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INFLUENCE OF THE LIGHT SOURCE AND CURING PARAMETERS ON MICROHARDNESS OF A SILORANE-BASED DENTAL COMPOSITE MATERIAL

The aim of the study was to determine the influence of the light source and the light-curing parameters (the distance of the material from the light source and time of light-curing) on microhardness of Flitek Silorane dental composite material. Standardized samples of Filtek Silorane material were cured using two types of Light Curing Units (LCUs) – halogen and LED. The distance of the light source and time of curing differed between samples. The Knoop's microhardness was tested using microhardness tester Micromet 5103. Using LED light curing unit allowed to achieve significantly higher microhardness of silorane-based dental material Filtek Silorane than using halogen light curing unit. Decreasing the distance from the light source to the surface of silorane-based material Filtek Silorane improved its microhardness. A prolonged curing time could compensate the drop in microhardness of Filtek Silorane material resulting from an increased distance from the light source to the surface of the material only in a limited range of intervals.

Keywords: dental composite material, light-curing, silorane, microhardness

1. Introduction

Light-cured dental composite materials have certain similarities with hard teeth's tissues. They consist of two basic components: organic matrix and inorganic fillers, bonded together by the third component called a bonding agent [1].

Modern monomers used in composites allow to improve the properties of those materials. Most of dental composite materials contain Bis-GMA resin and another standard monomer is UDMA [2]. This name relates to many monomers containing methacrylate groups and urethane bonds.

To improve the physical, chemical and mechanical properties of composite materials, the chemical content of individual components may be modified.

More often dental composites used in a dental practice are based on silorane matrix [3, 4]. Siloranes consist of a cyclic main silorane chain, which bestows hydrophobic properties, and cycloaliphatic oxirane groups connected through organic alkane groups directly to silica atoms. This type of chemical structure is responsible for the low polymerization shrinkage. The process of creating polymer network in light reactive siloranes relies on opening of oxirane ring under the cationic mechanism. Components based on siloranes have three-phase photoinitiation system, consisting of camphorquinone, Iodonium salt as a cationic photoinitiation and an electron donor [5, 6].

Long-term development of composite materials allowed to acquire filling materials with different properties. The main method of light-curing of composite materials is polymerization with light-curing units. With the increase of light intensity and the time of curing, the conversion degree and hardness of composite materials increase [7].

Hardness is one of the most important properties related to the resistance of the surface to deformation, indentation or scratching under external load [8]. Hardness of dental composites is influenced by many factors, such as: the content of organic matrix, type and content of the filler and the resin's degree of conversion [5]. Hardness of dental composites is lower than that of enamel. Determining the hardness of dental composites is considered as an indirect method of evaluating the degree of conversion of composites [9, 10].

Time of curing is an important parameter affecting physiochemical and mechanical properties of dental composite materials [11, 12]. Applying proper curing method is necessary, because properties of the surface depend on energy density, wave length, time of curing, and distance of the light source from the surface. Hardness of dental composites can depend also on the type of LCU used for curing. Higher light intensity used for polymerization leads to the increase of crosslinking of polymer chains and a better hardness [13].

Properly performed curing process allows to acquire better durability of the filling and decreases the risk of damaging the filling under the influence of masticatory forces. Hardness of a material and other mechanical properties influence significantly the quality of a dental filling.

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1.1. The aim of the research

The aim of the research was to determine the influence of the light source and the light-curing parameters (the distance from the light source and time of light-curing) on the microhardness of Flitek Silorane dental composite material.

2. Material and methods

2.1. Material

The research was performed on 70 standardized samples of Filtek Silorane dental composite (3M ESPE, shade A3) based on a silorane resin. The samples 7 mm x 3 mm x 3 mm were made by light-curing of the dental composite material in a specially prepared silicone mold.

TABLE 1
Characterization of Filtek Silorane dental material used in the research as declared by the producer

Material	Filtek Silorane
Producer	3M ESPE
Matrix	Silorane
Filler content [%]	76
Filler size [µm]	0.1-2.0

2.2. Methods

The material was polymerized using Elipar Highlight halogen LCU (3M ESPE), 75W with maximal irradiance of 700 mW/cm^2 and SmartLite LED LCU (DENTSPLY), 5W LED with maximal irradiance of 950 mW/cm^2 (Tab. 2). The distance of the light-curing unit (LCU) and the time of curing differed between the samples. The distance of the LCU was set with spacer rings 2 mm high.

TABLE 2
Characteristics of LCU used for light-curing

LCU	Elipar Highlight	SmartLite
Type	Halogen	LED
Producer	3M ESPE	DENTSPLY
Wavelength [nm]	410-500	450-490
Power [mW/cm^2]	700	950

The samples were cured for 10 s, 20 s, 30 s, 40 s, 50 s, 60 s or 70 s from a distance of 0 mm, 2 mm, 4 mm, 6 mm and 8 mm. The samples were stored in airtight polypropylene bags.

Microhardness of Filtek Silorane (3M ESPE) samples cured with halogen or LED LCUs was measured with Knoop test with Micromet 5103 microhardness tester by Buehler. Hardness was tested with 1 kG of force. Ten measurements were taken on each sample.

2.3. Statistical methodology

All calculations were performed with the use of StatSoft Inc. statistical software STATISTICA, version 10.0. and Excel 2007 calculation sheet.

Firstly, W Shapiro-Wilk test was used to check if the quantitative variables show the normal distribution. As the data met the requirements of normal distribution the Sudent's t-test was used to test the significance of differences between two groups (independent variables model). Significance of differences among more than two groups was tested with F ANOVA test with *post-hoc* Tukey's test. The statistical significance level was set at p=0.05.

3. Results

We obtained significantly higher microhardness of Filtek Silorane material cured with LED LCU when compared to the material cured with halogen LCU apart from the samples cured from the distance of 8 mm for 40 s and longer. The results are shown in Tables 3, 4, 5, 6 and 7.

TABLE 3
The comparison of microhardness of Filtek Silorane material cured with LED and halogen light source directly at the surface of the material in relation to curing time

Curing time [s]	LED light source Mean ± SD	Halogen light source Mean ± SD	p-value of Student's t-test
10	49.8 ± 4.2	41.4 ± 3.8	<0.001*
20	50.3 ± 4.9	43.6 ± 4.1	<0.001*
30	51.6 ± 3.8	45.0 ± 4.7	<0.001*
40	52.8 ± 5.2	49.9 ± 5.3	0.032*
50	53.6 ± 4.9	51.1 ± 4.7	0.026*
60	54.4 ± 4.2	52.4 ± 4.4	0.011*
70	55.8 ± 5.1	53.3 ± 5.6	0.048*

* an asterisk indicates a significant difference at p<0,05

TABLE 4
The comparison of microhardness of Filtek Silorane material cured with LED and Halogen light source from the distance of 2 mm in relation to curing time

Curing time [s]	LED light source Mean ± SD	Halogen light source Mean ± SD	p-value of Student's t-test
10	47.1 ± 3.2	38.0 ± 2.4	<0.001*
20	48.4 ± 4.9	41.9 ± 4.1	<0.001*
30	49.7 ± 3.9	43.1 ± 2.8	0.004*
40	51.1 ± 4.1	48.8 ± 4.5	0.026*
50	52.4 ± 5.2	50.2 ± 4.9	0.048*
60	53.5 ± 6.3	51.6 ± 5.2	0.039*
70	54.6 ± 5.6	52.1 ± 4.8	0.041*

* an asterisk indicates a significant difference at p<0,05

TABLE 5

The comparison of microhardness of Filtek Silorane material cured with LED and Halogen light source from the distance of 4 mm in relation to curing time

Curing time [s]	LED light source Mean ± SD	Halogen light source Mean ± SD	p-value of Student's t-test
10	44.6 ± 4.3	35.7 ± 3.9	<0.001*
20	47.9 ± 5.1	40.6 ± 4.2	<0.001*
30	48.4 ± 3.2	41.8 ± 4.8	<0.001*
40	49.8 ± 2.9	48.0 ± 2.4	0.048*
50	51.2 ± 3.1	49.1 ± 2.7	0.044*
60	53.0 ± 3.8	49.6 ± 3.1	0.023*
70	54.2 ± 4.8	50.8 ± 3.9	0.027*

* an asterisk indicates a significant difference at p<0,05

TABLE 6

The comparison of microhardness of Filtek Silorane material cured with LED and Halogen light source from the distance of 6 mm in relation to curing time

Curing time [s]	LED light source Mean ± SD	Halogen light source Mean ± SD	p-value of Student's t-test
10	41.8 ± 4.7	31.4 ± 3.9	<0.001*
20	45.2 ± 3.9	38.9 ± 4.1	<0.001*
30	47.0 ± 3.7	40.4 ± 4.8	<0.001*
40	48.3 ± 4.8	46.2 ± 3.2	0.036*
50	49.7 ± 5.1	47.6 ± 2.8	0.032*
60	51.5 ± 4.9	48.3 ± 2.7	0.041*
70	52.3 ± 4.7	50.1 ± 3.8	0.046*

* an asterisk indicates a significant difference at p<0,05

TABLE 7

The comparison of microhardness of Filtek Silorane material cured with LED and Halogen light source from the distance of 8 mm in relation to curing time

Curing time [s]	LED light source Mean ± SD	Halogen light source Mean ± SD	p-value of Student's t-test
10	37.0 ± 3.2	23.3 ± 3.0	<0.001*
20	40.4 ± 3.8	34.6 ± 3.9	<0.001*
30	42.6 ± 2.9	38.0 ± 4.1	<0.001*
40	44.1 ± 4.1	43.7 ± 4.8	0.127
50	46.3 ± 5.9	45.1 ± 4.6	0.259
60	47.2 ± 5.1	46.0 ± 4.8	0.573
70	48.4 ± 4.8	46.8 ± 5.1	0.237

* an asterisk indicates a significant difference at p<0,05

The microhardness of Filtek Silorane material cured with the halogen LCU increased with the exposure time. The highest microhardness of the sample was acquired for 70 s of curing. An increase in the hardness of the samples was observed also for decreasing distance to the LCU. The highest hardness of 53.3 HK was acquired for the sample cured directly at the surface for 70 s (Table 3).

The same observations were characteristic of the material cured with the LED LCU. The hardness of Filtek Silorane material cured with LED LCU increased with the exposure time. The highest hardness of 55.8 HK was acquired for a sample cured directly at the surface for 70 s (Table 3). The shorter the distance to the light source the higher the hardness of the cured material.

As the microhardness of the material decreases with the distance from the light source, it was important to determine the time on top of which increasing the exposure cannot compensate the drop in the microhardness resulting from the distance from the LCU. For this purpose ANOVA test was carried out. The results of ANOVA test for LED LCU and halogen LCU are shown in Tables 8 and 9. The results showed that there were not significant differences of the microhardness among the samples cured for different times from the distance of 8 mm both for LED and halogen LCU's.

TABLE 8

The results of the ANOVA test for the microhardness of Filtek Silorane composite material cured for different times with LED light curing unit (LCU) in relation to the distance from the light source

Distance from the LED LCU [mm]	ANOVA test F value	p-level
0	8.82	<0.001*
2	9.24	<0.001*
4	11.42	0.021*
6	7.14	0.032*
8	3.12	0.628

* an asterisk indicates significant values at the level p<0,05

TABLE 9

The results of the ANOVA test for the microhardness of the samples of Filtek Silorane composite material cured for different times with halogen light curing unit (LCU) in relation to the distance from the light source

Distance from the halogen LCU [mm]	ANOVA test F value	p-level
0	9.92	<0.001*
2	11.76	<0.001*
4	8.57	0.018*
6	3.11	0.012*
8	2.84	0.462

* an asterisk indicates significant values at the level p<0,05

For cases in which ANOVA showed significant differences among the groups of samples exposed to the light source for different times, *post-hoc* Tukey's test was performed. The Tukey's test showed the significant differences in microhardness between all the samples cured for different times directly at the surface of the material as well as from the distance of 2 and 4 mm. The p-values of Tukey's test for the consecutive samples of the material cured both with LED and halogen LCUs are shown at Figures 1 and 2.

Figure 1 shows that there was a significant difference between the microhardness of the samples cured with LED LCU up to 50 s from the distance of 6 mm, but increasing the time of exposure over 50 s resulted in a non-significant increase of microhardness. The results shown at Figure 2 reveal that using the halogen LCU results in significant increase of the microhardness of samples cured up to 40 s from the distance of 6 mm, but there is no further growth in microhardness above 40 s of exposure.

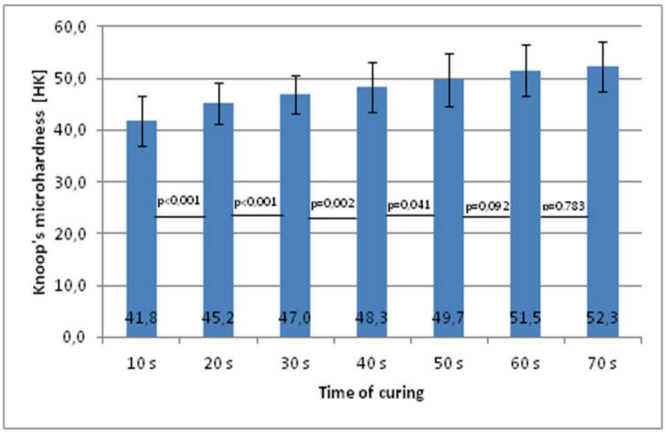

Fig. 1. Microhardness of Filtek Silorane composite material cured with LED light curing unit from the distance of 6 mm with calculated *p*-values of *post-hoc* Tukey's test between the consecutive groups.

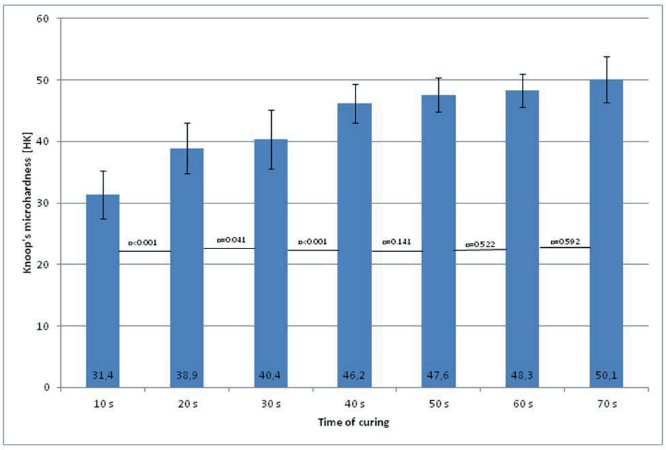

Fig. 2. Microhardness of Filtek Silorane composite material cured with halogen light curing unit from the distance of 6 mm with calculated *p*-values of *post-hoc* Tukey's test between the consecutive groups.

4. Discussion

Dental materials, like other biomaterials functioning in the environment of a living organism, must meet stringent requirements in terms of mechanical and physio-chemical properties [14, 15]

Microhardness has become one of the most commonly used methods for investigation of factors influencing polymerization. This is a result of research works proving a good correlation between hardness tests and spectroscopy [6, 16]. Previous research papers showed that longer exposure times result in increased cure depth of a composite resin, higher hardness and a better conversion degree [17, 18, 19].

Our results have shown that with the increase of the distance of the light source from the surface of the sample, the hardness of the silorane-based material cured with LCUs decreased. Previous research on the intensity of LED LCUs shows a significant decrease of the intensity with the distance to the cured surface of more than 4 mm. The decrease of the light intensity deteriorated the hardness, the compressive strength and the wear resistance of a composite material. The quality of curing decreased when the distance between the LCU and the surface of the material was higher than 4 mm [20].

Our research has shown that the average microhardness of the examined composite samples is between 23,3 and 55,8 HK. The results are compatible with the results of other authors [8, 21]. The samples cured with LED LCU achieve a significantly higher microhardness then the samples cured with halogen LCU. Thus, especially in difficult clinical situations, LED LCUs should be preferred for curing silorane-based dental composite materials.

To our best knowledge, there are no available research articles concerning the problem of compensation of the drop of microhardness resulting from an increased distance of the material from the light source by a prolonged time of exposure to the light emitted by an LCU. Our results have shown that expanding the time of curing up to 70 s by either LED or halogen LCU may result in significant increase of the microhardness of the silorane-based material when the distance from the LCU to the surface of the sample is up to 4 mm. When the distance equals to 6 mm expanding of the exposure time above 50 s is not reasonable if the curing procedure is carried out with LED LCU and above 40 s if halogen LCU is used. This observation has very important clinical implications proving that prolonged curing time can compensate an increased distance from the light source to the surface of the material only in a limited range of intervals. These distances should be precisely shown by a producer to ensure a proper microhardness of the final filling material. In our research it has been shown that there is a significant difference between the microhardness of the samples cured with LED LCU up to 50 s, but increasing the time of exposure above 50 s results in a non-significant increase of microhardness. When halogen LCU is used than there is a significant increase of the microhardness of samples cured up to 40 s from the distance of 6 mm, but there is no further growth in microhardness above 40 s of exposure. In our research Filtek Silorane achieved the highest hardness after curing directly at the surface of the sample, regardless of the LCU type.

5. Conclusions

1. Using LED light curing unit allows to achieve significantly higher microhardness of silorane-based dental material Filtek Silorane than using halogen light curing unit.
2. Decreasing the distance from the light source to the surface of silorane-based material Filtek Silorane improves its microhardness.
3. A prolonged curing time can compensate the drop in microhardness of Filtek Silorane material resulting from an increased distance from the light source to the surface of the material only in a limited range of intervals.

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