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The Use of Light-Cured Resin as an Alternative Method of Occlusal Splints Manufacturing – *In Vitro* Study

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Abstract

Background. Temporomandibular disorders are very common nowadays. One of the methods to treat these problems is occlusal splint therapy. Modern materials should be introduced to this treatment.

Objectives. The aim of this paper was to evaluate the properties of light-activated urethane dimethacrylate and the quality of the bonds it creates with thermoforming foils.

Material and Methods. Thermoforming foils were covered with light-cured resin. A bond was formed between the materials using an adhesive. A coating lacquer was used on the resin as a final preparatory step. Three laboratory tests were run: dye penetrant inspection, a Vickers microhardness test and a linear polymerization shrinkage test. The materials were layered and then cured with a polymerizing lamp emitting light of a wavelength of 400 Nm, according to the manufacturer's instructions. All the occlusal splints were fitted to upper dental arch. The devices had been made in an articulator on specially prepared gypsum models. The results were analyzed statistically using a one-sided binomial test, Spearman's rank-order correlation coefficient and the Friedman ANOVA ($p = 0.05$).

Results. In the dye penetrant inspection, only one sample out of sixty showed the effects of color penetration to the adhesive connection. The dye only penetrated the layer of lacquer coating the resin. The average value of the Vickers microhardness test with a load of $F = 50$ g applied to the material surface for 30 s was $HV_{0.05} = 7.43$ N/mm². The average linear shrinkage of the resin observed after polymerization was 1.175%.

Conclusions. Light-cured resin and an adhesive connection between the resin and thermoforming foil do not show susceptibility even to strong dye. The maximum polymerization shrinkage occurs immediately after curing. The light-cured resin that was tested seems to be a good alternative method for occlusal splints manufacturing (*Adv Clin Exp Med* 2014, 23, 6, 977–985).

Key words: occlusal splints, light-cured resin, dye penetrant inspection, Vickers microhardness, polymerization shrinkage.

According to De Kanter et al., the incidence of temporomandibular disorders ranges from 6% to 93% [1]. Various studies confirm that women suffer from temporomandibular disorders more often than men [2, 3]. Those disorders may be related to soft tissues, temporomandibular joints, stomatognathic system muscles or component parts of the nervous system.

One of the most common methods to treat temporomandibular disorders is occlusal splint therapy [4–8]. The first written report referring to that type of treatment was published by Karolyi in 1901 [9]. Since that time many different oral appliances for treating temporomandibular disorders have been introduced, with constantly changing shapes and concepts.

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Heat-cured polymethyl methacrylate is a material that has been commonly used in manufacturing occlusal splints. It was introduced by Walter Wright in 1937 as a material to manufacture denture bases [10]. It has many useful properties, such as adequate hardness, a low shrinkage level, chemical stability, resistance to abrasion, easy handling and processing. These features make it an excellent material for use in treating functional disorders of the stomatognathic system. It should be mentioned that heat-cured methacrylate is cheaper than light-cured resin. This makes it more accessible and improves the cost effectiveness of the treatment. However, despite its good properties, it's not a perfect material. The main problem is that some people have a hypersensitivity to the monomer that appears in acrylate materials, which can affect both dental technicians and patients, causing skin allergies and respiratory allergies [11]. Acrylate materials also have strong, pungent odor that disappears right after polymerization.

Advances in science have permitted researchers to start investigating materials that would provide better and more biocompatible clinical solutions. Good examples of such materials are light-cured resins and thermoformable materials, widely used in modern dentistry, e.g. for forming occlusal splints used in treating temporomandibular disorders, occlusal interferences and in orthognathic surgery [12–15].

The aim of this study was the *in vitro* evaluation of light-cured urethane dimethacrylate Lightdon Splint resin (Dreve Dentamid GmbH, Unna, Germany) used in combination with a thermoformable foil to construct occlusal splints. The materials were tested for dye penetration between the two materials, microhardness and linear polymerization shrinkage.

Material and Methods

Light-cured Lightdon Splint resin was applied on 1 mm thick Erkodur thermoformable foil (Erkodent GmbH, Pfalzgrafenweiler, Germany). The thermoforming process was used to mold them in a Erkoform 3D vacuum unit (Erkodent GmbH, Pfalzgrafenweiler, Germany). The chemical composition of Lightdon Splint resin is tetrahydrofurfuryl-2-methacrylat (25–50%), polyurethaneacrylate (10–25%), poly-i-buthylmethacrylat (2.5–10%) and photo initiator (0.5–0.7%) [16]. Lightdon Bonding adhesive (Dreve Dentamid GmbH, Unna, Germany) was used to bind the resin to the thermoformable foils, allowing the occlusal surface to be prepared with accurate canine guidance. After polymerization, finishing with milling burs and polishing the last surface, the manufacturer advises covering the resin surface with Plaquit lacquer (Dreve Dentamid GmbH) to protect the resin from deposition of dental plaque [17].

The light-curing lamp used in the study (Individo Light Box, Voco GmbH, Cuxhaven, Germany) had a power of 36 W and emitted light of a 400 Nm wavelength. The exposure times for particular components were as follows:

- Lightdon Bonding adhesive: 1 min;
- Lightdon Splint resin: 5 min;
- Plaquit lacquer: 4 min.

The materials were applied and cured according to the manufacturers' recommendations.

All the occlusal splints were attached to upper dental arche (Fig. 1). The appliances had been prepared by the same person on gypsum models in a Protar 3 articulator (KaVo Dental GmbH, Biberach, Germany). The final wet polishing was done in a Rotopol 22 polisher (Struers, Rodovre, Denmark) using waterproof silicon carbide paper disks (Struers) with decreasing grain sizes (FEPA 320, 2400 and 400).

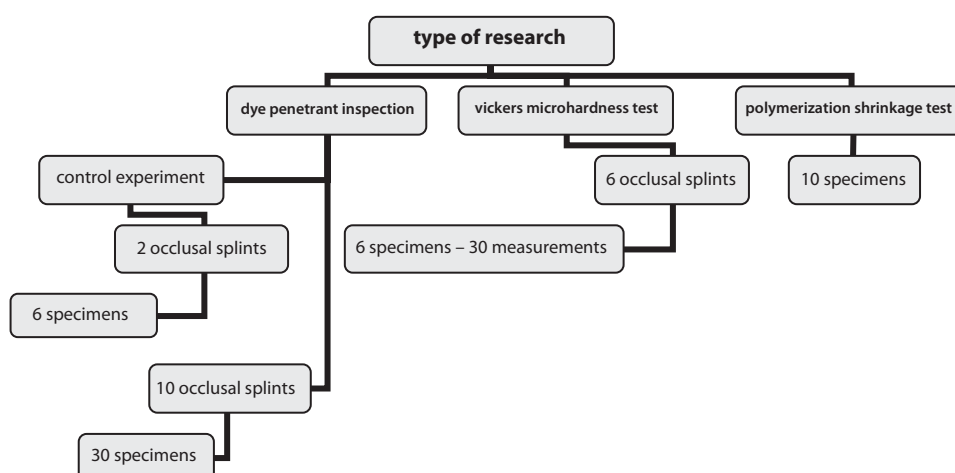


Fig. 1. The flowchart presents the number of occlusal splints and specimens used in the study

Three laboratory tests were designed and run: dye penetrant inspection, a Vickers microhardness test and a linear polymerization shrinkage test.

Dye Penetrant Inspection

This test was designed to test the susceptibility to moisture penetration of the light-cured resin, lacquer and the bonding material between the resin and the thermoformable foils. To achieve this, 10 similar occlusal splints were made. The materials were layered and exposed to light according to manufacturers' recommendations. Then each appliance was divided into 3 parts by cutting it between the maxillary canine and the first premolar on both left and right sides (Fig. 2). After cutting, the new surfaces were polished, coated with Plaquit lacquer and exposed to light to prevent any dye penetration from this side. A total of 30 such specimens were prepared. The prepared specimens were subjected to thermocycling (2500 cycles in distilled water) using a Festo FPC 101 Step Controller (Festo AG & Co. KG, Esslingen, Germany). Each cycle comprised 27 s of immersion time at temperatures of $5^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $55^{\circ}\text{C} \pm 2^{\circ}\text{C}$, and 15 s for taking the samples to another bath.

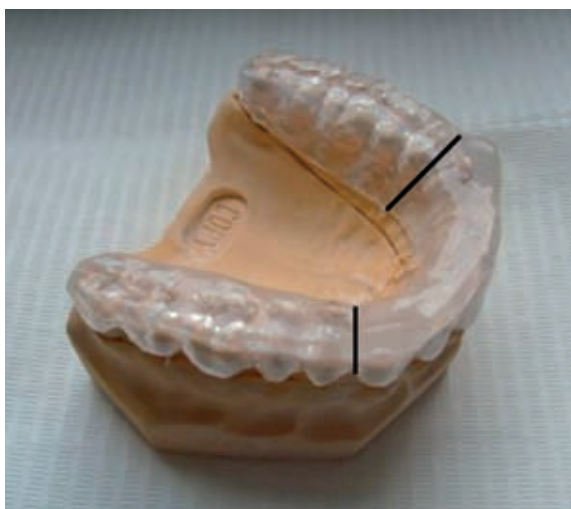


Fig. 2. An example of an occlusal splint ready for dye penetrant inspection. Erkodur base foil (width 1 mm) is covered with Lightdon Bonding adhesive, Lightdon Splint light-cured resin and Plaquit lacquer. The cutting lines were marked in black, dividing every splint into 3 samples

The specimens were then immersed for 24 h in a 2% solution of methylene blue at 37°C . After that time, the samples were rinsed with water and allowed to dry. Subsequently, each preparation was cut transversely in the middle and polished. The penetration of the dye was evaluated on the surface that was formed after the cut from the vestibular

and lingual sides. The samples were examined under a Leica MZ12 microscope (Meyer Instruments, Houston, Texas, USA) at a magnification of $\times 32$.

As a control, another 6 specimens, not coated with Plaquit lacquer, were prepared, but in this case the Lightdon Bonding adhesive was replaced with an alginate isolator. Further procedures were the same as described above.

Vickers Microhardness Test

The part of the study was designed to evaluate the microhardness of the resin as an essential feature of materials used in the prosthetic rehabilitation of patients with greater chewing forces. The Vickers method uses a square-based diamond pyramid (called an indenter) with an apical angle of 136° between the surfaces to evaluate the hardness. The indenter is pressed into the surface of the test piece using a prescribed force (F) and time. The measurement is usually performed at an ambient temperature between 10°C to 35°C [18]. The Vickers hardness (HV) number is then determined by the ratio F/A where F is the force applied to the diamond and A is the surface area of the resulting indentation, according to following equation:

$$\text{HV} = 0.102 \times F/A = 0.1891 F/d^2 \text{ [N/mm}^2\text{]}$$

where

HV: Vickers hardness,

F : load in Newtons [N],

0.1891: Vickers constant,

$d = d_1 + d_2/2$: the average value of the imprint diagonal [mm],

d_1, d_2 : diagonal values [mm].

The test was carried out 24 h after polymerization. The load used in this study ($F = 50 \text{ g}$) was applied to the surface for 30 s. To achieve this, 6 identical occlusal splints were prepared with profiled canine guidance. All the materials were layered and then exposed to UV light according to manufacturers' recommendations. Then each appliance was cut at the level of the canine guidance and 5 mm further, in the direction of the first premolar tooth (Fig. 3). Subsequently, gypsum cylinders were prepared and the specimens were localized inside them, to provide stability during the test (Fig. 4). All the specimens were then subjected to wet polishing. A Neophot 32 microscope (Carl Zeiss, Jenna, Germany), was used to carry out the measurements. Five imprints were made on each of 6 prepared cross-sectional specimens (30 measurements in all). The indenter was then pressed, starting from the apex point of canine guidance and moving towards the Erkodur plate in 0.45 mm intervals. The diagonals of the imprints were measured according to the norm EN ISO 6507-1:2005 [18].



Fig. 3. An example of an occlusal splint prepared for the Vickers microhardness test. The cutting lines were marked in black. The distance between cuts is 5 mm, and was marked with a green line



Fig. 4. A gypsum cylinder with specimen localized inside it. The black arrow indicates the location where the measurement was started for the Vickers microhardness test, and its direction

Polymerization Shrinkage Test

The aim of this test was to assess the amount of linear polymerization shrinkage of the light-cured resin at specified time intervals and in specified storage conditions. For this purpose, 10 specimens were prepared in the same silicon matrix. The samples were made of light-cured resin and had the dental arch shape. Three stainless steel pins were implanted in each sample, forming a shape similar to a triangle (with sides approximately 40 mm in length). The triangle sides were marked A, B and C (Fig. 5). A ZKM 05-250 D measuring unit (Carl Zeiss, Jena, Germany) was used to measure the 3 sides of each triangle. Subsequently,

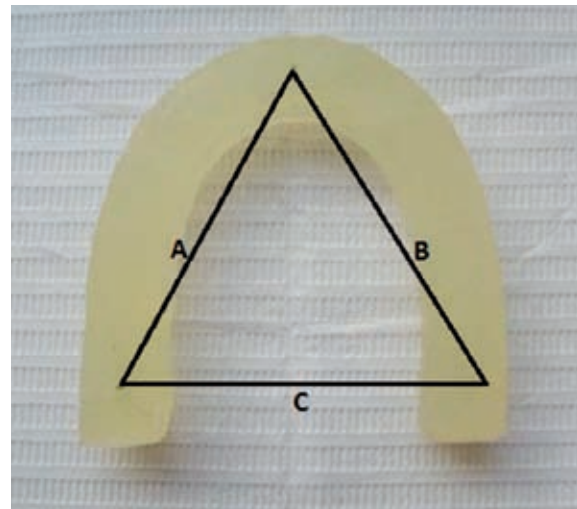


Fig. 5. Example of a sample prepared for the polymerization shrinkage test. The black lines form a triangle with its vertices showing the position of three stainless steel pins. The length of sides A, B and C was about 40 mm in each sample

every line segment (A, B and C) of each sample was measured 4 times: before polymerization; 1 h after light curing; 24 h after polymerization; and finally after being placed in a 37°C water bath (H&H Device Technology, Dresden, Germany) for 24 h. The polymerization time for each sample was 4 min. Differences in the length changes of line segments were examined between:

I: the length measured before polymerization and the length measured 1 h after polymerization (0–1 h);

II: the length measured 1 h after polymerization and the length measured 24 h after polymerization (1–24 h);

III: the length measured after placing the material in the water bath for 24 h and the length measured 24 h after polymerization (H₂O-24 h).

Statistical Analysis

The statistical analysis was performed using STATISTICA software (version 10, StatSoft Inc., Tulsa, Oklahoma, USA). For all the tests, $p = 0.05$ was treated as statically significant.

The results of dye penetrant inspection were analyzed statistically using separate one-sided binomial tests for the vestibular and lingual sides of each sample. The chance value of dye penetration in the observations was 0.5.

The average value of Vickers microhardness was calculated, as well as standard deviation. The results were then analyzed statistically using Spearman's rank-order correlation coefficient.

All differences and length changes of all 3 sides (A, B and C) in the polymerization shrinkage test

were analyzed under different experimental conditions, separately for each line segment, using the nonparametric Friedman ANOVA. For parameters that showed statistically significant differences, *post-hoc* tests were performed. The average value of length changes was calculated, as well as standard deviation.

Results

Dye Penetrant Inspection

For the vestibular surface, there was no dye penetration into the adhesive connection between the resin and the thermoformable foil in any of 30 measurements (Fig. 6). The one-sided binomial test proved that this result differed substantially from the assumed value of color penetration probability [$P_{(p=q=0.5)} [[Y = 0]; p < 0.0001]$. The lack of color penetration is statistically significant. There was only one instance of color penetration from the lingual side (Fig. 7). The one-sided binomial test proved that this result differed substantially from the assumed value of color penetration probability [$P_{(p=q=0.5)} [Y \leq 1]; p < 0.0001]$. The low incidence of dye penetration is statistically significant.

In the control experiment, dye penetration occurred in all 6 measurements (on both the vestibular and lingual surfaces). Dye eventually penetrated the space between the resin and the thermoformable foil. In these tests the Lightdon Bonding adhesive was replaced with an alginate isolator (Fig. 8).

There was no dye penetration into the bulk of the light-cured resin; however, dye penetrated across the entire width of the lacquer coating the resin. The mean value of the lacquer thickness was measured using a Philips XL 30 ESEM scanning

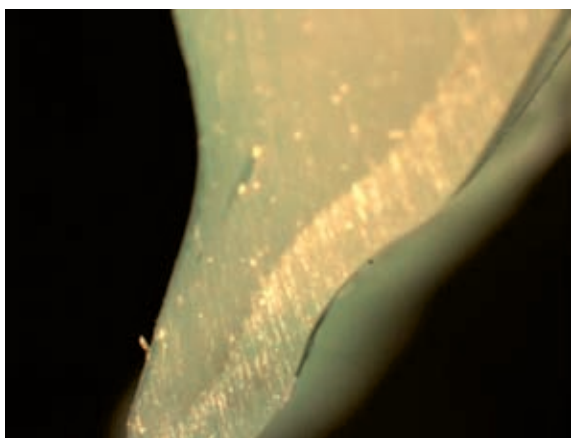


Fig. 6. The absence of dye penetration into the adhesive connection between the tested resin and the thermoformable foil in one of the samples (lingual side surface, magnification $\times 32$)

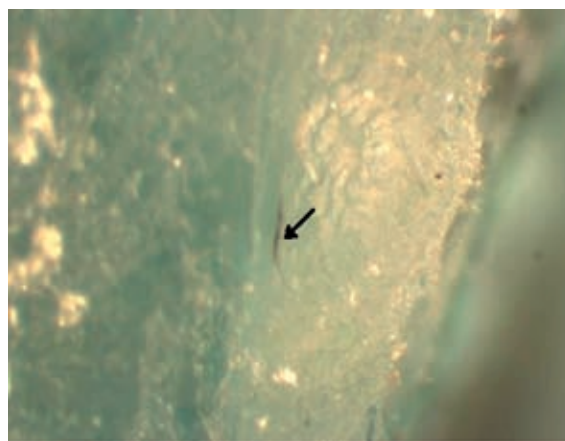


Fig. 7. Visible minor dye penetration into the adhesive connection between the light-cured resin and the thermoformable foil (lingual side surface, magnification $\times 50$)

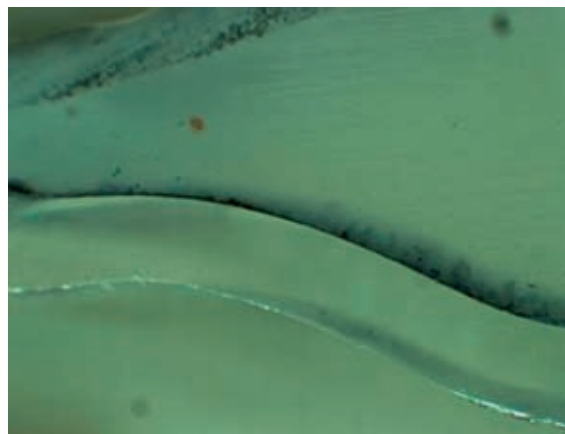


Fig. 8. Visible dye penetration into the space between the resin and the thermoformable foil. In this case Lightdon Bonding adhesive was replaced with alginate isolator for a control test (magnification $\times 32$)

electron microscope (FEI, Hillsboro, Oregon, USA). Thirty randomly selected measurements were performed with a magnification of $\times 1500$ (Table 1). After the data analysis had been done, the average thickness of the lacquer layer was calculated as $5.975 \mu\text{m}$.

Vickers Microhardness Test

Thirty measurements were done to calculate the average value of Vickers microhardness (Table 2). The average value of Vickers microhardness obtained in the study was $HV_{0.05} = 7.43 \text{ N/mm}^2$ (SD = 0.84). Spearman's rank-order correlation coefficient was computed separately for each specimen to assess the relationship between Vickers microhardness and the depth of the light-cured resin layer on Erkodur foil. There was no correlation between the 2 variables in any of the samples (Table 3).

Table 1. Plaquit lacquer thickness obtained among 30 randomly selected locations on different samples

Thickness of Plaquit lacquer layer in [μm] magnification $\times 1500$					
1.	6.25	11.	6.17	21.	7.85
2.	6.01	12.	5.77	22.	5.14
3.	6.13	13.	5.36	23.	6.57
4.	5.53	14.	4.81	24.	8.09
5.	6.17	15.	5.31	25.	7.45
6.	4.96	16.	4.89	26.	4.88
7.	6.01	17.	5.14	27.	5.28
8.	5.12	18.	5.80	28.	4.80
9.	5.85	19.	7.69	29.	4.16
10.	5.20	20.	5.93	30.	8.57

Table 2. Vickers microhardness values obtained in the study (HV 0.05)

Specimen number	Number of Vickers microhardnes measurement [N/mm^2]				
	I	II	III	IV	V
1.	7.6	7.3	7.43	7.22	7.7
2.	8.85	8.42	8.27	8.47	8.07
3.	8.42	7.74	8.63	8.63	8.58
4.	6.4	6.72	7.7	7.48	7.22
5.	6.26	6.5	7.0	6.83	6.75
6.	7.06	6.47	6.4	6.5	6.13

Table 3. Spearman's rank-order correlation coefficients between Vickers microhardness and the depth of the light-cured resin layer on Erkodur foil in all the analyzed specimens

Specimen number	Number of measurements	r_s	p
1.	5	0.1	0.87
2.	5	-0.7	0.19
3.	5	0.6	0.32
4.	5	0.6	0.28
5.	5	0.6	0.28
6.	5	-0.7	0.19

Polymerization Shrinkage Test

Line Segment A

The differences observed in the 3 length changes of line segment A were statistically significant [Friedmann ANOVA ($n = 10$) $\chi^2 = 15.80$; $df = 2$; $p = 0.0004$]. The *post-hoc* analysis showed that in line segment A the length changes described above as set I (before polymerization and 1 h after polymerization) were significantly greater than the changes described as set II [Friedman *post-hoc* > 1.6 ; $p < 0.001$] and those described as set III [Friedman *post-hoc* > 1.07 ; $p < 0.05$]. The length differences in sets II and III did not differ significantly.

Line Segment B

The differences observed in the 3 length changes of line segment B were statistically significant [Friedman ANOVA ($n = 10$), $\chi^2 = 18.20$; $df = 2$; $p = 0.0001$]. The *post-hoc* analysis proved that in line segment B the length changes in set I were significantly greater than the changes in set II [Friedman *post-hoc* > 1.86 ; $p < 0.0001$] and in set III [Friedman *post-hoc* > 1.07 ; $p < 0.05$]. The length differences in sets II and III did not differ significantly.

Line Segment C

The differences observed in the 3 lengths of line segment C were statistically significant [Friedman ANOVA ($n = 10$), $\chi^2 = 15.80$; $df = 2$; $p = 0.0004$]. The *post-hoc* analysis proved that in line segment C the length changes in set I were significantly greater than the changes in set II [Friedman *post-hoc* > 1.6 ; $p < 0.001$] and in set III [Friedman *post-hoc* > 1.07 ; $p < 0.05$]. The length differences in sets II and III did not differ significantly.

The study showed that polymerization shrinkage cause length changes in particular segments. The average values of the differences in length changes of line segments A, B and C (with standard deviation) are shown in Table 4. The average linear shrinkage after polymerization was approx. 1.175%.

Discussion

Dye Penetrant Inspection

Making occlusal splints from 2 different materials carries the risk of lack of a tight bond between the layers. This situation can lead to problems with microorganisms, discoloration and the separation of the components of the appliance. Knowing the shrinkage of methacrylate resins, it

Table 4. Average values and standard deviation of length differences of line segments A, B and C obtained during the polymerization shrinkage test under changing environmental conditions and time

Difference in length changes of line segments	Average difference [mm]	Standard deviation [mm]
Difference A[0–1 h]	0.450	0.071
Difference A[1–24 h]	0.008	0.039
Difference A[H ₂ O–24 h]	0.040	0.079
Difference B[0–1 h]	0.441	0.059
Difference B[1–24 h]	–0.008	0.026
Difference B[H ₂ O–24 h]	0.047	0.039
Difference C[0–1 h]	0.517	0.156
Difference C[1–24 h]	–0.004	0.018
Difference C[H ₂ O–24 h]	0.029	0.067

seems appropriate to use thermocycling to reflect the clinical situation before samples are subjected to dye [19]. Out of 60 measurements done from the vestibular and lingual sides, only one instance of dye penetration to the adhesive connection was observed. This one case is not statistically significant. There was no dye penetration into the deeper structure of the resin; dye only penetrated the lacquer coating layer. However, due to its small average thickness of 5.975 μm , this technique of making occlusal appliances can still be regarded as resistant to coloring agents. The additional test using an isolating layer confirmed the methodological validity of the test performed. Research by Arias et al. and other authors has shown that the procedures followed in the current study are appropriate for evaluating the penetration of the dye into the adhesive connection [20–23]. Omitting the lacquer coating is not recommended, but it should be emphasized that it is not absolutely necessary.

Vickers Microhardness Test

In the light of contemporary knowledge it seems reasonable to use the Vickers method to measure the microhardness of thin, hard coatings. Sharp and well-formed imprints were obtained using this procedure, which ensured the accuracy of the results. The study showed that changes in the Vickers microhardness with the increasing depth of the test material are not statistically significant. It can be concluded that the layer of light-cured resin is fully polymerized throughout its entire thickness. When compared to the results published by Danesh et al., in which the microhardness

of different available light-cured and chemically hardened resins was measured [24], the average microhardness value obtained in the current study is lower. It should be noted that in both studies the same prescribed values of load and pressing time were applied. The average values of Vickers microhardness given by Chuenarrom et al. for both dentin and tooth enamel are much higher than the value of microhardness obtained for Lightdon Splint resin [25]. In fact, from the analysis of these data it can be concluded that the tested material has a lower hardness level than other available resins.

Polymerization Shrinkage Test

Polymerization shrinkage is one of the biggest and most important problems associated with the use of light-cured resins, and it occurs in the hardening process of every polymer. In occlusal splint therapy and restorative dentistry the phenomenon has considerable significance. The main task of occlusal appliances is to provide occlusal stability. When it is necessary to make an occlusal splint, either a direct method (in the patient's mouth) or an indirect method (in an articulator) can be used. Thanks to light-cured resins, more time can be spent forming and properly shaping the occlusal appliance. Even if the model of the splint is close to perfect, a high level of polymerization shrinkage could ruin the work and disrupt the vertical dimensions as well as the occlusal contacts. A research study carried out by Cattani-Loriente et al. showed that the linear shrinkage of popular composite resins used to restore hard dental tissues ranged from 1.54% to 2.11% [26]. In another study, Arora et al. presented the linear polymerization shrinkage of 4 available acrylate materials polymerized at high temperature as ranging from 0.37% to 1.18% [27]. In the current study the linear polymerization shrinkage of Lightdon Splint resin was calculated as approximately 1.175%; this is a reasonably low degree of shrinkage, which definitely makes clinical work easier. However, it must be emphasized that the length changes of line segments A, B and C in set I (the length measured before polymerization and the length measured 1 h after polymerization) were statistically more significant than the changes in sets II and III. In their research, Inoue et al. showed that the type of light, its intensity and time exposure have a significant influence on polymerization shrinkage [28]. It is therefore very important to follow the polymerization instructions specified by the manufacturer. The influence of time and environment on the material were taken under consideration during the tests conducted in the current study. It is commonly known that one of the properties that

characterizes polymers is their ability to absorb water from the environment; the organic matrix and inorganic filler content are responsible for that process. The absorption of water leads to the degradation of polymer bonds due to hydrolysis [29], which means destabilization of the occlusion may occur due to increasing expansion of the material. In their study, Danesh et al. proved that in light-cured resins the absorption of water is much higher than among the chemically-hardened polymers used in the manufacture of occlusal splints [30]. These considerations led the authors of the current study to investigate whether the absorption of water from the environment would have a significant influence on the behavior of the polymer. The test did not reveal any statistically significant changes in the lengths of line segments A, B or C in sets II and III. This result indicates that neither the passage of time after polymerization nor

the humidity of the environment have any implications in terms of increasing the volume of the tested resin. Clinically, this means there would be no destabilization of occlusion.

Within the limitations of this *in vitro* study it was concluded that neither the light-cured resin nor the adhesive connection between the resin and the thermoformable plate showed susceptibility to dye penetration. The application of Lightdon Bonding improves the contact tightness between the tested resin and thermoformable foil. Coating the resin with lacquer is not absolutely necessary. The microhardness of the resin after polymerization is consistent. The maximum polymerization shrinkage occurs immediately after curing. A moist environment and the passage of time do not destabilize the resin structure after polymerization. The light-cured resin tested may be an alternative material for use in occlusal splints manufacturing.

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