

Influence of photopolymerization parameters on the mechanical properties of polymer-ceramic composites applied in the conservative dentistry

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In this paper, the results of study of mechanical properties for four commercial polymer-ceramic composites applied in the conservative dentistry are presented, including one new silorane based composite and three standard composites based on methacrylate compounds. Influence of the type of light of diode and halogen polymerization lamps on the microhardness, flexural strength and elasticity were studied. Both exposed and unexposed specimens were taken into account. An exposure time was also differentiated (40 sec and 60 sec). Basic statistics of the analysed material parameters were determined. A post hoc test (Newman-Keuls) was performed in order to evaluate differences between microhardness of the materials studied, as well as Kruskal-Wallis test to evaluate differences in flexural strength and elasticity modulus of the material. It has been indicated that there is an impact of the type of lamp on the microhardness and flexural strength of composites with methacrylate matrix and lack of such impact in the case of composites containing siloranes. Additionally, it has been found that an increase of photopolymerization time has a significantly different impact on the mechanical properties depending on the type of irradiated material.

Key words: materials for dental fillings, polymer-ceramic composites, flexural strength, microhardness

1. Introduction

Nowadays, composite materials applied for dental restorations are increasingly better adapted to the conditions in human oral cavity. Besides the typical requirements for such materials, e.g., good aesthetic properties, ease development, X-ray contrast, mechanical strength and resistance to wear, it is necessary to ensure the minimum polymerization shrinkage and appropriate elasticity. The last two characteristics

reflect formation of marginal fissure in the filling-tooth hard tissue system [1]. The marginal fissure leads to bacterial leakage and results in secondary caries formation and clinical degradation of the filling [2]. Thus, verification of quality of the new composite dental materials and selection of photopolymerization procedures requires cooperation between material and mechanical engineering experts with the dentists.

From the clinical point of view a promising material group with minimized polymerization shrinkage are polymer-ceramic composites, where silica micro-

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particle or nanoparticle fillers, as well as siloranes are used as a matrix material. Such materials have a hybrid matrix in a half made of silorane particles and oxiranes [3], [4]. It is well known that composites with silorane matrix are characterized by suitable mechanical properties from the mechanical point of view. However, they are not well examined in terms of operational factors [3]–[5].

A photopolymerization procedure has a significant impact on the physical-mechanical properties of polymer-ceramic composites. Intensity and light spectrum plays an important role, as well as the exposure time [7], [8]. The impact of photopolymerization parameters on the mechanical properties of composites has been presented in [9]–[13]. Versluis [14] drew attention to the problem of microhardness local gradient in the lamp impact area. Nowadays a significant progress in the development of polymerization lamps technology can be observed. In dental practise, besides halogen lamps, the diode lamps are commonly applied. Modern lamps using electroluminescent diodes are characterised by the following: low energy consumption, moderate radiation intensity and very high durability. In the literature a large number of reports on the polymerization lamp selection with regard to mechanical properties of light-curing composites can be found [1], [8], [15]–[20]. However, there is not much information regarding new composites with silorane matrix.

The aim of the studies presented in this paper was to investigate the influence of different polymerization procedures (type of lamp, exposure time) on the mechanical properties and anisotropy of the new dental composite, containing siloranes, comparing to three microhybrid composites based on methacrylates.

2. Materials and methods

Three composites based on methacrylate compounds were studied, with the following trade names: Gradia Direct Anterior (GDA), Gradia Direct Posterior (GDP) and Herculite XRV (H), and new composite with silorane matrix – Filtek Silorane (FSi).

Physical parameters of the materials studied are given in Table 1.

Filtek Silorane is an innovative composite with silorane matrix and silica filler. It is characterized by low polymerization shrinkage of approx. 1.5% and flexural strength of 120 MPa [3]. Gradia Direct Anterior and Gradia Direct Posterior are materials based on methacrylates. They have moderate mechanical properties but they stand out good clinical properties and they have been commonly applied in the dental practise in recent years. Herculite XVR is a traditional composite based on methacrylates with good mechanical properties (elasticity modulus E-10,1 MPa) [21].

In the present study photopolymerization with two types of light was applied: L.E.Demetron 1 (SDS/Kerr) diode lamp and Astralis 7 (Ivoclar Vivadent) halogen lamp. Technical data of lamps are given in Table 2.

The exposure times of 40 sec and 60 sec were applied. Time of 40 sec is recommended by the materials' manufacturer. The authors introduced experimentally the time of 60 sec based on the literature reports [8], [16], [17], where it is suggested that the extension of the exposure time can improve polymerization efficiency by the increase of conversion degree. The degree of conversion is how much percent of double bonds in the polymer structure have been satu-

Table 1. List of composites studied

Composite	Manufacturer	Type	Filler content (wt%)	Filler particles size
Filtek Silorane (FSi)	3M ESPE	Silorane	76	0.1–1 μm 0.04–1.7 μm
Gradia Direct Ant (GDA)	GC	Microhybrid	73	Average 0.85 μm
Gradia Direct Post. (GDP)	GC	Microhybrid	77	Average 0.85 μm
Herculite XRV (H)	Kerr	Microhybrid	79	0.6 μm

Table 2. Specification of the applied photopolymerization lamps

Lamp	Type	Manufacturer	Wave length [nm]	Power density [mW/cm ²]
L.E.Demetron 1 (LED)	LED	SDS/Kerr	450÷470	200÷800
Astralis 7 (HAL)	Halogen	Ivoclar-Vivadent	400÷510	750

rated (converted to a single bond) [22]. Depending on the photopolymerization process conditions the degree of conversion is in a range between 65% and 80% [19], [23].

The work was divided into two parts:

- microhardness studies,
- flexural strength and elasticity modulus studies.

2.1. Microhardness studies

Microhardness study allows indirect evaluation of some important composite properties from the clinical point of view. As was experimentally demonstrated [1] there is a correlation between composite microhardness and value of polymerization shrinkage. Moreover, it has been found that there is a relationship between microhardness and composite restoration wear in *in vitro* simulation conditions [23]. The microhardness study can also be used for evaluating local photopolymerization gradient, i.e., inhomogeneities of composite properties in the lamp impact area [14].

In the presented work for microhardness studies eight specimens from each material were prepared, with the shape of disc with a diameter of 10 mm and thickness of 1 mm. A group of specimens from each material was divided into subgroups of 2 specimens assigned to the type of lamp and specified exposure time. Microhardness measurements were made with Vickers method by means of Futertech FM 700 device (Future-tech Corp. Japan), with loading of 50 g. Penetration time of indenter was set to 15 sec. The measurements were performed at 10 points on each specimen surface studied. Measurement coordinates were set possibly to cover the whole surface of the specimen. They were the same for all specimens. The test was carried out on both exposed (lc) and unexposed (nlc) specimen surfaces.

2.2. Flexural strength studies

For the strength studies a TFS test (Three Point Flexural Strength) was applied. Three point flexural test is recognized by the International Organization for Standardization as applicable in dental strength studies of polymer composites [24]. From each material 40 specimens in a form of rectangular prismatic beam were prepared, with dimensions of 2 mm × 2 mm × 25 mm according to PN-EN ISO 4049. A set of specimens from each material were divided into subgroups with 10 specimens assigned to the type of lamp and specified exposure time.

3. Results

3.1. Microhardness

Microhardness test results are presented in Tables 3 and 4. The results were grouped based on: material (GDA, GDP, FSi, H), type of lamp (HAL, LED), exposed and unexposed surface (LC, NLC). The following were specified: size of basic measurement subgroup N , average value, standard deviation, and coefficient of variation. Filtek Silorane microhardness increased after extension of the exposure time with diode lamp (LED), while remained unchanged after extension of the exposure time with halogen lamp. A similar tendency was found for Herculite XRV material. FSi material was characterized by higher hardness compared to studies with methacrylates based composites GDA and GDP. Additionally, FSi showed comparable flexural strength and elasticity modulus to H material.

Analysing impact of the type of lamp it should be noticed that the new material FSi based on siloranes showed the highest microhardness, compared to methacrylate based composites, at a level of 51–52 MHV and exposition to halogen lamp for 40 sec. In case of this material the extension of exposure time to LED lamp caused increased microhardness, while the exposure time to HAL lamp had no significant influence on the microhardness.

The lowest microhardness was found for GDA (25 MHV) and GDP (from 33 to 35 MHV). The extension of exposure time to LED lamp had a positive effect on microhardness of GDA material, however it had a negative impact on GDP microhardness. The exposure time to HAL lamp had a slight and ambiguous effects for both materials.

In the case of H material the average microhardness value was between 36 MHV and 43 MHV. The extension of exposure time both with LED lamp and HAL lamp had a positive effect on microhardness of this material. Microhardness of H material was in a range of 36 to 43 MHV. A significant impact of the exposure time when using LED lamp has been demonstrated. Considering different microhardness values between exposed LC and unexposed NLC surfaces a statistical significance of differences was analysed using post hoc Newman–Keuls test. Test results in most cases for Gradia Direct Anterior and Gradia Direct Posterior materials demonstrated no significant differences between LC and NLC surfaces. For H material significant difference was observed between microhard-

Table 3. Descriptive statistics of composite microhardness exposed to halogen lamp.
CT – exposure time, LC – exposed surface, NLC – unexposed surface

Composite	Lamp	CT	S	N	Mean	Std. dev.	C. Var. %
GDA	HAL	40 s	LC	20	25.70	0.95	3.68
			NLC	20	29.43	1.49	5.07
		60 s	LC	20	28.20	1.18	4.18
			NLC	20	27.74	0.98	3.52
H	HAL	40 s	LC	20	38.64	1.60	4.14
			NLC	20	38.88	1.55	3.98
		60 s	LC	20	41.64	0.78	1.87
			NLC	20	39.36	1.76	4.48
GDP	HAL	40 s	LC	20	34.12	2.09	6.11
			NLC	20	34.03	1.94	5.70
		60 s	LC	20	33.77	1.17	3.45
			NLC	20	35.44	1.68	4.74
FSi	HAL	40 s	LC	20	52.25	2.21	4.23
			NLC	20	50.28	1.64	3.27
		60 s	LC	20	51.68	1.99	3.85
			NLC	20	49.07	2.00	4.07

Table 4. Descriptive statistics of composites microhardness exposed to diode lamp.
CT – exposure time, LC – exposed surface, NLC – unexposed surface

Composite	Lamp	CT	S	N	Mean	Std. dev.	C. Var. %
GDA	LED	40 s	LC	20	26.44	1.44	5.44
			NLC	20	26.02	0.56	2.15
		60 s	LC	20	29.43	1.38	4.69
			NLC	20	29.91	1.44	4.81
H	LED	40 s	LC	20	36.93	1.04	2.82
			NLC	20	36.38	1.07	2.95
		60 s	LC	20	40.14	0.69	1.73
			NLC	20	43.29	1.65	3.80
GDP	LED	40 s	LC	20	35.10	1.53	4.35
			NLC	20	34.50	1.38	3.99
		60 s	LC	20	33.21	1.63	4.89
			NLC	20	33.32	2.06	6.19
FSi	LED	40 s	LC	20	46.51	1.36	2.93
			NLC	20	44.37	1.16	2.62
		60 s	LC	20	53.16	1.40	2.63
			NLC	20	50.31	2.09	4.15

ness on both LC and NLC sides when using LED lamp and HAL lamp after 60 sec. However, no significant differences between LC and NLC were indicated, after using LED lamp for 40 sec. In the case of FSi material no significant differences between results obtained for exposed and unexposed surfaces were shown. Generally, greater differences have been found for diode lamp than for the halogen lamp.

It should be emphasized that the differences between testing options for FSi material are in many cases significant, however in smaller range than in the case of GDA, GDP and H materials. For FSi composite only in four cases no statistically significant differences between results groups were shown, NLC LED 60 sec and NLC HAL 40 sec and NLC LED 60 sec and NLC HAL 60 sec, as well as LC HAL 40 sec and

LC LED 60 sec, and LC HAL 40 sec and LC HAL 60 sec. For the material based on methacrylates – GDA in seven cases no differences were indicated, whereas for H material no differences were observed in five cases.

3.2. Flexural strength and modulus of elasticity

In Tables 5 and 6, descriptive statistics of flexural strength and modulus of elasticity are presented. The highest average flexural strength was demonstrated for FSi material after using halogen lamp for 60 sec (120 MPa). The values are consistent with the ones

Table 5. Statistics of flexural strength study results

Composite	Lamp	CT	N	Mean	Std. dev.	C. Var.
GDA	HAL	40 s	10	75.78	5.87	7.74
		60 s	10	81.46	4.51	5.53
	LED	40 s	10	61.88	5.94	9.60
		60 s	10	64.30	11.89	18.49
H	HAL	40 s	10	112.37	10.72	9.54
		60 s	10	107.22	13.62	12.70
	LED	40 s	10	102.63	12.22	11.90
		60 s	10	105.94	8.14	7.68
GDP	HAL	40 s	10	84.83	3.66	4.32
		60 s	10	82.25	6.10	7.41
	LED	40 s	10	75.00	8.61	11.48
		60 s	10	68.72	9.16	13.33
FSi	HAL	40 s	10	111.08	8.08	7.28
		60 s	10	120.09	11.97	9.96
	LED	40 s	10	107.00	9.78	9.14
		60 s	10	106.54	11.34	10.65

Table 6. Statistics of modulus of elasticity study results

Composite	Lamp	CT	N	Mean	Std. dev.	C. Var.
GDA	HAL	40 s	10	4.24	0.25	5.96
		60 s	10	4.54	0.22	4.86
	LED	40 s	10	4.33	0.25	5.76
		60 s	10	5.05	0.26	5.26
H	HAL	40 s	10	8.49	0.20	2.39
		60 s	10	8.93	0.49	5.48
	LED	40 s	10	8.18	0.46	5.63
		60 s	10	8.59	0.37	4.35
GDP	HAL	40 s	10	5.43	0.25	4.69
		60 s	10	6.01	0.31	5.12
	LED	40 s	10	5.65	0.20	3.49
		60 s	10	6.29	0.41	6.59
FSi	HAL	40 s	10	7.30	0.64	8.80
		60 s	10	7.40	0.65	8.82
	LED	40 s	10	8.08	0.55	6.81
		60 s	10	8.30	0.68	8.23

reported in the literature [3]. The lowest average flexural strength was obtained for GDA material after using diode lamp for 40 sec (62 MPa). In the case of all materials studied higher strength values were observed for halogen lamp, while extension of exposure time to 60 sec had no impact on the strength increase.

The highest average modulus of elasticity was obtained for H material after using halogen lamp and exposure time of 60 sec (8.93GPa). The lowest average modulus of elasticity was observed for GDA material after using halogen lamp and exposure time of 40 sec (4.24 GPa). In most cases the increase of modulus of elasticity was observed after using diode lamp. Only in one case of H material such dependence was not demonstrated. Additionally, it has been shown that the increase of modulus of elasticity was favoured by the longer exposure time.

In order to evaluate a statistical significance of the impact of photopolymerization parameters on the strength and elasticity modulus of the composites studied a Kruskal–Wallis test (K–W) was applied.

Assuming significance level ($p < 0.05$) the differences between the results of flexural strength studies were classified. For GDA material differences between LED and HAL groups were considered as significant, whereas differences related to time were not considered. For H material no statistically significant differences between the results were observed, meaning that in the case of this material impact of the exposure time and type of lamp was negligible. In the case of GDP material a significant differences between strength in the groups HAL 60 sec and LED 60 sec, as well as HAL 40 sec and LED 40 sec were demonstrated. Regarding FSi material no statistically

significant differences between different groups were observed.

The K–W test in many cases has indicated large differences between values of modulus of elasticity. For GDA material significant differences were observed between results in LED group when changing exposure time, and differences between groups LED 60 sec and HAL 40 sec. In the case of H material significant differences between LED 40 sec and HAL 60 sec were observed. For GDP material significant differences were shown between the following groups: HAL 40 sec and HAL 60 sec, HAL 40 sec and LED 60 sec, LED 40 sec and LED 60 sec. Whereas for FSi material no significant differences for modulus of elasticity were shown.

4. Discussion

Nowadays, different photopolymerization technologies of light-cured materials are being applied. Commonly used are halogen lamps and diode lamps, which were developed as an alternative. The diode lamps are applied due to some drawbacks of the halogen lamps. In halogen lamps as a result of heat loss only 10% of energy is converted to visible spectra, and due to the applied filters only 1% of the total lamp energy is utilized in the blue light stream. Moreover, lower temperature occurring in the oral cavity during photopolymerization process with LED lamp has a significant impact on the patient's comfort [26]. However, the most commonly applied are standard halogen and diode lamps. Many publications report that LED lamps allow obtaining mechanical properties of composite close to the one obtained with HAL lamp at lower radiation intensity [27], [28]. The studies presented not fully support such dependence. In most cases mechanical properties after using LED lamp were significantly poorer. The slightest differences have been indicated for FSi material based on siloranes.

Flexural strength is very important due to stresses arising in the filling structure, being a result of the chewing process. Microhardness is an important measure of the surface mechanical properties, the materials should be resistant to impact of food particles and processes occurring due to contact of the opposing teeth. Surface properties should be in a particular specified range to enable the appropriate food fragmentation, and should not impact wearing of the opposing teeth.

Flexural strength testing according to ISO4049 requires application of specimens with dimensions con-

strain repeated irradiation of the same specimen. Such a method of preparation reflects inhomogeneity of specimen properties, because some irradiations overlap. Furthermore, in the areas of beams irradiated as first a shrinkage occurs that generates stresses, resulting in damage in unpolymerized part [29]. Due to these processes the obtained flexural strength values and modulus of elasticity do not fully represent real properties of composite. The low values of flexural strength can indirectly indicate increased susceptibility to shrinkage and shrinkage stresses. FSi composite due to its structure and polymerization method is less susceptible to the above phenomena. In the studies presented this composite indicated the highest flexural strength. In the materials based on methacrylate system, a light-initiated polymerization procedure leads to a change of double bonds of material molecules to single ones, which contributes to reduction of volume and polymerization shrinkage. It is considered that using pre-polymerized molecules reduces the number of double bonds in the composite structure, reflecting hybrid materials to be more inhomogeneous, but indicates lower polymerization shrinkage and better mechanical characteristics. Studies confirmed a similar strength and elastic properties only in the case of one methacrylate microhybrid composite H and composite based on siloranes. A stiffness of H composite was the highest. Whereas strength of GDA composite is lower than permitted value specified in ISO4049 standard, however this composite is applied in the anterior.

5. Conclusion

1. It has been indicated that a change of polymerization lamp from halogen to diode in the case of Filtek Silorane composite based on siloranes, has no significant impact on its flexural strength, while it slightly improves its elasticity, but at the same time reduces its microhardness. A comparable influence has been demonstrated for standard Herculite XRV material, with no change in modulus of elasticity. In the case of the remaining materials based on methacrylates (Gradia Direct Anterior, Gradia Direct Posterior), application of the diode lamp reduces flexural strength, but improves modulus of elasticity (after extending the exposure time) and has no impact on the microhardness.
2. It has been demonstrated that the extension of the exposure time with halogen lamp for Filtek Silorane, improves its flexural strength, but has no impact on the elasticity and microhardness. Re-

spectively, in the case of Herculite XRV material, flexural strength and elasticity remain unchanged, while microhardness increases. Regarding GDA and GDP materials the following has been concluded: no impact of the exposure time on the strength, slight positive impact on the modulus of elasticity, and slight but ambiguous impact on microhardness.

3. It has been shown that the extension of exposure time with diode lamp in the case of FSi material has no influence on the strength and elasticity, whereas significantly increases microhardness. A similar impact has been observed for Herculite XRV material. For GDA and GDP no impact of the extension time on the strength, positive impact on the modulus of elasticity, and varied impact on the microhardness have been shown.
4. Based on the studies conducted it can be concluded that for the materials based on silorane compounds application of the LED lamp is more advantageous.

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