photopolymer resin layer by layer. The parts are manufactured on a building platform located in liquid resin. After a layer is cured, the building platform descends by one layer thickness, and a new layer of resin is spread over the previous one. This procedure is repeated until the parts are completely built [9]. The accuracy of the structures produced varies according to the geometries being replicated, the method of manufacture, and the materials being used. SLA can fabricate structures with a layer thickness of 25 µm up to 100 µm [10]. The x/y axes exposure of each distinct layer can be modified as the z axis incrementally evolves in the building process. In the SLA approach, the depth of cure, which ultimately determines the z axis resolution, is controlled by the photo-initiator and the irradiant exposure conditions (wavelength, power and exposure time/velocity). Still, significant amounts of residual initiator remain and this permits parts to be post-cured in a UV oven to promote completion of the curing process [11]. During post-curing, exposure to ultraviolet (UV) light triggers the formation of additional chemical bonds within a printed part, making the material stronger and stiffer. The photo-initiator in the material requires a co-initiator for an efficient polymerization process to occur. A co-initiator is a separate compound that does not absorb light but interacts with an activated photo-initiator to produce a reactive species. The efficiency of any given photo-initiator is governed by a number of factors. For high absorptivity to be achieved, there obviously must be a relatively good match between the absorption spectrum of the photo-initiator and the emission spectrum of the light source. Curing units generally transmit in the 350 nm to 540 nm visible region. The processes of photo-initiation and growth of the polymer chains are thoroughly discussed by Stansbury [12]. Heat accelerates this process and enables a more complete bond formation for a fast and highly effective post-cure resulting in optimal material properties.

Information regarding the influence of post polymerization processing over the mechanical features of 3D-printed occlusal splints in the literature is scarce. The aim of the study is determining the influence of the polymerization device’s light source on the mechanical characteristics of the occlusal splint.

2. Material and methods

2.1. Material

The material used for production of occlusal splints was Dental LT Clear (Formlabs Inc., USA) photopolymer resin, CE-certified as biocompatible Class IIA [13]. It is a monomer based on acrylic esters containing methacrylic oligomer > 70 % w/w, glycol methacrylate < 20 % w/w, pentamethylpiperidyl sebacate < 5 % w/w (co-photo-initiator) and phosphine oxide < 2.5 % w/w (photo-initiator).
Post processing for this type of resin is crucial for the mechanical properties and biocompatibility of the final product.

2.2. Digital workflow
The following steps are part of the process of 3D printing, as illustrated in figure 1.

1/ Acquiring patient’s data – scanning the dentition with an intraoral scanner or scanning physical plaster models with a desktop optical scanner.
2/ Digital design in CAD software – the splint was designed using exocad DentalCAD v. 2.2 (exocad GmbH, Germany). Open STL file export is needed.
3/ Preparation for printing – the design file was imported into the printer’s software PreForm v. 2.19.1 (Formlabs Inc., USA). A few parameters needed to be set before printing starts – layer thickness, orientation of the printed object, layout onto the printer platform, adding supporting structures [14].
4/ 3D printing with the Form 2 SLA printer (Formlabs Inc., USA).
5/ Post polymerization processing consisted in two consecutive steps:
   5.1/ rinsing the parts with 99.5% isopropyl alcohol (IPA) for five min in Form Wash (Formlabs Inc., USA), which removes any uncured liquid resin before post-curing. The device’s magnetically coupled impeller agitates IPA to flow around every nook and cranny of the printed object. Manual rinsing is an option, but if left in the IPA for longer than 10 minutes, the parts can warp. Form Wash automatically raises the parts out of the alcohol after the wash cycle is over, so they can dry well before post-curing. A minimum of 30 minutes in the open at room temperature is required for the IPA to completely dry.
   5.2/ post-curing with UV light for 20 minutes at 80 °C in the Form Cure (Formlabs Inc., USA) polymerization unit.
6/ Removal of supporting structures and polishing.
The flexural strength, elasticity modulus and hardness were examined on test specimens that went through different protocols of post polymerization processing in terms of wavelength, power of the light source and temperature. The following polymerization devices with the following specifications are used (table 1.).
- Light curing device FormCure (Formlabs Inc., USA)
- Laboratory curing device bre.Lux 2 (Bredent, Germany)
- Ultraviolet curing lamp Sanitas (Hans Dinslage GmbH, Germany)

| Light source | Number of sources | Heating | Power (W) | Wavelength (nm) | Rotary plate |
|--------------|-------------------|---------|-----------|-----------------|--------------|
| Form Cure    | LED               | max 80°C| 39        | 405             | +            |
| bre.Lux 2    | LED               | max 65°C| 130       | 370-500         | +            |
| Sanitas      | UV-A              | -       | 36        | 365             | -            |

2.3. Test specimens
Test specimens for both methods were digitally designed in Blender v 2.73 and produced following the described workflow. Regular polishing protocol for resin oral appliances was executed. For Vickers hardness test, the size of the specimens was diameter of 20 mm and height of 3 mm, in
compliance with ISO 6705-1 [15]. The specimens were divided into 3 groups. In each group 10 indents were made (n = 8, N = 24).

For flexural strength testing, the specimens produced were of size $3.3 \times 0.64 \text{ mm}^3$, according to ISO 20795-1 [16]; 24 specimens were divided into 3 groups (n = 8, N = 24).

Every group underwent a different post-curing protocol: Group 1 – with Form Cure (light-emitting diode (LED), wavelength 405 nm, heating at 80 °C for 20 min), Group 2 – with bre.Lux 2 (LED, wavelength range between 370 – 500 nm, heating at 45 °C for 20 min) and Group 3 – with Sanitas (UV-A lamp, wavelength 365 nm, no heating and no rotary plate for 20 min). Group 2 was further studied 24 hour after post-curing.

2.4. Vickers hardness test

The hardness was measured using a Zwick Roell ZHVµ Micro Vickers hardness tester (Indentec Hardness Testing Machines Ltd., UK). It uses a square-based diamond pyramid as indenter [17] with an angle of 136 ° between opposite faces. A load of 0.5 kg (HV0.5) was applied for (dwell time) 10 s. Each indentation’s two diagonals were measured under 20× magnification by making the two lines interact at two diagonally opposite corners (figure 2 (a), (b) and (c) horizontally and then vertically.

![Figure 2. Vickers hardness indentations on specimens with different post-cure. (a) – Form Cure, (b) – bre.Lux 2 and (c) – Sanitas.](image)

2.5. Flexural strength and elasticity modulus

The flexural strength was tested by a MultiTest 2.5-i Tensile and Compression Test System (Mecmesin Ltd., UK) and the elasticity modulus was calculated by using the system’s Emperor™ software. For the test, an additional specimen group was added (Group 2 after 24 h) due to the hypothesis that the material will behave differently immediately after curing by bre.Lux 2 and 24 hours after the cure. This was necessary because of the significantly larger number of light sources and higher power of the polymerization device.

2.6. Statistical analysis

Paired-samples T Test in SPSS v 19.0.0 (IBM Inc., USA) was used to determine the mean difference between each two sets of observations. The significance level was accepted to be $p < 0.05$. For Vickers hardness test, three pairs of parameter results were compared, and for flexural strength and elasticity modulus, 12 pairs.

3. Results

After analyzing the results from Vickers hardness testing (table 2), no effect on the hardness by the different post-polymerization protocols was discovered. Between Group 1 and 2, $p = 0.095$, between Group 1 and 3, $p = 0.732$, between Group 2 and 3, $p = 0.227$.

The maximum force $F_{\text{max}}$ (N) applied in the flexural strength test showed effect ($p < 0.001$) only when comparing the parameter in Group 1 and 3, Group 2 and 3, Group 2 after 24 h and 3. No other statistical differences occurred.
4. Discussion
In this study, the different methods of post-curing in terms of wavelength and temperature proved that the larger wavelength in the UV spectrum and the higher temperatures resulted in better properties. The differences in the protocols applied did not affect the hardness of the material, as it depends not only on the structure, but on the finishing and polishing of the surface as well. The material presented improved strength qualities 24 hours after post-curing in comparison with the tests ran immediately after post-curing.

The optimal mechanical properties were obtained when post-curing was applied with the FormCure polymerization device with a wavelength of 405 nm, heating at 80 °C and continuous rotation of the object in the polymerization chamber. Adhering to these parameters will ensure the final curing of the material. The use of wavelengths below 400 nm and the lack of additional heating resulted in a significantly more fragile material with an elasticity modulus lower by approximately 1000 MPa.

In cases of centric form of bruxism, where sustained tooth clenching forces on a smaller surface are immense (especially in the distal area of the dentition), the strength of the material and its elasticity are of paramount importance.

5. Conclusions
The correct post-curing method is of crucial importance for the material to ensure sufficient mechanical properties. Heating the material during the process proved its significance and, along with the exact wavelength of 405 nm, is mandatory for the production of 3D-printed occlusal splints. Further research on the impact of post-curing on biocompatibility in vivo is possible.

Table 2. Vickers hardness test results (HV0.5).

| Sample | Indent 1 | Indent 2 | Indent 3 | Indent 4 | Indent 5 | Indent 6 | Indent 7 | Indent 8 | Mean | SD  |
|--------|----------|----------|----------|----------|----------|----------|----------|----------|------|-----|
| Group 1| 19       | 20       | 20       | 19       | 19       | 19       | 20       | 19       | 19.38| 0.518|
| Group 2| 18       | 21       | 21       | 20       | 20       | 21       | 20       | 19       | 20   | 1.069|
| Group 3| 19       | 20       | 20       | 19       | 19       | 20       | 19       | 19       | 19.5 | 0.756|

The results from the flexural strength (FS) test and the calculated elasticity modulus (E) in MPa, along with the mean and the standard deviation (SD) from descriptive statistics are given in Table 3. When examining FS and E between each pair of groups, the paired samples T test showed significance in each pair of specimens (p < 0.05). In eight of the twelve pairs compared altogether, the p-value was p < 0.001, which points to the high significance of the test results.

Table 3. Flexural strength and elasticity modulus test results.

| Sample | Group 1 | Group 2 | Group 2 after 24 h | Group 3 |
|--------|---------|---------|--------------------|--------|
|        | FS max  | E       | FS max  | E       | FS max  | E       | FS max  | E       |
| 1      | 209.3   | 129.44  | 3422.41 | 203.9   | 118.02  | 3073.68 | 203.3   | 125.75  | 3553.85 | 132.3   | 75.83   | 2450.67 |
| 2      | 207.2   | 126.87  | 3431.63 | 211.7   | 119.17  | 3102.15 | 209.9   | 128.51  | 3388.21 | 127.4   | 78.02   | 2606.57 |
| 3      | 208.3   | 127.50  | 3431.58 | 210.0   | 125.60  | 3198.25 | 215.0   | 124.40  | 3073.72 | 138.9   | 84.19   | 2709.80 |
| 4      | 207.1   | 126.78  | 3431.60 | 212.7   | 124.83  | 3153.44 | 209.7   | 128.42  | 3388.14 | 139.8   | 79.18   | 2664.51 |
| 5      | 207.8   | 127.23  | 3474.98 | 205.6   | 120.9   | 3193.32 | 209.7   | 120.18  | 3201.22 | 130.9   | 80.11   | 2693.46 |
| 6      | 209.0   | 127.96  | 3431.58 | 211.2   | 118.19  | 3190.84 | 209.5   | 125.45  | 3321.03 | 142.8   | 82.62   | 2594.95 |
| 7      | 213.9   | 130.97  | 3518.33 | 211.6   | 122.4   | 3198.54 | 208.9   | 124.52  | 3189.25 | 137.8   | 78.98   | 2450.65 |
| 8      | 206.6   | 126.50  | 3431.51 | 209.8   | 123.1   | 3120.85 | 209.3   | 124.84  | 3354.12 | 140.9   | 82.94   | 2623.72 |
| Mean   | 208.6   | 127.91  | 3446.70 | 209.6   | 121.5   | 3153.9  | 209.4   | 125.3   | 3308.69 | 136.4   | 80.23   | 2599.29 |
| SD     | 2.32    | 1.54    | 33.01   | 3.15    | 2.93    | 49.4    | 3.15    | 2.62    | 149.28  | 5.46    | 2.82    | 100.13  |
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