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Mechanical properties of 3D-printed occlusal splints after different isopropanol washing times

Syventävien opintojen kirjallinen työ

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Background: The use of 3D printing for manufacturing occlusal splints is convenient; however, further studies are needed to optimize their material properties. The aim of this study was to evaluate the effect of different isopropanol (IPA) washing times on the properties of 3D-printed occlusal splints.

Materials and methods: A total of 132 bar-shaped specimens printed using Formlabs Dental LT Clear V1 and V2 resins were investigated. The printed specimens from each resin were divided into three groups according to IPA washing time: 5, 20, and 60 minutes (n = 22 per group). Half of the specimens from each group were subsequently water-aged. Flexural strength, flexural modulus, water sorption, and water solubility were measured. In addition, surface characteristics were evaluated using a non-contact 3D optical profilometer. Data were statistically analyzed using ANOVA and Tukey's HSD test.

Results: The results showed that longer IPA washing times generally resulted in lower flexural strength values, except in the water-aged V2 group. Flexural modulus was lower in the non-water-aged groups. Longer IPA washing times also increased water sorption. Furthermore, specimens in the water-aged V1 group were more prone to cracking and fracture after prolonged IPA washing. In contrast, short IPA washing times resulted in residual unpolymerised resin on the specimen surfaces. Based on the overall findings, a washing time of 20 minutes appeared to provide the most balanced performance among the tested groups.

Conclusion: The duration of IPA washing had a significant effect on the performance of 3D-printed occlusal splint resins. Excessive washing negatively affected several material properties. Therefore, optimization of the IPA washing protocol is essential to achieve adequate cleaning while maintaining the material performance.

Keywords: 3D-printing, post-treatment, occlusal splint, isopropanol, mechanical properties

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1. INTRODUCTION

Occlusal splints are used in dentistry for the treatment of temporomandibular disorders (TMD) [1]. Temporomandibular disorders include pain and dysfunctions of the muscles of the face and the temporomandibular joints [2]. Occlusal splints seem to be effective in treating the symptoms of TMD such as myofascial pain and preventing tooth wear caused grinding teeth especially during sleep. However occlusal splints do not cure the root cause of TMD [3]. Myofascial pain is relieved through the reduction of masticatory muscle activity when using an occlusal splint [4].

Occlusal splints are often made from polymethyl methacrylate (PMMA). The usual way of fabricating them with this material is by taking a mold of the teeth with alginate. Then a model of the teeth is made from dental plaster. Finally the PMMA is applied on the plaster model and it is cured in one of various ways such as cold polymerisation [5,6]. Making occlusal splints has become much more convenient recently with the technological progress in digital impressions with computer-aided design/computer-aided manufacturing (CAD/CAM) and 3D-printers. In CAD/CAM a digital impression of the patients teeth is taken with a scanner either intraorally or indirectly from a plaster model and a dental device can be made with a computer program. The occlusal splint will then be fabricated using a technic called milling. In milling the shape of the occlusal splint is made by drilling material and this results in a lot of material waste. Lastly it is now possible to use 3D-printers to make occlusal splints. Using this method the patients mouth is first scanned and then a 3D-printer is used to construct the occlusal splint. 3D-printing has a number of advantages. It is more comfortable for the patient, it is less time consuming, it will not have distortion that come over time to physical impressions, it has less material waste and it can be cheaper in the long run. The downside of 3D-printing is that the mechanical properties of the materials are weaker compared to PMMA and the scanners can still be inaccurate [7-9].

There are various mechanisms for 3D printers. Two of the most common mechanisms for 3D-printers are stereolithography (SLA) and digital light processing (DLP). In SLA a laser cures the top layer of the liquid resin whereas in DLP a light projector is used. After printing the product must be cleaned so that there is no unpolymerized resin remaining on the surface of the product. The materials used in this study are recommended to be rinsed with isopropanol (IPA) for 20 minutes by the manufacturer. Finally the product should be post-cured to increase the conversion rate of the polymers.

There is still a very limited amount of literature on the effects of post rinsing when fabricating a dental device with a 3D-printer. Thus the aim of this study was to investigate the effect of the isopropanol washing time on the properties of 3D-printed occlusal splints. The null hypothesis of isopropanol wash time having no effect on the properties was tested.

2. MATERIALS AND METHODS

2.1 Flexural strength and flexural modulus

In total 132 bar-shaped specimens were printed with 3D-printer that uses SLA (Formlabs Form 3B+). The material for half of the specimens was Formlabs Dental LT clear V1(n=66) and for the other half Formlabs Dental LT clear V2(n=66). The dimensions of the specimens were 3x10x60mm. After printing excess resin is washed with isopropanol in an ultrasonic cleaning unit (Quantrex 90, L&R Ultrasonics, Kearny, NJ, USA). Both materials were divided into three groups(n=22) with different isopropanol washing times. The aforementioned washing times were 5 minutes, 20 minutes and 60 minutes. When IPA-wash was finished the specimens were left to dry for 30 minutes before post curing. Post curing was conducted with a post curing unit (Otoflash G171, BEGO GmbH&Co, Bremen, Germany). For Dental LT clear V1 the post curing was done for 20 minutes in 80 degrees celsius. For Dental LT clear V2 the post curing was done for 60 minutes in 60 degrees celsius. All the different washing time groups in both materials were yet again divided to two groups(n=11). The other group was boiled in 100 degree deionised distilled water for 16 hours before testing while the dry group was left in the air atmosphere without any additional handling.

The length of the specimens were measured before testing. The specimens were measured for their flexural strength (MPa) and modulus (GPa). The measurements were conducted with a three point bending test by a universal testing machine (Model LRX; Lloyds Instruments Ltd., Hampshire, UK). The span between the two supporting edges was 50 mm and the crosshead speed was 5 mm/min. The specimens were bent for 12 mm or until they were broken into pieces. The amount of broken specimens were documented.

Flexural strength (σ) can be calculated with the formula below where F is the value for force at its highest point. L is the distance between the supporting edges (50mm). b stands for width of the specimen ($\approx 10\text{mm}$) and h for height of the specimen ($\approx 3\text{mm}$)

$$\sigma = \frac{3F_{\max}L}{2bh^2}$$

Flexural modulus (E) can be calculated with the formula below where F is the force at the chosen point D. L is the distance between the supporting edges (50mm). b stands for width of the specimen ($\approx 10\text{mm}$) and h for height of the specimen ($\approx 3\text{mm}$)

$$E = \frac{3F_1L^3}{4bh^3D_1}$$

2.2 Water solubility and water sorption

Six specimens from each group of both materials were measured for water sorption and water solubility. The specimens were made dry by inserting them into a vacuum desiccator containing freshly dried silica at 37 ± 1 °C for 24h. The mass of the specimens were first measured with a digital analytical balance (XS105; Mettler Toledo, Greifensee, Switzerland) with an accuracy of 0.1mg. Then the specimens were put to 100 degree distilled water for 16 hours and the mass was measured immediately after aging them in the water. After that the specimens were left to dry and their weight was measured regularly until there was no more change in the weight.

$$\text{Water sorption} = 100 \times (M_2 - M_3)/M_1$$

$$\text{Water solubility} = 100 \times (M_1 - M_3)/M_1.$$

M1 is the initial mass of the specimens. M2 is the weight immediately after water immersion. M3 is the stable weight after drying in 55 degree air.

2.3 Three-Dimensional Microlayer structure

One specimen was randomly selected from each subgroup and covered with gold by using a sputtering device for the inspection of its surface on a microscopical level. The inspection of the surface was made by using a scanning electron microscope (SEM) (JSM-5500, JEOL Ltd., Tokyo, Japan). The specimens were inspected at four different spots with 50x, 250x, 500x, 1000x, 2000x and 4000x magnification. Additionally the specimens were inspected with a 3D optical profilometer (ContourGT-I, Bruker Nano, Inc., Tucson, AZ, USA). With this we could measure the dimensions of the micro layer structure.

2.4 Statistical analysis

A one-way ANOVA was performed to analyse the statistical significance of the IPA wash time for flexural strength, flexural modulus, length of the specimens, water sorption and water solubility using a statistical software (JMP Student Edition 19, JMP Statistical Discovery LLC, Cary, NC, USA). The amount of broken specimens and crack formation on the specimens were not statistically analysed. A *p* value of less than 0.05 was considered statistically significant.

3. RESULTS

The *p* values for the statistical analyses are shown in the table below

Table 1. Statistical analysis of flexural properties, length of the specimens, water sorption and water solubility

	Flexural strength	Flexural modulus	Length	Water sorption	Water solubility
V1	<0.0001	0.0198	0.8094	0.092	<0.0001
V2	<0.0001	0.0341	0.5474	0.0348	0.0012
V1 aged in water	0.0156	0.0992	0.2707		
V2 aged in water	0.3709	0.1128	0.1955		

According to the results the flexural strength is clearly lower with longer IPA wash time in the groups that were not aged in water. Similar result was also seen in the water aged V1 group. The water aged V2 group was not affected by the IPA wash time. Water aging also decreased the flexural strength in both V1 and V2 materials. Flexural modulus showed a decrease in the non-water aged groups with a longer IPA wash time but no statistically significant difference was seen in the water aged groups. However flexural modulus also decreased in both materials when aged in water. For specimen length there was no correlation with the IPA wash time but water aged specimens were slightly longer in both groups. The difference in water sorption was not significant in V1 group but slightly higher water sorption was found in the V2 group when the IPA wash time got longer. Water solubility was clearly higher with a longer IPA wash time in both V1 and V2 groups. The mean values and standard deviation for flexural strength, flexural modulus, length of the specimens, water sorption and water solubility are shown in the tables (2-5)

Table 2. Flexural strength results of the tested resins before and after aging (MPa)

	5min	20min	60min
V1	130±5.65	123±4.34	118±32.6
V2	80.8±3.04	79.1±3.50	72.9±5.06
V1 aged in water	68.3±5.40	66.9±5.53	55.9±14.0
V2 aged in water	54.9±4.36	54.1±4.96	56.4±1.42

Table 3. Flexural modulus results of the tested resins before and after aging (GPa)

	5min	20min	60min
V1	5.44±0.532	5.26±0.436	4.88±0.312
V2	3.22±0.183	3.19±0.204	2.99±0.242
V1 aged in water	2.97±0.335	3.01±0.475	3.40±0.600
V2 aged in water	2.15±0.207	2.14±0.220	2.29±0.072

Table 4. Lengths of the specimens before and after aging (mm)

	5min	20min	60min
V1	9.82±0.04	9.82±0.05	9.84±0.07
V2	9.69±0.09	9.71±0.09	9.67±0.07
V1 aged in water	9.99±0.10	9.94±0.06	9.99±0.08
V2 aged in water	9.78±0.15	9.66±0.21	9.75±0.06

Table 5. Values for water sorption in the left and values for water solubility in the right (%)

Water sorption

Water solubility

	5min	20min	60min	5min	20min	60min
V1	5.158±0.021	5.132±0.021	5.166±0.033	0.955±0.019	0.990±0.064	1.150±0.020
V2	3.039±0.011	3.044±0.007	3.06±0.014	0.476±0.010	0.483±0.008	0.499±0.007

In the Table 6 the amount of broken specimens during the three point bending tests is displayed.

Table 5. The amount of broken specimens in the three point bending test (%)

	5min	20min	60min
V1	67	55	64
V2	9	9	18
V1 aged in water	40	60	90
V2 aged in water	18	0	0

In the water aged V1 group the amount of broken samples seems to be higher when the IPA wash time was longer. This was not the case in the other groups. In the water aged V1 group even if the samples didn't break there was crack formation in the specimens and the rate of cracks was higher with longer IPA wash times. Some cracks were also observed in the water aged V2 group with 60 minute IPA washing time.

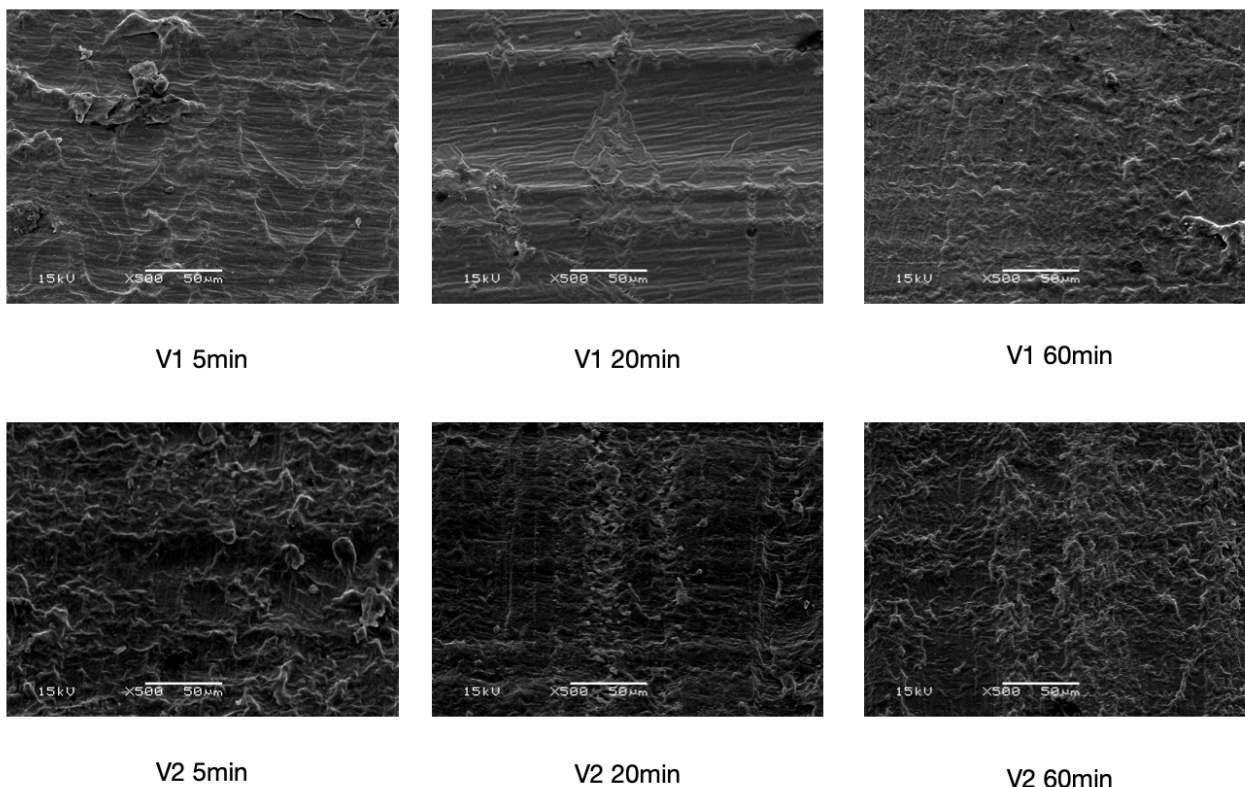


Figure 1. The images taken with the scanning electron microscope (SEM) with 500x magnification.

The images taken with SEM using 500x magnification are seen in Figure 1. In both groups it can be noticed that 5 minute IPA washing time still leaves some irregularities on the surface. 20 minute IPA washing time seems to give the smoothest result while prolonged IPA washing in the 60 minute samples seems to leave a rugged surface.

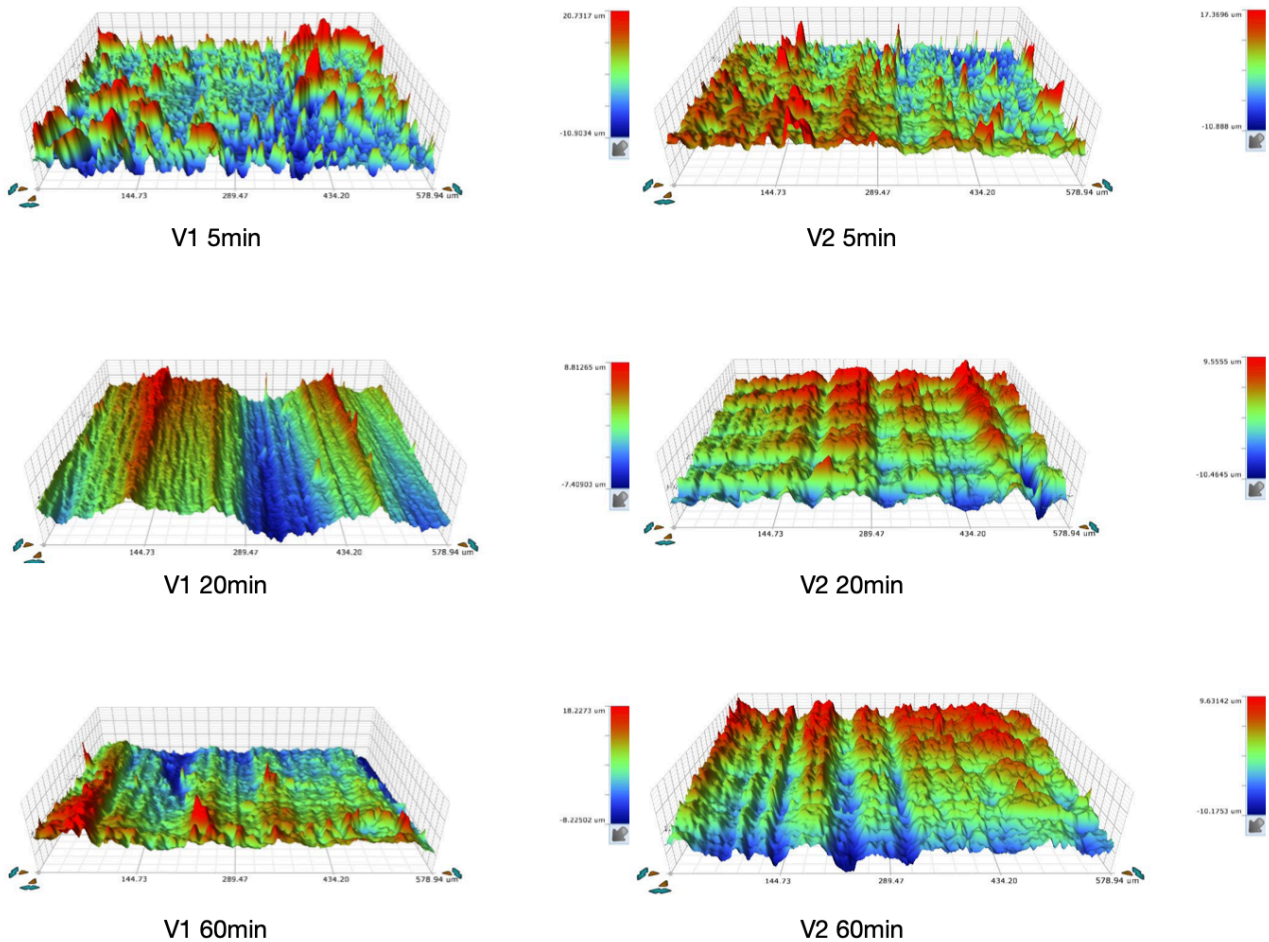


Figure 2. 3D images of the surface taken with the 3D optical profilometer. Note that the scale is slightly different for every image.

The 3D images of the surface taken with the optical profilometer are seen in Figure 2. In the 5 minute groups a significant amount of irregularities on the surface can be seen. The 20 minute groups show the smoothest result while the 60 minute groups have a slightly more rugged appearance.

4. DISCUSSION

In this study the effects of IPA wash time on the properties of 3D-printed occlusal splints were investigated. These properties included flexural strength, flexural modulus, water sorption, water solubility as well as the three dimensional consistency of the surface of the occlusal splints. The results indicate that the null hypothesis of there being no effect with different IPA wash times can be rejected. The decline in the properties also depends on the material of the occlusal splint.

The post-treatment of 3D-printed materials plays a big role on the properties. As found in this study the physical properties can change. Similar results have been found in other studies [10]. One aspect that should be considered when making materials for medical purposes is cytotoxicity. The exact formulas of the resins used in this study are not revealed but the manufacturer reveals that the materials contain Bisphenol A dimetachrylate, urethane metachrylate, metachrylate monomers and photoinitiators. According to a study some of the components in the resins could be cytotoxic [11]. It was also found that proper post-treatment of the 3D-printed objects ameliorated the toxicity of the materials [12]. Thus the effects of the material on the body could be unfavourable if the IPA washing time is not long enough.

IPA itself is cytotoxic [13]. When 3D-printing the IPA is inside a post rinsing unit. Therefore exposure to vaporising IPA is minimised but there is still exposure especially when inserting and taking the product out of the unit. For this reason other ways for post rinsing could be investigated. For example ethanol and butyl glycol can be used as an alternative for IPA in post rinsing [14].

In this study an SLA type 3D-printer was used. For post-curing a light curing unit was used. Using a different printer type and post-cure method can have an effect on the properties of the occlusal splints in addition to the IPA wash time. According to a study the type of post-cure was found to affect the flexural strength and flexural modulus and water sorption among other properties [15]. In the same study the type of the printer on the other hand can lead to a difference in the smoothness of the surface, water sorption and water solubility. Therefore the effect of IPA washing in combination with different printer types and post-curing methods should be further investigated.

Half of the specimens in this study were aged in boiling water. The purpose of this is to stimulate the conditions in the mouth when the occlusal splint is in actual use. Aging in water is a standard procedure when investigating materials for dental applications [16]. In this study aging in water led to a clear decrease in flexural strength and flexural modulus. Also the length of the specimens increased which would indicate to water sorption. With the V1 material significantly more broken specimens were observed in the water aged group when the IPA wash time was longer. This means that the occlusal splints would get especially weak when in use.

According to the manufacturer of the 3D-printer and the resins of this study, the recommended time for IPA wash time was 20 minutes which also seems to be the optimal according to this study. The longer the IPA wash time is the weaker the resulting occlusal splints will be depending on the material. Therefore excessive washing should be avoided. However not washing the material enough will lead to a large amount of unwanted unpolymerized resin on the splints.

5. CONCLUSION

We can conclude from the results that excessive washing of 3D-printed occlusal splints with isopropanol will lead to weaker occlusal splints that are more prone to cracking and breaking. The splints will also be more water soluble and the surface of the splint will get slightly more rugged with a very long IPA washing time. On the other hand washing too little will lead to having remains of unpolymerized resin on the surface of the splints which leads to an irregular appearance when inspected on the micro level.

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