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Degree of conversion of dental composite materials in relation to different light-curing parameters

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ABSTRACT

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

Design/methodology/approach: The article provides an insight on factors influencing conversion degree of composite materials. Standardized samples made of Herculite XRV based on a methacrylate resin and Filtek Silorane based on silorane resin and were cured using two types of Light Curing Units (LCUs) – halogen and LED. The samples were cured at different distances and for different times.

Findings: Research has showed that the polymerization of Filtek Silorane composite is significantly slower than polymerization of Herculite XRV composite. Extending exposure time does not compensate for decrease of light intensity caused by increase of the distance of the light source from the surface of cured composite.

Research limitations/implications: Further studies on degree of conversion of dental composite materials will allow to expand the knowledge on characteristics of materials used in dental clinical practice.

Practical implications: Evaluation of curing methods, curing parameters and good knowledge on units used in light-curing of composite materials allow to acquire filling materials with best functional qualities.

Originality/value: The paper presents degree of conversion of composite materials based on different matrixes and cured with different methods.

Keywords: Composites; Light-curing; Silorane; Methacrylate; Degree of conversion

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MATERIALS

1. Introduction

Dental composites have been widely used as direct and indirect materials for teeth restorations. These materials are composed of an active organic matrix and inorganic fillers to ensure material's properties [1].

Commercial dental composite most often contains a mixture of various dimethacrylates, glass- and/or silicon dioxide fillers and a photoinitiator system [2]. Because of high functional qualities Bis-GMA/TEGDMA and UDMA/TEGDMA are the most popular matrixes in dental composites. Composite materials with silorane matrix gradually gain on popularity.

The properties of dental composites are influenced by many factors: mechanical, physical and chemical properties of the monomer, type of the filler and the coupling agent, size and concentration of filler particles and method of photocuring process including the intensity of curing light, exposure time and distance to the light source [3-7].

Optimum curing conditions for dental composite material, including sufficient curing time and irradiance level, precise positioning of the light curing unit (LCU) over the restoration site and applying the composite in thin layers, are necessary for acquiring a resin that is properly cured [8-10].

Incomplete polymerization of dental composite materials results in increased amount of unreacted monomers that may penetrate the matrix and be released into oral cavity [11].

Physical and mechanical properties of photo-cured composites directly depend on the degree of conversion of polymerized materials. The degree of conversion (DC) is determined as a ratio of the aliphatic C=C double bonds in a polymerized sample to the total number of C=C bonds in the uncured material [12].

Degree of conversion impacts directly on the properties of dental composites. Because of that valuation of DC is essential [13, 14]. The degree of conversion of popular composites is measured as a ratio of vinyl stretching band (C=C) found at wavelength of 1635cm^{-1} in cured and uncured composites [15, 16].

Fourier transform infrared (FTIR) spectroscopy is a technique used to determine the DC [17-19]. Proper preparation is necessary for the FTIR spectroscopy in order to obtain appropriate spectral data that will provide an accurate evaluation of the conversion degree [13, 20-24].

Many studies on conversion degree of composite materials with methacrylate matrixes showed the need to compare the properties of those materials to silorane-based composite materials [25-27].

The FTIR spectroscopy was performed to compare the degree of conversion of cured organic matrix to uncured one in dental composites with different matrixes and under different exposure parameters.

To test the correlation coefficient besides cured samples, uncured samples of methacrylate-based material Herculite XRV and silorane-based material Filtek Silorane were used.

The correlation coefficient shows the correlation between spectra of uncured and cured samples.

The lower the correlation coefficient, the higher the degree of conversion of functional groups in cured material.

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 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/cm^2 and SmartLite LED LCU (DENTSPLY), 5W LED with maximal irradiance of 950 mW/cm^2. 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 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 determines the time of light-curing in seconds,
- HAL – dental composite cured with halogen LCU,
- LED – dental composite cured with LED LCU.

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

The spectra of cured samples of dental composites were made with use of spectrometer FTIR Spectrum 100 by PerkinElmer. Spectrometer was connected to a computer, which allowed for fast and precise preparation of IR spectrums of dental composites. All results acquired in the measurements of IR spectrum were used to create spectra database, used for automatic evaluation and comparison of acquired IR spectra.

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 population. 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 differences test: t-Student or U Mann-Whitney test. Significant differences between more than two groups were tested with F (ANOVA) or Kruskal-Wallis test (in case of not complying with 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

It was noticed that polymerization of silorane-based composite material (Filtek Silorane) cured with LED LCU directly at the surface of the sample for up to 30 s proceeds slowly, which is indicated by slight change of correlation coefficient for those curing times (Fig. 1).

Table 1.
Correlation coefficients between chemical structures of uncured and cured samples of dental materials with different matrixes in relation to different light-curing parameters

Material sample	Correlation coefficient
uncured	1.0000
FS-0-10-LED	0.9911
FS-0-20-LED	0.9893
FS-0-30-LED	0.9737
FS-0-40-LED	0.9499
FS-0-50-LED	0.9275
FS-0-60-LED	0.8891
uncured	1.0000
FS-0-10-HAL	0.9958
FS-0-20-HAL	0.9905
FS-0-30-HAL	0.9811
FS-0-40-HAL	0.9669
FS-0-50-HAL	0.9610
FS-0-60-HAL	0.9433
uncured	1.0000
H-0-10-LED	0.9586
H-0-20-LED	0.9315
H-0-30-LED	0.9152
H-0-40-LED	0.8862
H-0-50-LED	0.8647
H-0-60-LED	0.8432
uncured	1.0000
H-0-10-HAL	0.9975
H-0-20-HAL	0.9888
H-0-30-HAL	0.9863
H-0-40-HAL	0.9082
H-0-50-HAL	0.8867
H-0-60-HAL	0.8661
uncured	1.0000
H-6-10-LED	0.9936
H-6-20-LED	0.9914
H-6-30-LED	0.9828
H-6-40-LED	0.9824
H-6-50-LED	0.9163
H-6-60-LED	0.8556
uncured	1.0000
H-8-10-HAL	0.9942
H-8-20-HAL	0.9930
H-8-30-HAL	0.9911
H-8-40-HAL	0.9902
H-8-50-HAL	0.9894
H-8-60-HAL	0.9887
uncured	1.0000
FS-4-10-LED	0.9993
FS-4-20-LED	0.9950
FS-4-30-LED	0.9879
FS-4-40-LED	0.9762
FS-4-50-LED	0.9246
FS-4-60-LED	0.8971

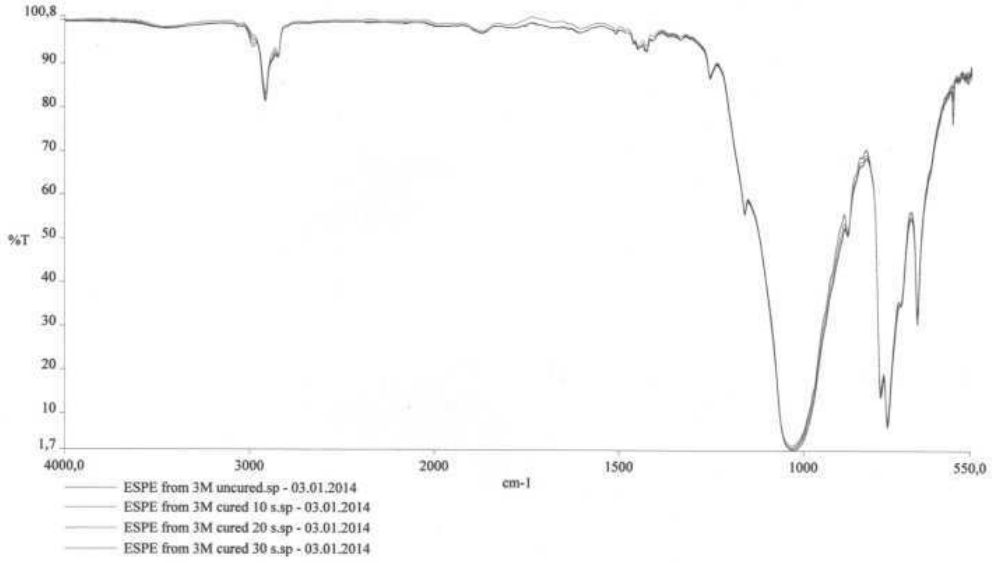

Fig. 1. The IR spectra of samples of silorane-based material (Filtek Silorane) cured with LED LCU directly at the surface of the material for 10s, 20s, 30s and uncured sample

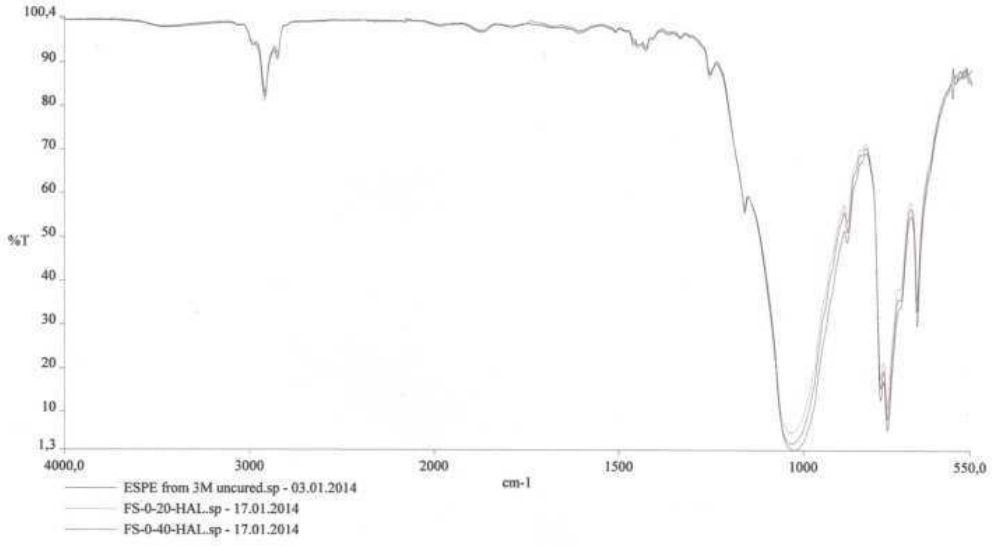

Fig. 2. The IR spectra of samples of silorane-based material (Filtek Silorane) cured with halogen LCU directly at the surface of the material for 20s and 40s and uncured sample

Decline of correlation coefficient was noticed for silorane-based material (Filtek Silorane) cured with halogen LCU directly at the surface of the sample for 40 s (Fig. 2). For curing time of up to 30 s the change of correlation coefficient is similar to the change of correlation coefficient of silorane-based material (Filtek Silorane) cured with LED LCU also directly at the surface of the sample. This implies that type of LCU used for curing of silorane-based material (Filtek Silorane) does not influence the rate of polymerization process.

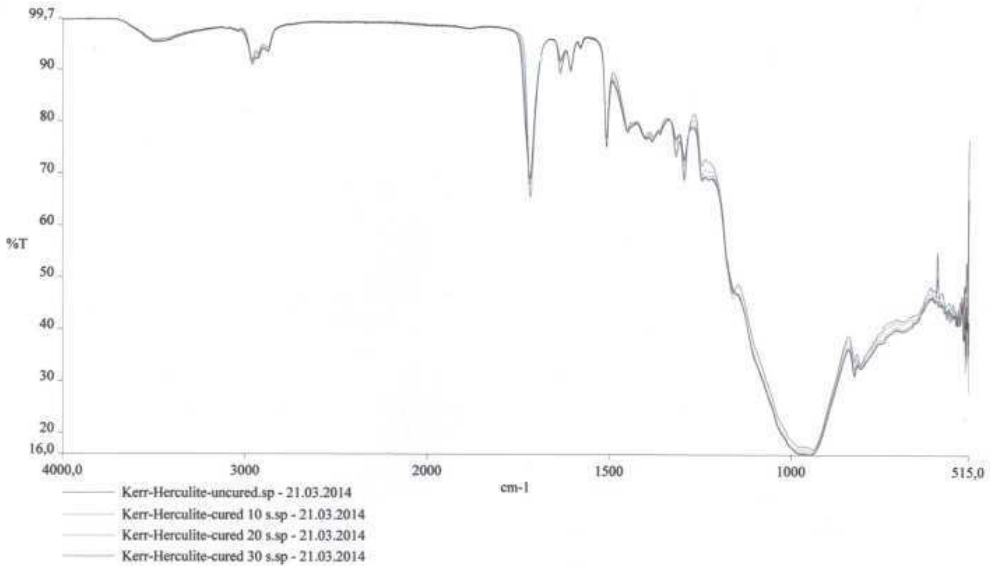

Fig. 3. The IR spectra of samples of methacrylate-based material (Herculite XRV) cured with LED LCU directly at the surface of the material for 10s, 20s and 30s and uncured sample

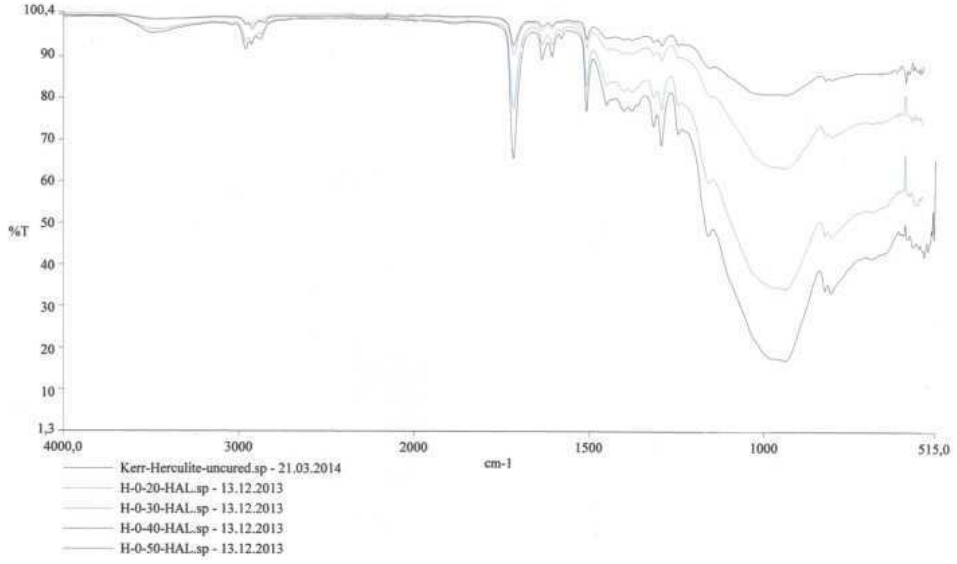

Fig. 4. The IR spectra of samples of methacrylate-based material (Herculite XRV) cured with halogen LCU directly at the surface of the material for 20s, 30s, 40s and 50s and uncured sample

Significant decline of correlation coefficient was noticed for samples of methacrylate-based material (Herculite XRV) cured with LED LCU directly at the surface of the sample for 30 s. This indicates rapid rate of polymerization process, resulting in fast curing of composite material. It may be a result of significant decrease of amount of acrylic monomers contained in methacrylate-based material (Herculite XRV).

With extension of exposure time to 50 s, gradual decline of correlation coefficient was noticed for methacrylate-based material (Herculite XRV) cured with halogen LCU directly at the surface of the sample

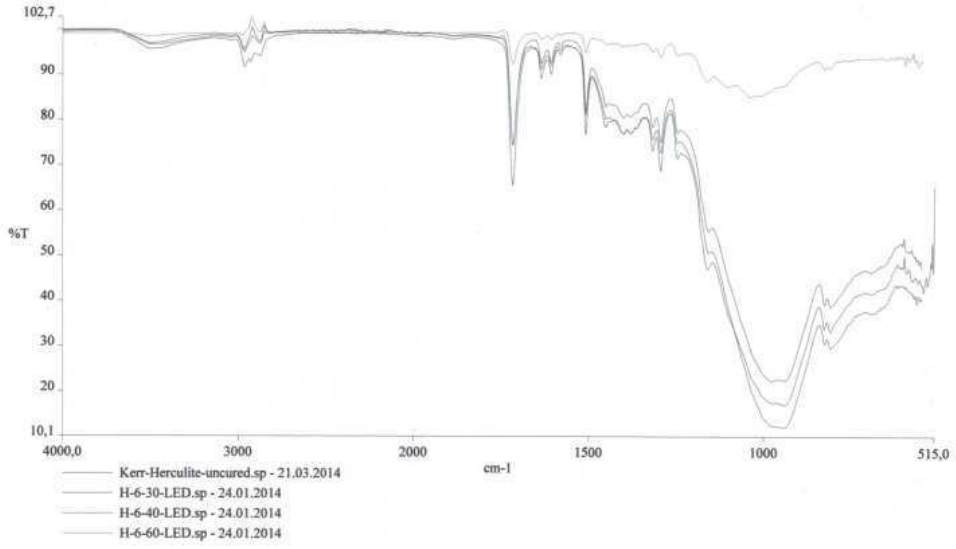

Fig. 5. The IR spectra of samples of methacrylate-based material (Herculite XRV) cured with LED LCU at the distance of 6mm from the surface of the sample for 30s, 40s and 60s and uncured sample

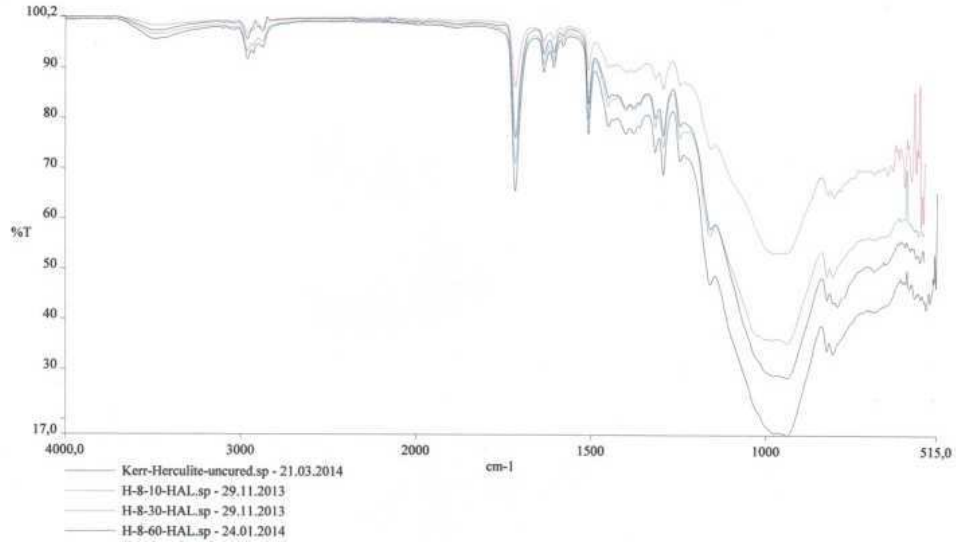

Fig. 6. The IR spectra of samples of methacrylate-based material (Herculite XRV) cured with halogen LCU at the distance of 8mm from the surface of the sample for 10s, 30s and 60s and uncured sample

Significant decline of correlation coefficient was noticed for methacrylate-based material (Herculite XRV) cured with LED LCU at the distance of 6 mm after reaching 40 s. Increase of curing rate implies increase of reactivity of radicals of unsaturated acrylic groups.

Methacrylate-based material (Herculite XRV) cured with halogen LCU at the distance of 8 mm for up to 60 s, polymerized at a slow rate, which shows that extensive distance of the light-source from the surface of the material during the curing can't be compensated by extended time of curing.

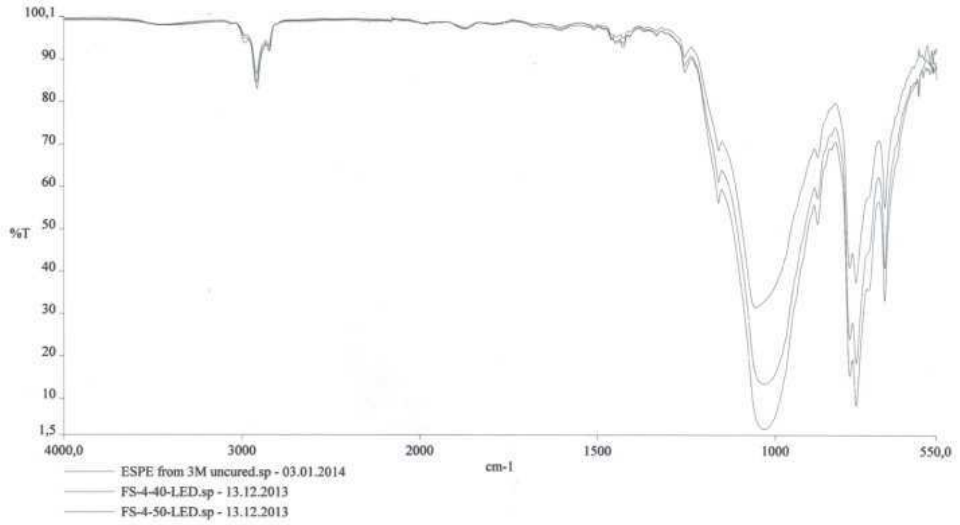

Fig. 7. The IR spectra of samples of silorane-based material (Filtek Silorane) cured with LED LCU at the distance of 4mm from the surface of the sample for 40s and 50s and uncured sample

Significant decline of correlation coefficient can be noticed for silorane-based material (Filtek Silorane) cured with LED LCU at the distance of 4 mm for more than 40s. This indicates increase of curing rate of silorane-based material (Filtek Silorane) because of cationic polymerization of oxirane groups contained in the composite.

5. Discussion

FTIR spectroscopy is a commonly used method for investigation of materials in gaseous, liquid or solid phase. It evaluates the interaction between electromagnetic radiation and vibrations of the chemical bonds among atoms [12].

Degree of conversion of dental composite materials results from the monomer reaction during the polymerization [28].

Insufficient polymerization of a material can result in a deterioration of mechanical strength and lower resilience to degradation, increase of marginal leakage and occurrence of recurring caries as a result of bacterial leakage [29, 30]. By focusing on the polymerization process it is possible to achieve proper cross-linking of composite matrix resulting in increase of physiochemical and mechanical properties of a material and increase of clinical durability of filling [29, 31-33]. Research on mechanical properties and possible modifications is important for improvement of quality of composite fillings. Such studies are conducted by producers of composite materials to improve and perfect the materials by implementing changes in resin and filler composition. However, conditions in oral cavity greatly differ from conditions of in vitro studies [30, 34].

Latta at al. have used FTIR to estimate the DC of two commercial composites at three different depths of polymerization and noticed that the DC decreased significantly with increasing depth of cure [35]. Mills et. al. [36] noticed better depth of polymerization in materials cured with LED LCU comparing to materials cured with halogen LCU using similar light intensity (290mW/cm^2 for LED LCU and 300mW/cm^2 for halogen LCU).

Carried out research showed that the polymerization of Filtek Silorane composite is significantly slower than polymerization of Herculite XRV composite. It is a result of presence in the silorane composite of oxirane groups, less reactive in the cationic polymerization. Highest conversion of double bonds (Herculite XRV) and highest conversion of oxirane groups (Filtek Silorane) was noticed for samples cured directly at the surface.

Murdock et al. have determined the Barcol hardness and DC (by FTIR) of two commercial dental composites at different depths and exposition time, concluding that both caused changes in DC [37]. In the study of three dental composites, Neves et al. have noticed a relationship between DC (by FTIR) and Vickers hardness [38].

Ruyter and Oysaed studied the influence of visible and ultraviolet light on the polymerization of dental composites

at different curing depths. Using FTIR, they noticed that DC was higher for composites cured with visible light [39]. Arikawa et al. [40] and Price et al. [41] found a correlation between the distribution of irradiance and the hardness of composite materials.

Oréfice et al. [42] have studied polymerization kinetics and mechanical properties of a specific commercial dental composite. They have noticed a good relationship between degree of conversion and Vickers microhardness, under specific polymerization time.

In our research faster rate of curing process was noticed for Herculite XRV composite rather than for Filtek Silorane composite cured directly at the surface of the samples.

Various researches conclude that depth of cure is influenced by many factors, including intensity and spectrum of light and time of curing [43-47]. Absorption and dispersion of light by filler particles can decrease light transmission in the material. Important factors influencing the depth of polymerization are: light transmission through composite material and light intensity emitted by LCU. During the light-curing of dental composites, highest light intensity is observed directly at the surface of the material, decreasing gradually with increasing layers of composite material [44]. During restoration of deep cavities, when the distance of light source from the surface of the composite is significant, it is necessary to take into account possibility of insufficient polymerization.

Our research showed that faster rate of polymerization process of Herculite XRV composite comparing to Filtek Silorane composite may cause increased polymerization shrinkage of the methacrylate-based composite.

Carried out research showed that longer exposure times does not compensate for decrease of light intensity caused by increase of the distance of the light source from the surface of cured composite. It was observed that curing of Herculite XRV composite with halogen LCU at the distance of 8mm resulted in low conversion degree of multifunctional acrylic monomers. Decline of light intensity caused by the increased distance of halogen LCU from the surface of Herculite XRV composite material had inhibited the process of polymerization. Extending the time of curing to 60s did not increase the rate of polymerization of Herculite XRV composite.

6. Conclusions

Extending exposure time does not compensate for decrease of light intensity caused by increase of the distance of the light source from the surface of cured composite.

Polymerization of Filtek Silorane composite is significantly slower than polymerization of Herculite XRV composite.

The highest conversion of double bonds (Herculite XRV) and the highest conversion of oxirane groups (Filtek Silorane) was noticed for samples cured directly at the surface.

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