



The effect of the curing time and the distance from the light source on hardness of Methacrylate and Silorane resin-based dental composite materials

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ABSTRACT

Purpose: The aim of the work was to test hardness of composite dental materials with different resins in relation to different light-curing parameters.

Design/methodology/approach: The following article provides and insight into factors influencing hardness of composite materials. Standardized samples made of Herculite XRV based on a methacrylate resin and Filtek Silorane based on silorane resin were tested using two types of Light Curing Units (LCUs) – halogen and LED. The distance of light source and time of curing differed between samples.

Findings: Filtek Silorane composite compared to Herculite XRV composite guarantees higher hardness, regardless of the used LCU type. Using LED LCU compared to halogen LCU allows to obtain higher hardness both for Herculite XRV and Filtek Silorane composite. The lower the distance of light source the higher the hardness of composite material.

Research limitations/implications: Further studies will provide additional information on other properties such as compressive strength, wear resistance and light transmission.

Practical implications: This article presents important comparison between older and newer composite technology. It provides practical information on polymerization methods.

Originality/value: Article shows broad spectrum of different curing methods, important to the composite use in dentistry.

Keywords: Composites; Light-curing; Silorane; Methacrylate; Hardness

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PROPERTIES

1. Introduction

Recent decades brought great improvements in the field of dental composites [1]. In 1962 Bowen has introduced bisphenol A glycidyl methacrylate (Bis-GMA) as a dental restorative material, which started the evolution of dental composites. The composite's properties such as viscosity and polarity has been changed many times, by modification of functional groups [2]. Majority of available composite resins are based on methacrylates [1].

Recently, a new composite material – silorane was made commercially available. The name of the material indicates a hybrid compound of siloxane and oxirane functional moieties [3]. The development of the composite materials increased their strength, wear resistance and esthetics [4].

One of the most important properties of dental composites is their hardness. The hardness of dental filling materials is an important measure and informs how easy the structure of a material can be finished and determines its resistance to scratch during the lifetime. Finishing and polishing is important for aesthetic reasons. Cracks can influence the material fatigue and cause premature damage. Hardness can be generally defined as a resistance to permanent penetration or indentation of the surface. A material can be considered hard if it is resistant to the indentation by a hard indenter, such as a diamond.

Hardness of composite dental materials is usually measured on the Knoop scale. Knoop hardness is acquired by measuring the length of the longest diagonal of the indentation made by the diamond indenter and calculating necessary load to create the projected area. The bigger the projected area, the lower the reading on the scale.

Knoop's hardness test is used to measure the hardness of thin plastic or metal sheets and also brittle materials. Applied load does not exceed 35N. The load is applied to a carefully prepared diamond indenter with a pyramidal shape. The test measures the longer diagonal of the indentation in the material. The number on Knoop hardness scale (KHN, Knoop hardness number) is acquired by dividing the test load by the projected area:

$$KHN = F/d^2$$

In the formula above F is an applied load and d is the length of indentation along its longer axis in mm. The main disadvantage of this method is the need of polishing the test surface. This is necessary to acquire a flat sample and is more time consuming than in other, less precise methods.

The aim of the work was to test hardness of dental composite materials with different matrixes in relation to different light-curing parameters.

2. Material and method

The research was performed on 70 standardized samples of Herculite XRV dental composite (KERR, shade A3) based on a methacrylate resin and 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 acquired by light-curing dental composites in a specially prepared silicone mold.

The materials were polymerized using Elipar Highlight halogen LCU (3M ESPE), 75W with maximal irradiance of 700 mW/cm² and SmartLite LED LCU (DENTSPLY), 5W LED with maximal irradiance of 950 mW/cm². The distance of the LCU and the time of curing differed between the samples. The distance of the light-curing unit (LCU) was set with spacer rings 2 mm high.

The time of light-curing of the dental composite is the time of polymerization of unsaturated components. Polymerization of the photoactive particles is initiated by the light emitted by the halogen and LED LCUs during the curing. 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 coding for light-cured samples of dental composites used in the paper was as follows:

type of the material – distance in millimeters – time of light-curing in seconds – type of the LCU

The following codes were established:

- H – Herculite XRV dental composite (KERR),
- FS – Filtek Silorane dental composite (3M ESPE),
- The numbers between 0 and 8 determines the distance between the LCU (halogen or LED) and the surface of cured dental composite during the curing,
- The numbers between 10 and 70 determine the time of light-curing in seconds,
- HAL – dental composite cured with halogen LCU,
- LED – dental composite cured with LED LCU.

An example of sample coding:

H-0-40-HAL – Herculite XRV dental composite light-cured directly on the surface (0 mm) for 40 s with the halogen LCU.

The hardness of Herculite XRV and Filtek Silorane samples cured by the halogen or LED LCUs was measured with Knoop test in the Institute of Material Engineering at the West Pomeranian University of Technology in Szczecin in accordance to PN-EN ISO 6507-1. The tests were performed with Micromet 5103 microhardness tester by Buehler. In the experiment the load of 10 N was applied.

The tests were performed three time for each cured sample. The hardness of the tested dental material using the Knoop test (HK) was calculated using following formula:

$$HK = 14,23 \cdot 10^3 \cdot \frac{F}{d^2}, \text{ where:}$$

F – load, kg,

d – the longer diagonal, μm.

3. Statistical methodology

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

Quantitative variables were expressed by: mean, standard deviation, median, minimal and maximal value (range) and 95% CI (Confidence Interval). The qualitative variables were expressed by numerical values.

The W Shapiro-Wilk test was used to check if the quantitative variable came from normally distributed data. The Levene’s (Brown-Forsythe) test was used to check the hypothesis on equal variances.

The difference significance between two groups (independent variables model) was tested using significance difference test: t-Student or U Mann-Whitney test. The significant difference between more than two groups was tested with F (ANOVA) or Kruskal-Wallis test (in case of non-compliance to ANOVA test requirements).

The strength and direction of correlation between variables was tested using correlation analysis calculating Pearson and/or Spearman correlation coefficients. The statistical significance level was set at p=0.05.

4. Results

Table 1. Hardness of Herculite XRV material cured with halogen LCU in relation to the distance from the light source and the curing time

		The distance from the light source, mm				
		0	2	4	6	8
Curing time, s	10	38.2	36.3	33.7	28.5	18.0
	20	41.4	40.9	39.1	37.6	33.7
	30	49.6	41.7	40.4	38.6	36.0
	40	49.8	48.6	47.8	45.4	43.4
	50	48.3	47.9	44.3	42.3	43.0
	60	46.2	43.8	42.4	41.6	41.4
	70	44.1	42.3	41.3	40.8	41.1

The hardness of Herculite XRV material cured with halogen LCU, increased with duration of curing. The highest hardness of 49,8 HK was acquired with direct curing on the surface of the sample for 40 seconds. A longer exposure to the light caused a small decrease in hardness. The shorter the distance from LCU the higher the hardness of the samples (Tab. 1, Fig. 1).

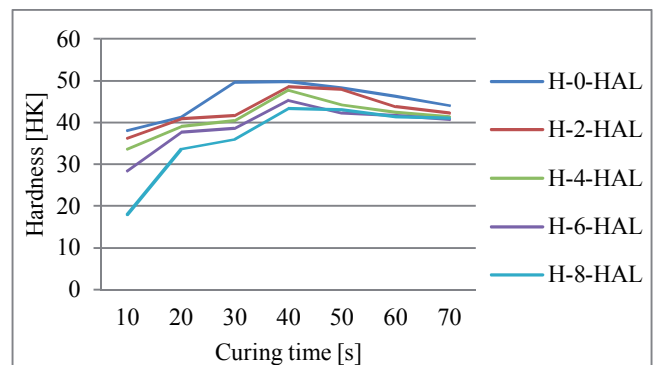


Fig. 1. Hardness of Herculite XRV material cured with halogen LCU in relation to the distance from the light source and the curing time

The highest hardness of the Herculite XRV sample cured with LED LCU was acquired for 50 s of curing from the distance of 0 mm. Longer curing time caused a decrease in hardness. The test showed a clear relation between the distance from the light source and hardness of the Herculite XRV dental composite. The closer the distance of the LCU, the bigger the hardness of the cured material (Tab. 2, Fig 2).

Table 2. Hardness of Herculite XRV material cured with LED LCU in relation to the distance from the light source and the curing time

		The distance from the light source, mm				
		0	2	4	6	8
Curing time, s	10	49.4	46.3	43.2	40.6	32.1
	20	50.1	47.6	45.6	44.1	34.2
	30	51.5	47.7	47.0	45.6	36.3
	40	51.8	49.0	48.1	47.0	37.7
	50	53.7	50.4	49.2	48.1	40.1
	60	50.6	48.9	47.6	46.0	38.3
	70	48.8	46.3	44.8	42.3	36.5

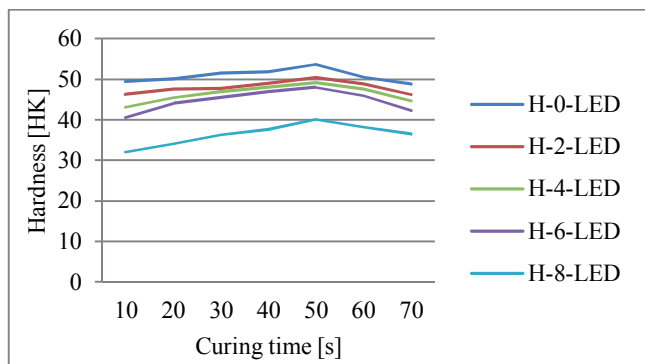


Fig. 2. Hardness of Herculite XRV material cured with LED LCU in relation to the distance from the light source and curing time

Table 3. Hardness of Filtek Silorane material cured with halogen LCU in relation to the distance from the light source and the curing time

Curing time, s	The distance from the light source, mm				
	0	2	4	6	8
10	41.4	38.0	35.7	31.4	23.3
20	43.6	41.9	40.6	38.9	34.6
30	45.0	43.1	41.8	40.4	38.0
40	49.9	48.8	48.0	46.2	43.7
50	51.1	50.2	49.1	47.6	45.1
60	52.4	51.6	49.6	48.3	46.0
70	53.3	52.1	50.8	50.1	46.8

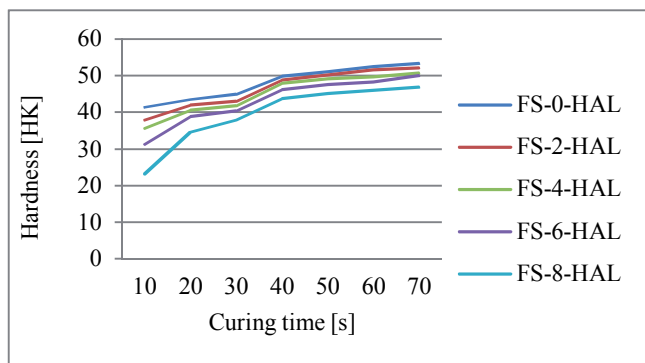


Fig. 3. Hardness of Filtek Silorane material cured with halogen LCU in relation to the distance from the light source and the curing time

The hardness of Filtek Silorane material cured with halogen LCU increased constantly with a longer exposure

time. The highest hardness of the sample was acquired for 70 s of curing. An increase in the hardness of the samples was observed for decreasing distance to the LCU. The highest hardness of 53.5 HK was acquired for the sample cured directly on the surface for 70 seconds (Tab. 3, Fig. 3).

Hardness of Filtek Silorane material cured with LED LCU increased with the exposure time. The highest hardness of 55.8 HK was achieved for a sample directly cured on the surface for 70 seconds. The closer the distance from the light source the higher the hardness of the cured material (Tab. 4, Fig. 4).

Table 4. Hardness of Filtek Silorane material cured with LED LCU in relation to the distance from the light source and the curing time

Curing time, s	The distance from the light source, mm				
	0	2	4	6	8
10	49.8	47.1	44.6	41.8	37.0
20	50.3	48.4	47.9	45.2	40.4
30	51.6	49.7	48.4	47.0	42.6
40	52.8	51.1	49.8	48.3	44.1
50	53.6	52.4	51.2	49.7	46.3
60	54.4	53.5	53.0	51.5	47.2
70	55.8	54.6	54.2	52.3	48.4

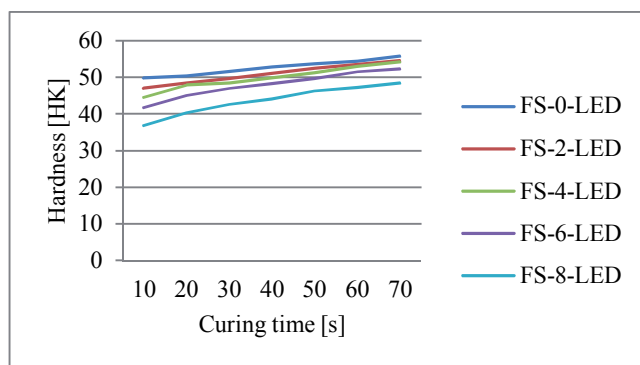


Fig. 4. Hardness of Filtek Silorane material cured with LED LCU in relation to the distance from the light source and the curing time

The Filtek Silorane material had higher hardness than Herculite XRV material. The highest hardness of Filtek Silorane samples was achieved for the LED LCU with a 70 s exposure and direct contact with the surface of the sample. The results of the hardest samples of Herculite XRV and Filtek Silorane cured with two different LCUs

were compared. Higher hardness of both materials was observed when the LED LCU was used (Fig. 5).

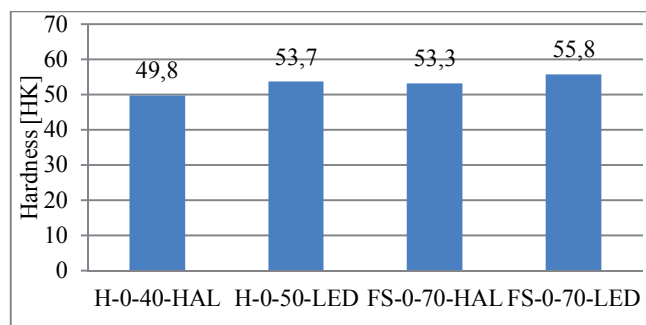


Fig. 5. The highest hardness readings of Herculite XRV and Filtek Silorane materials in relation to the LCU used, the distance from the light source and the curing time

The mean hardness (Tab. 5) of the material based on methacrylate matrix was 43.2 (6.1) and for silorane matrix was 46.8 (6.0). The hardness of samples based on silorane matrix was significantly higher when compared to the samples based on methacrylate matrix (values of the test statistics of U Mann-Whitney $Z=-3.80$, $p=0.0001$).

Figure 6 shows mean values and standard deviations of Knoop hardness test results of materials based on methacrylate and silorane matrixes.

Table 5. The statistics of the Knoop hardness test

	Herculite	Filtek	p value
mean (SD)	43.2 (6.1)	46.8 (6.0)	
95%CI	[41.8;44.7]	[45.3;48.2]	
range (min-max)	18.0-53.7	23.3-55.8	
median	44.0	48.3	$p=0.0001$

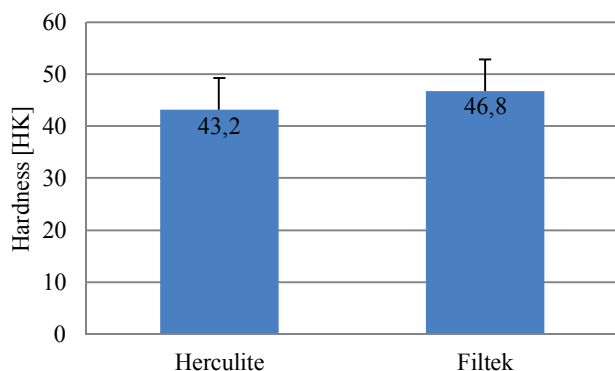


Fig. 6. The statistics of the Knoop hardness test

The mean hardness (Tab. 6) of the material based on methacrylate matrix polymerized with halogen LCU was 41.1 (6.2) and based on silorane matrix 44.5 (6.7). The mean hardness of the methacrylate-based material polymerized with LED LCU was 45.3 (5.3) and for silorane-based was 49.0 (4.4).

The hardness differed depending on the material and LCU type (values of the test statistics of U Mann-Whitney $H=31.96$, $p=0.0001$).

The hardness of the methacrylate-based materials cured with halogen light was significantly lower than the hardness of methacrylate-based material cured with LED LCU ($p=0.0288$). It was also lower than the hardness of silorane-based material polymerized with LED LCU ($p=0.0001$).

The hardness of the silorane-based materials polymerized with LED LCU was significantly higher than hardness of methacrylate-based materials polymerized with LED LCU ($p=0.0285$) and silorane-based material polarized with halogen LCU ($p=0.0120$).

Table 6. The statistics of the Knoop hardness test in relation to the LCU type

	Herculite		Filtek		p value
	HAL	LED	HAL	LED	
mean (SD)	41.1 (6.2)	45.3 (5.3)	44.5 (6.7)	49.0 (4.4)	¹ 0.0288
95%CI	[39.0;43.3]	[43.5;47.2]	[42.2;46.8]	[47.5;50.5]	² 0.0001
range (min-max)	18.0-49.8	32.1-53.7	23.3-53.3	37.0-55.8	³ 0.0285
median	41.6 ^{1,2}	47.0 ^{1,3}	46.0 ^{2,4}	49.7 ^{3,4}	⁴ 0.0120

Figure 7 shows mean values and standard deviations of Knoop hardness test results of materials based on methacrylate and silorane matrixes in relation to LCU type.

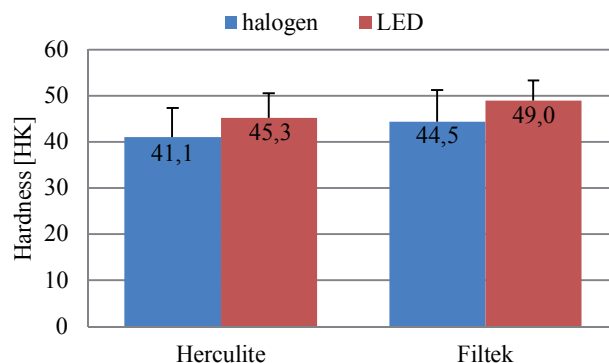


Fig. 7. The statistics of the Knoop hardness test in relation to the LCU type

Hardness of methacrylate-based and silorane-based materials increased with longer exposure time. Correlation coefficient for methacrylate-based material was $R=0.26$, $p=0.0318$ and for silorane-based material $R=0.71$, $p=0.0001$. Correlation with the exposure time was significantly higher for the silorane-based material ($p=0.0005$) (Tab. 7, Figs. 8,9).

Table 7. Correlation of the Knoop hardness and time of exposure (R – correlation coefficient)

Herculite		Filtek		p value
R	p	R	p	
0.26	0.0318	0.71	0.0001	0.0005

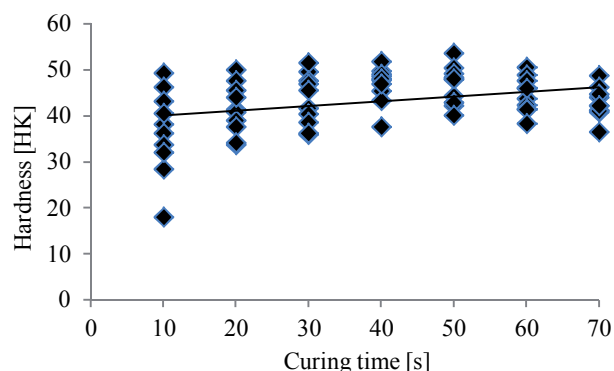


Fig. 8. Correlation of the Knoop hardness and time of exposure of methacrylate-based material

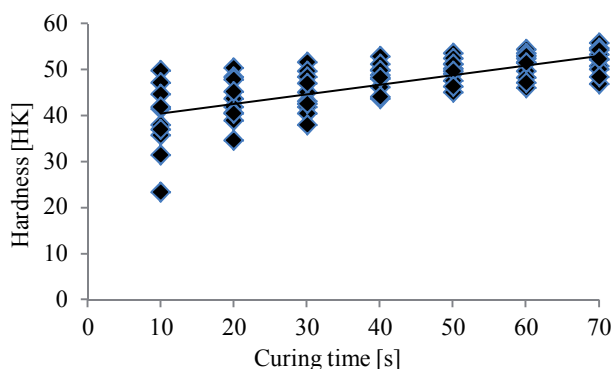


Fig. 9. Correlation of the Knoop hardness and time of exposure of silorane-based material

The hardness of methacrylate-based and silorane-based materials decreased with the increasing distance from the light source. The correlation coefficient for the

methacrylate-based material was $R=-0.66$, $p=0.0001$ and for the silorane-based material $R=-0.53$, $p=0.0001$. There was no statistically significant difference between the correlation coefficients ($p=0.2428$) (Tab. 8, Figs. 10,11).

Table 8. Correlation of the Knoop hardness and the distance from the light source (R – correlation coefficient)

Herculite		Filtek		p value
R	p	R	p	
-0.66	0.0001	-0.53	0.0001	0.2428

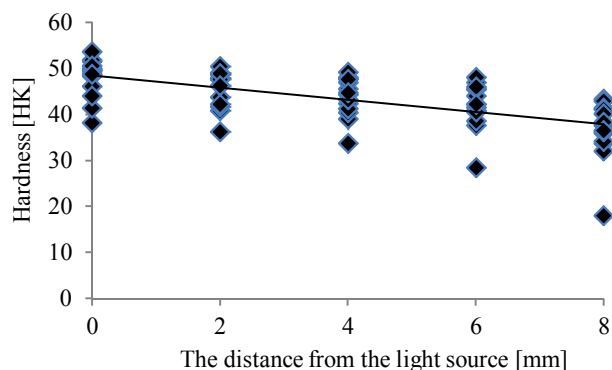


Fig. 10. Correlation of the Knoop hardness readings and the distance from the light source of the methacrylate-based material

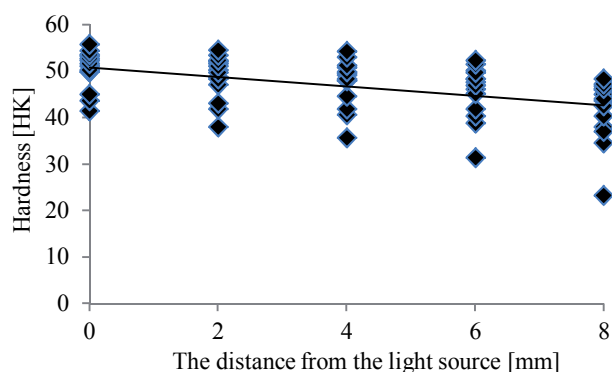


Fig. 11. Correlation of the Knoop hardness readings and the distance from the light source of the silorane-based material

Hardness of methacrylate-based and silorane-based materials increased with longer exposure time. The correlation coefficient for the methacrylate-based material cured with the halogen LCU was $R=0.53$, $p=0.0010$ and for

silorane-based material $R=0.87$, $p=0.0001$. The correlation coefficient for the methacrylate-based material cured with LED LCU was $R=0.12$, $p=0.4888$ (no statistically significant correlation) and for the silorane-based material $R=0.68$, $p=0.0001$ (Tab. 9, Figs. 12-15).

Table 9.
Correlation of the Knoop hardness and time of exposure (R – correlation coefficient) in relation to the LCU type

Herculite				Filtek			
HAL		LED		HAL		LED	
R	p	R	p	R	p	R	p
0.53	0.0010	0.12	0.4888	0.87	0.0001	0.68	0.0001

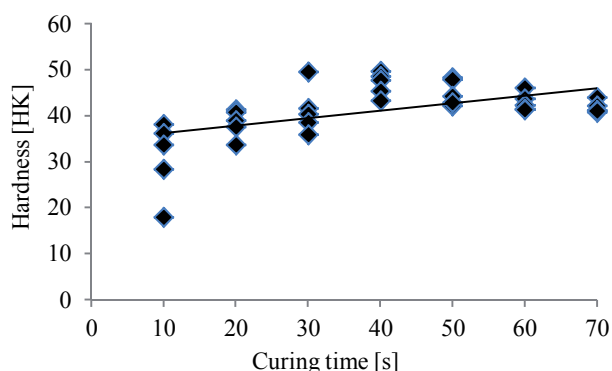


Fig. 12. Correlation of the Knoop hardness and exposure time of methacrylate-based material cured with halogen LCU

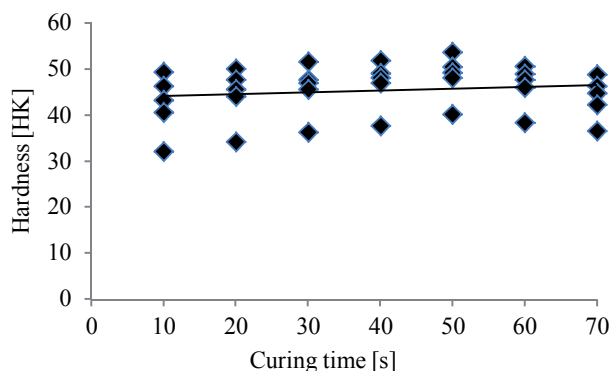


Fig. 13. Correlation of the Knoop hardness and exposure time of methacrylate-based material cured with LED LCU

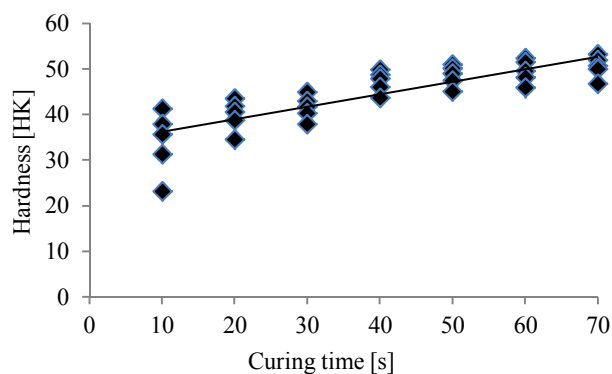


Fig. 14. Correlation of the Knoop hardness and exposure time of silorane-based material cured with halogen LCU

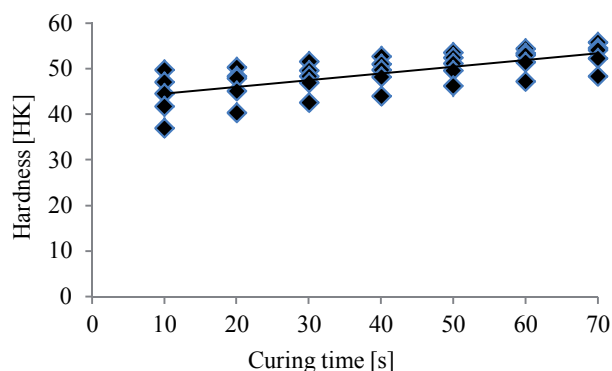


Fig. 15. Correlation of the Knoop hardness and exposure time of silorane-based material cured with LED LCU

Hardness of methacrylate-based and silorane-based materials decreased with an increase of the distance from the light source. The correlation coefficient for the methacrylate-based material cured with halogen LCU was $R=-0.48$, $p=0.0038$ and for the silorane-based material $R=-0.43$, $p=0.0097$. The correlation coefficient for the methacrylate-based material cured with LED LCU was $R=-0.88$, $p=0.0001$ and for the silorane-based material $R=-0.70$, $p=0.0001$ (Tab. 10, Figs. 16-19).

Table 10.
Correlation of the Knoop hardness and the distance from the light source (R – correlation coefficient) in relation to LCU type

Herculite				Filtek			
HAL		LED		HAL		LED	
R	p	R	p	R	p	R	p
-0.48	0.0038	-0.88	0.0001	-0.43	0.0097	-0.70	0.0001

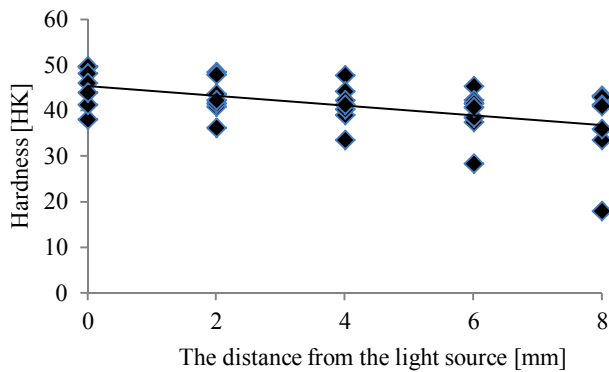


Fig. 16. Correlation of the Knoop hardness and the distance from the light source of the methacrylate-based material cured with halogen LCU

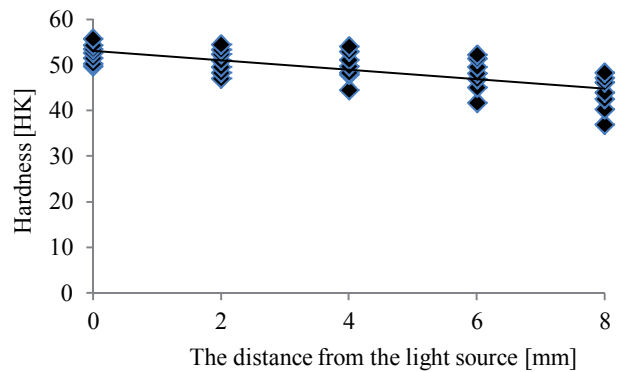


Fig. 19. Correlation of the Knoop hardness and the distance from the light source of the silorane-based material cured with LED LCU

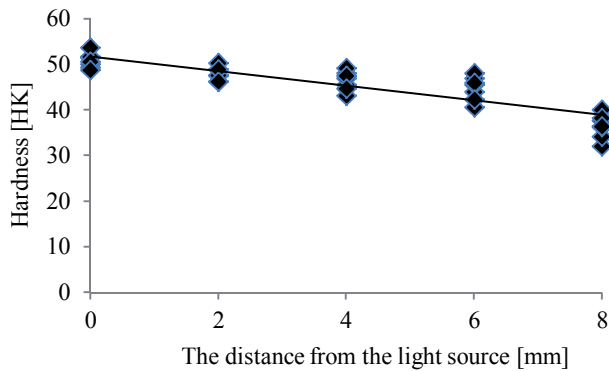


Fig. 17. Correlation of the Knoop hardness and the distance from the light source of the methacrylate-based material cured with LED LCU

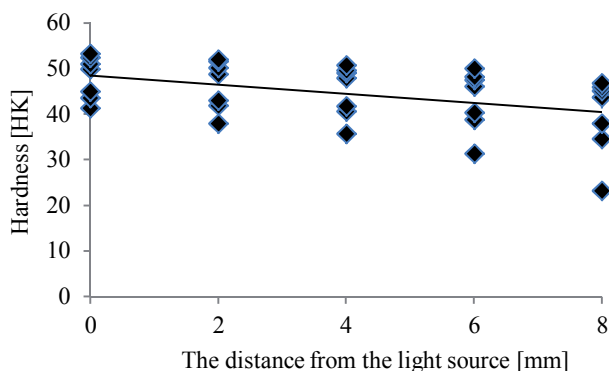


Fig. 18. Correlation of the Knoop hardness and the distance from the light source of the silorane-based material cured with halogen LCU

5. Discussion

Many authors use the Knoop test in their research for the microhardness analysis of composite materials. The test is performed by applying a static pressure by pyramidal diamond indenter, with angles of 172° and 130° degrees [5-8]. The Vickers test is more frequently used to evaluate the microhardness of composite materials [9-11]. Also Brinell and Barcola method is used to evaluate the hardness of composite materials [12-14]. Regardless of a chosen test method, the results of hardness of a material obtained are always comparable between Vickers, Knoop and Brinell or Barcola method. It is possible to calculate given results of the hardness using corresponding coefficients and in some cases, like in case of Vickers and Brinell tests, the readings partially concur.

Microhardness has become one of the most commonly used methods for investigating the factors influencing the polymerization process [15-17]. Some research works prove a good correlation between the hardness tests and spectroscopic methods [18-20].

Some authors emphasize a strong correlation between a conversion degree and hardness of a composite material [21]. Tanoue et. al. [22,23], evaluating the properties of composite materials (hardness, polymerization shrinkage, resistance to bending, Young's modulus, water sorption, solubility in water) have recognized microhardness and solubility in water as characteristics showing the correlation between material properties and the degree of resin conversion. Hardness of a material depends not only on the conversion coefficient but also the type and a quantity of a filler. Asmussen [16] and Harris et. al. [24] have proposed a method based on a dynamic

thermomechanical analysis (DTMA), as a non-invasive method for evaluation of polymerization processes and their effectiveness. Therefore, this method seems to be appropriate for analysis of the properties of composite materials.

Many research works are performed on various mechanical properties and their possible changes. Rouhollahi et.al. [25] tested the correlation between the thickness of a polymerization layer and microhardness of composite materials. They have shown that polymerization of a 3 mm thick layer allows to obtain necessary properties for a clinical use of composite materials, but the best results of microhardness in the Vickers test were obtained for a 2 mm layer. Voltarelli et.al. [26] and Uhla et.al. [27] determined the correlation between a method of polymerization of a composite material and its microhardness. In both research projects, after accelerated ageing of a material, the best results were obtained for halogen LCU. The samples polymerized with a xenon LCU, right after the curing, obtained the highest readings in the microhardness test, but after ageing their hardness decreased significantly. Bauer et.al. [28] showed that polymerization of composite materials should not be shorter than 20 seconds. Only after this time it is possible to obtain satisfying results both in a 3-point bending test and the Vickers hardness test. Those results were also proven by Bhamra et.al. [29], who had shown that the application of a required level of irradiance was necessary for proper polymerization and an increase in the dose did not improve mechanical properties of the material significantly.

Previous research works have shown that longer exposure times result in increased cure depth of a composite resin, higher hardness and a better conversion degree [30-32]. Our own results show that hardness of the Herculite XRV dental composite material increased proportionally with time of exposure, regardless of the LCU type used. In case of halogen LCU, the highest hardness was obtained for 40 s of exposure time and for LED LCU the highest hardness was obtained for 50 s exposure time. The hardness of Filtek Silorane dental composite increased with the exposure time in the whole range from 10 s to 70 s. Comparing both materials, higher hardness was obtained for Filtek Silorane dental composite material polymerized with LED LCU. It may result from the content of oxirane groups in the Filtek Silorane composite material which increases the hardness of the cured composite material.

The analysis of the results shows significant correlation between the distance from the light source and obtained hardness of the dental composite material – samples which

were cured directly on the surface had higher hardness. An increased distance from the LCU, both for halogen and LED LCU, results in a decrease of hardness of the polymerized dental composites. The lowest hardness is observed for the Herculite XRV dental material polymerized from the distance of 8 mm.

Our results have shown that hardness of the tested composite materials is between 18 and 55.8 HK and have proved the conclusions of some previous research [33-36].

6. Conclusions

1. Hardness of Herculite XRV dental composite material increased with the exposure time, regardless of the LCU type (halogen or LED). In case of halogen LCU maximal hardness was achieved after 40 s of exposure, while for LED LCU it was observed after 50 s of exposure.
2. The longer the distance from the light source, both for halogen and LED LCU, the lower the hardness of Herculite XRV dental composite material. A significant decrease in hardness was observed for the distance of 8 mm from the light source.
3. Hardness of the Filtek Silorane dental composite material increased with exposure time ranging from 10 s to 70 s. The highest values of hardness were observed when the composite material was light-cured directly on the sample surface.
4. The application of LED LCU allows to obtain higher hardness readings both for Herculite XRV and Filtek Silorane composite materials when compared to halogen LCU.
5. Filtek Silorane composite material achieves higher hardness readings, regardless of the LCU type used.

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