

Agata M. NIEWCZAS  
Daniel PIENIAK  
Paweł OGRODNIK

## RELIABILITY ANALYSIS OF STRENGTH OF DENTAL COMPOSITES SUBJECTED TO DIFFERENT PHOTOPOLYMERIZATION PROCEDURES

### ANALIZA NIEZAWODNOŚCIOWA WYTRZYMAŁOŚCI KOMPOZYTÓW STOMATOLOGICZNYCH PODDANYCH ZRÓŻNICOWANYM PROCEDUROM FOTOPOLIMERYZACJI\*

*Abstract: The aim of this study was evaluation of chosen photopolymerization procedures on strength and reliability of dental composites based on siloranes and composites based on methacrylate compounds in 3-points bending test conditions. The following composites were tested: Filtek Siloran (FSi), Gradia Direct Anterior (GDA), Gradia Direct Posterior (GDP), Herculite XRV (H). Photopolymerization was conducted by means of two types of light: LED lamp and halogen lamp. Exposure times of 40 and 60 seconds were applied. For the strength studies a three-point bending test was used (TFS). Twenty rectangular beam-shaped samples (N=20) from each material were prepared for the studies. For each studied case an average value and standard deviation were determined. To assess significance of differences a variation analysis was performed. Then, the results from each specimen were approximated by two-parameter Weibull distribution. Distribution scale parameter was calculated (as a characteristic strength) and shape parameter (as a material reliability index). It has been demonstrated that in 3-point bending test conditions in case of silorane-based composite the type of lamp has no impact on the strength, however it can improve its reliability. In case of conventional methacrylate-based materials application of LED lamp instead of halogen lamp reduces material strength, but increases its reliability. Additionally, it has been shown that the extension of exposure time – in case of FSi material and halogen lamp, increases material strength, however it has no impact on reliability of the material.*

**Keywords:** reliability, flexural strength, dental composites, photopolymerization.

*Celem badań była ocena wpływu wybranych procedur fotopolimeryzacji na wytrzymałość i niezawodność kompozytów stomatologicznych opartych na siloranach oraz kompozytów opartych na związkach metakrylanowych w warunkach testu na 3-punktowe zginanie. Badano kompozyty o nazwach handlowych: Filtek Siloran (FSi), Gradia Direct Anterior (GDA), Gradia Direct Posterior (GDP), Herculite XRV (H). Zastosowano fotopolimeryzację dwoma rodzajami światła: lampą diodową oraz lampą halogenową. Przyjęto czas naświetlania 40 sek. oraz 60 sek. Do badań wytrzymałości został zastosowany test na zginanie trójpunktowe (TFS). Przygotowano próbki do badań w formie belek prostopadłościennych o liczności N = 20 z każdego materiału. Wyznaczono wartość średnią i odchylenie standardowe dla każdego badanego wariantu. Do oceny istotności różnic przeprowadzono analizę wariancji. Następnie wyniki każdej próby aproksymowano dwuparametrowym rozkładem Weibull'a. Obliczono parametr skali rozkładu (jako wytrzymałość charakterystyczną) oraz parametr kształtu (jako wskaźnik niezawodności materiału). Wykazano, że w warunkach testu na 3-punktowe zginanie rodzaj lampy nie ma wpływu na wytrzymałość w przypadku kompozytu opartego na siloranach, natomiast umożliwia poprawę jego niezawodności. W przypadku konwencjonalnych materiałów opartych na metakrylanach zastosowanie lampy diodowej w miejsce lampy halogenowej obniża wytrzymałość materiału, jednak zwiększa jego niezawodność. Ponadto wykazano, że zwiększenie czasu naświetlania – w przypadku materiału FSi i lampy halogenowej zwiększa jego wytrzymałość, natomiast nie ma wpływu na niezawodność. W pozostałych przypadkach wytrzymałość na ogół pozostaje na stałym poziomie lecz zwiększa się niezawodność materiału.*

**Słowa kluczowe:** niezawodność, wytrzymałość na zginanie, kompozyty stomatologiczne, fotopolimeryzacja.

#### 1. Introduction

Present-day laboratory studies of new biomaterials, such as dental composites, in many cases include not only studies of direct material characteristics but also prediction of preservation of these characteristics in operating conditions. Therefore, investigations of mechanical strength of composites are often extended by reliability analysis consisting in application of Weibull distribution as a failure probability distribution model, and estimation of the distribution parameters [20, 23, 27]. Reliability assessment is one of the components of complex risk analysis at clinical decision making by a dentist [11].

Weibull modulus (shape parameter) is adapted as a variation rate of material strength. A high Weibull modulus  $m$  indicates a potentially higher clinical reliability [4, 15]. The scale parameter of Weibull distribution specifies characteristic value of material strength, which corresponds to 63.2% of cases of failure of the studied material [3, 27]. Generally, a characteristic strength value ( $\sigma_0$ ) depends on the material composition, photopolymerization and failure mechanisms [9].

Mechanical strength assessment of composites is usually per-

(\*) Tekst artykułu w polskiej wersji językowej dostępny w elektronicznym wydaniu kwartalnika na stronie [www.ein.org.pl](http://www.ein.org.pl)

formed based on the bending tests. Three-point bending test (3PBT) is acknowledged by the International Organization for Standardization as a valid test for strength testing of dental polymer composites [9]. Three point bending can be compared to chewing process by molar and premolar teeth with regards to mechanics [8] – fig. 1.

Studies of flexural strength of dental composites can be used among the others for the assessment of photopolymerization effectiveness, especially for evaluation of the effect of polymerization time and type of the lamp [1, 6, 10, 14, 20]. The most often applied lamps for polymerization in dental practice are LED and halogen lamps. The lamps using light-emitting diode are characterized by low energy consumption, moderate radiation intensiveness and very high durability.

Flexural strength test is a very important criterion of the clinical usability of composite materials. It is especially crucial in the context of dynamical development of dental composites, particularly introduction of silica fillers with particle sizes in a range of 0.1 nm to 100 nm, and siloranes as matrix material. Composites made of nano-particles with silorane matrix are characterized by a minimum polymerization shrinkage and fair mechanical

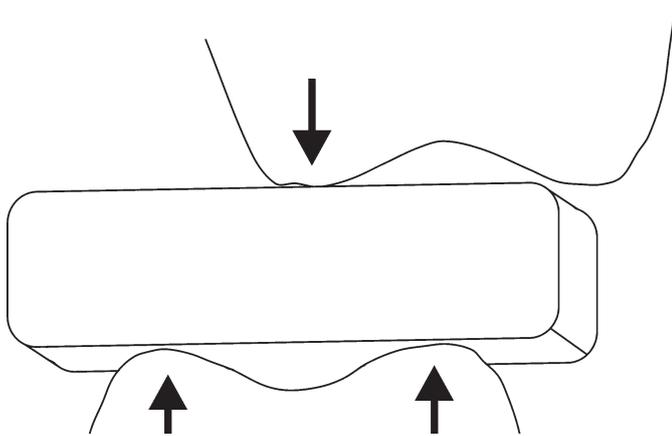


Fig. 1. Food crushing by three-point bending with molar and premolar teeth [8]

properties (microhardness, flexural strength) [12,17,18] and satisfactory resistance to ageing and thermal fatigue [24,25]. However, in literature there is not much information available regarding new composites based on siloranes.

The aim of this study was to determine impact of photopolymerization technology, including exposure time and type of lamp, on the reliability of composites based on siloranes and standard methacrylate compounds, in laboratory flexural strength test conditions.

## 2. Materials and methods

The following commercial composites were studied: Filtek Silorane (3M ESPE) – FSi, Gradia Direct Anterior (GC) – GDA, Gradia Direct Posterior (GC) – GDP, Herculite XRV (Kerr) – H. Composites data can be found in Table 1.

Currently there are many dental light-cured composite materials available on the market. Most of them have a methacrylate matrix, which consists of few chemical compounds. A main group are monomers, such as for example Bis-GMA resin and its derivatives, urethane dimethacrylate and UDMA. The methacrylate matrix includes also comonomers, such as for example: TEGDMA and HEMA, which have a lower molar mass and reduce viscosity of basic resin.

In case of silorane based composites, silorane matrix consists of a hybrid, which is in half composed of silorane particles and oxiranes

[12,17]. Such difference in matrix composition results in opening of silorane rings during polymerization, which causes their straightening and broadening, different from methacrylates, where monomers couple with each other by moving towards each other, resulting in significant volume reduction, and poses negative clinical effects due to polymerization shrinkage [7,12].

In case of both methacrylates and siloranes matrix the filler consists of silica based particles and fluoride aluminum silicate glass particles. Agent binding a resin with inorganic filler is most often organosilicon, vinyl and amine compounds.

Table 1. A list of studied composites

Material	Manufacturer	Type	Filler content (wt%)	Filler particles size
Filtek Silorane (FSi)	3M ESPE	Silorane	76	0,1 – 1 $\mu\text{m}$
Gradia Direct Ant (GDA)	GC	Microhybrid	73	average 0,85 $\mu\text{m}$
Gradia Direct Post. (GDP)	GC	Microhybrid	77	average 0,85 $\mu\text{m}$
Herculite XRV (H)	Kerr	Microhybrid	79	0,6 $\mu\text{m}$

The authors applied light-curing procedures by means of two type of lamps: LED lamp L.E. Demetron 1 (SDS/Kerr) and halogen lamp (Ivoclar Vivadent). Technical specification of the lamps is given in table 2.

Table 2. Specification of the lamps

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

The applied exposure times were 40 and 60 seconds. Forty seconds is recommended by manufacturer of the materials. The authors introduced 60 seconds, based on the literature reports [5,13,21], which suggested that the extension of the exposure time improved polymerization effectiveness as a result of increase of the degree of conversion. The degree of conversion indicates a percentage of double bonding in polymer structure that underwent saturation (converted to single bond) [26]. Depending on the photopolymerization process conditions conversion degree is in a range between 65% and 80% [22,30].

### 2.1. Flexural strength studies

For flexural strength studies a three-point bending strength test was applied (TFS). Test samples (N = 20) were prepared in a shape of rectangular prism with dimensions of 2mm x 2mm x 25mm, according to PN-EN ISO 4049 standard. Tests were carried out at crosshead speed of 0.5 mm/min, with 20 mm distance between the supports. Supports radius and crosshead radius were 1 mm (Fig. 2).

Strength ( $\sigma$ ) was calculated based on the following formula:

$$\sigma = \frac{3PL}{2bd^2} [\text{MPa}] \quad (1)$$

where:

$P$  – loading during the test [N]

$L$  – distance between the supports [mm]

$b$  – specimen width [mm]

$d$  – specimen thickness [mm]

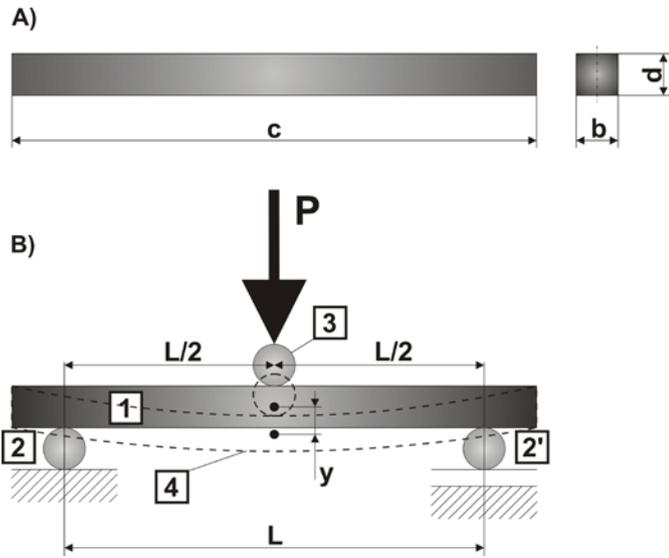


Fig.2. Scheme of the specimen (A) and test stand for the strength studies in 3-point bending test conditions - TFS (B): 1 – specimen; 2 – constant support; 2' – sliding support; 3 – loading crosshead; 4 – deflected beam; L – distance between supports; c, b, d - specimen dimensions; y – beam deflection

2.2. Statistical analysis

Strength test results were divided into groups based on: composite type (FSi, GDA, GDP, H), type of lamp (HAL, LED) and exposure time (40 sec, 60 sec). In order to verify a significance of differences between sets of results a variation analysis of one variable was performed (ANOVA). F – Snedecor test was applied. Next, to evaluate a significance of direct differences between the sets assigned to particular photopolymerization procedures HSD Tukey's test was used.

For reliability analysis of the studies composites a two-parameter Weibull distribution was applied. Generally, a cumulative distribution function ( $P_f$ ) of Weibull distribution (with positive parameters  $\sigma_0$ ,  $m$ , and  $\sigma_u$ ) is described by [16]:

$$P_f = 1 - \exp \left[ -N \left( \frac{\sigma - \sigma_u}{\sigma_0} \right)^m \right] \quad (2)$$

where:

- $\sigma$  – failure load,
- $\sigma_0$  – scale parameter,
- $m$  – shape parameter,
- $\sigma_u$  – location parameter,
- $e$  – constant ( $e = 2.71828\dots$ ),
- $N$  – sample size,
- $P_f$  – probability of failure.

In case when the sample size  $N$  is constant in all studied sets (here specified by exposure time and type of lamp),  $N$  can be neglected in the calculations [2, 32].

If assuming location parameter value equal to zero  $\sigma_u = 0$ , Weibull distribution becomes two-dimensional. With these assumptions based on the equation (2) a formula for survival probability can be formulated,  $P_s$ :

$$P_s = 1 - P_f = 1 - \left( 1 - \exp \left[ - \left( \frac{\sigma}{\sigma_0} \right)^m \right] \right) \quad (3)$$

The above equation after finding a logarithm can be reduced to the linear form

$$y = ax + b \quad (4)$$

thus:

$$\ln \left[ \ln \left( \frac{1}{P_s} \right) \right] = m \ln(\sigma) - m \ln(\sigma_0) \quad (5)$$

An intersection point of Y axis and approximating line depends on  $-m \ln(\sigma_0)$ , while a slope parameter of approximating curve is a shape parameter of Weibull distribution  $m$ .

3. Test results

Descriptive statistics of flexural strength test results are given in table 3: sample size ( $N$ ), average value, minimum and maximum value, standard deviation values, variation coefficient and Weibull modulus.

The highest average flexural strength value was obtained for FSi material after using HAL lamp and 60 seconds exposure time (119.5 MPa). Filtek Siloran (FSi) showed the highest strength in all photopolymerization procedures (HAL 40, HAL 60, LED 40, LED 60).

Table 3. Descriptive statistic of flexural strength test results

Material	LCU	Time [s]	Descriptive statistic of RBC bending strength [MPa]					Co.Var. [%]
			N	Mean	Min	Max	St.Dev.	
FSi	HAL	40	20	108,15	85,10	126,00	9,00	8,32
		60	20	119,50	100,00	142,00	9,92	8,30
	LED	40	20	108,26	90,90	134,00	11,95	11,04
		60	20	107,76	82,60	143,00	13,15	12,20
GDA	HAL	40	20	77,77	63,90	86,60	5,52	7,10
		60	20	74,98	58,10	89,00	8,68	11,58
	LED	40	20	68,16	57,00	81,80	6,86	10,07
		60	20	67,48	41,80	83,30	9,35	13,86
GDP	HAL	40	20	84,15	76,60	91,00	3,74	4,45
		60	20	82,03	67,00	94,00	6,12	7,46
	LED	40	20	79,66	62,60	92,60	7,88	9,90
		60	20	76,08	56,10	95,70	11,97	15,73
H	HAL	40	20	107,23	69,90	125,00	13,26	12,37
		60	20	99,60	49,40	130,00	16,61	16,68
	LED	40	20	102,61	76,50	122,00	10,16	9,90
		60	20	102,05	79,90	124,00	12,09	11,84

The lowest strength in all groups was demonstrated for GDA material (67.48 MPa in LED 60 set).

The results of variance analysis obtained from F – Snedecor test (tab. 4) allowed rejecting zero hypothesis on the lack of differences in the effect of different photopolymerization procedures with regards to three materials: FSi, GDA and GDP. The largest differences have been observed in group of GDA results ( $F = 8.58$ ). There have been no significant differences in comparisons of H material strength groups.

In direct comparisons of result groups in HSD Tukey's test (Tab. 5) there were no significant differences in strength of FSi material observed in three cases:

- between HAL 40 and HAL 60 groups,
- between HAL 60 and LED 40,
- between HAL 60 and LED 60.

Table 4. Variance analysis results (F – Snedecor test)

Analysis of Variance (p < 0,05)								
Material	SS – Effect	df - Effect	MS – Effect	SS - Error	df - Error	MS – Error	F	p
FSi	1968,09	3	656,03	9408,01	76	123,79	5,30	0,002266
GDA	1546,80	3	515,60	4567,62	76	60,10	8,58	0,000056
GDP	718,22	3	239,40	4880,74	76	64,22	3,73	0,014753
H	608,14	3	202,71	13321,91	76	175,29	1,16	0,332002

Table 5. Post-hoc HSD Tukey's test results (p &lt; 0,05)

FSi				
	{1} - M=108,14	{2} - M=119,50	{3} - M=108,25	{4} - M=107,76
HAL 40s {1}		0,009864	0,99999	0,999588
HAL 60s {2}	0,009864		0,0108	0,007141
LED 40s {3}	0,99999	0,0108		0,999066
LED 60s {4}	0,999588	0,007141	0,999066	
GDA				
	{1} - M=77,765	{2} - M=74,980	{3} - M=68,155	{4} - M=67,475
HAL 40s {1}		0,66862	0,001193	0,000538
HAL 60s {2}	0,66862		0,033632	0,015878
LED 40s {3}	0,001193	0,033632		0,992558
LED 60s {4}	0,000538	0,015878	0,992558	
GDP				
	{1} - M=84,145	{2} - M=82,030	{3} - M=79,660	{4} - M=76,075
HAL 40s {1}		0,837899	0,295872	0,011169
HAL 60s {2}	0,837899		0,786106	0,096001
LED 40s {3}	0,295872	0,786106		0,494301
LED 60s {4}	0,011169	0,096001	0,494301	
H				
	{1} - M=107,22	{2} - M=99,600	{3} - M=102,61	{4} - M=102,04
HAL 40s {1}		0,271535	0,68929	0,60542
HAL 60s {2}	0,271535		0,88935	0,936703
LED 40s {3}	0,68929	0,88935		0,999175
LED 60s {4}	0,60542	0,936703	0,999175	

Additionally, significant differences have been demonstrated in strength of GDA material while comparing the following results groups: HAL 40 and LED 40, HAL 40 and LED 60, HAL 60 and LED 40, as well as HAL 60 and LED 60. In case of GDP material significant differences were shown by comparing groups HAL 40 and LED 60, as well as HAL 60 and LED 60. In case of H material there were no significant differences in strength (resulting from photopolymerization procedure).

In table 6 the results of approximation of experimental data by means of Weibull distribution are given: coefficient of determination  $R^2$ , characteristic strength (scale parameter)  $\sigma_0$  and Weibull modulus (shape parameter)  $m$ . Average  $m$  parameter values and standard deviation are also shown in sets assigned to particular materials: FSi, GDA, GDP, H.

The highest Weibull modulus was obtained for result group GDP HAL 40 ( $m = 27,28$ ). The highest average value of modulus was indi-

Table 6. Coefficient of determination  $R^2$ , characteristic strength  $\sigma_0$  (MPa), Weibull modulus  $m$  of FSi, GDA, GDP, H composites in 3-point bending test

Material	LCU	Time [s]	$\sigma_0$	$R^2$	Weibull mod. $m$	Weibull mod. $m$	
						Mean	St.Dev.
FSi	HAL	40	112	0,97	14,25	12,31	2,38
		60	122	0,95	14,46		
	LED	40	112	0,93	10,65		
		60	112	0,93	9,88		
GDA	HAL	40	81	0,98	16,82	11,63	3,90
		60	81	0,97	10,24		
	LED	40	72	0,93	11,90		
		60	72	0,90	7,54		
GDP	HAL	40	86	0,96	27,28	15,69	8,45
		60	85	0,97	16,00		
	LED	40	84	0,97	11,88		
		60	79	0,94	7,60		
H	HAL	40	116	0,95	8,91	9,00	2,42
		60	106	0,86	5,88		
	LED	40	105	0,97	11,76		
		60	106	0,93	9,44		

cated for GDP composite ( $m = 15,69$ ), however in this case also the largest standard deviation was observed ( $st.dev. = 8,45$ ). It confirms a considerable diversification of the results between the photopolymerization subgroups. For FSi material the lowest deviation of the average  $m$  modulus value ( $st.dev. = 2,38$ ) was noticed. The lowest average value of Weibull modulus was obtained for H material ( $m = 9,00$ ;  $st.dev. = 2,42$ ), also for this material a minimum modulus value of the whole studied population ( $m = 5,88$ ) was demonstrated.

A linear approximation after logarithmic transformation of the flexural strength test results, regression equations and coefficients of determination are shown in Figures 3 – 6.

#### 4. Discussion

A varied effect of photopolymerization procedures depending on the type of composite on the flexural strength of the studied materials and their reliability has been shown. In a group of methacrylate based composites the average strength in the tested specimen (tab. 3) decreases in the statistically significant manner after switching from halogen (HAL) to LED lamp in case of GDA and GDP materials, whereas in case of H material changes are insignificant (tab. 4). It has been indicated that H is characterized by rather large scatter of results of each observation (variation coefficient 9.90% – 16.68%). A similar tendency of strength reduction has been also noticed while increasing exposure time from 40 to 60 seconds.

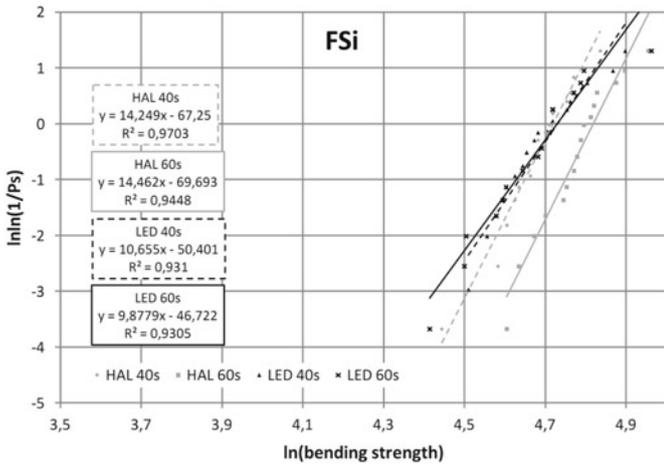


Fig. 3. Aproximation of probability distribution of flexural strength of FSi composite

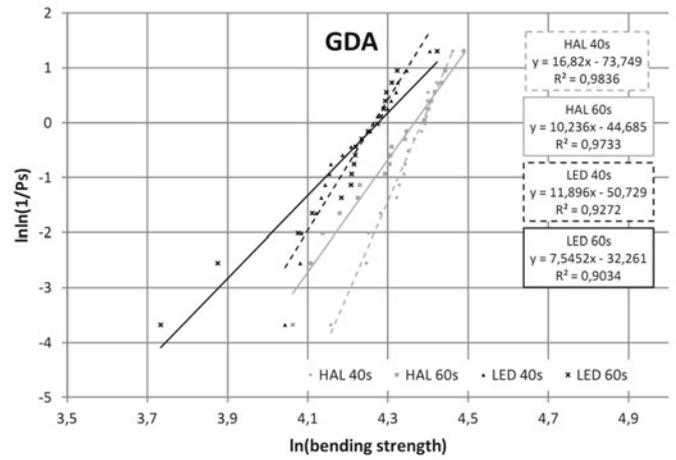


Fig. 4. Aproximation of probability distribution of flexural strength of GDA composite

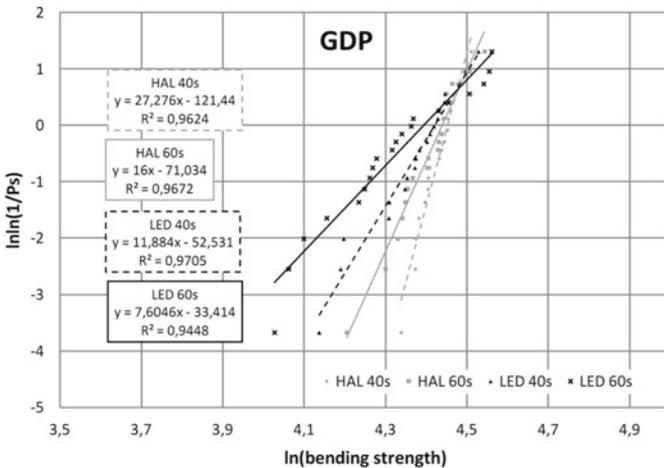


Fig. 5. Aproximation of probability distribution of flexural strength of GDP composite

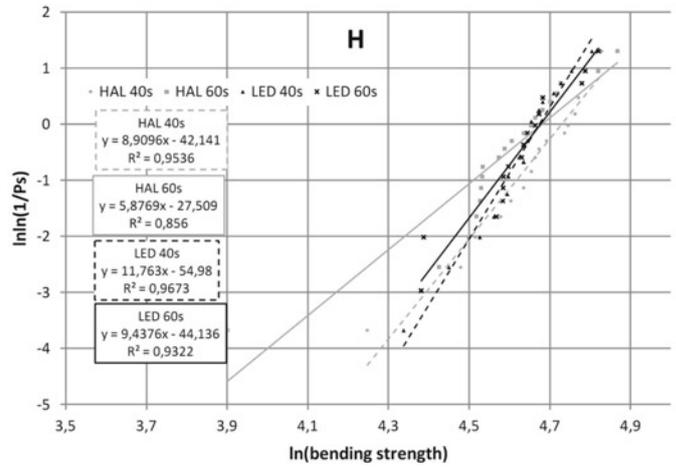


Fig. 6. Aproximation of probability distribution of flexural strength of H composite

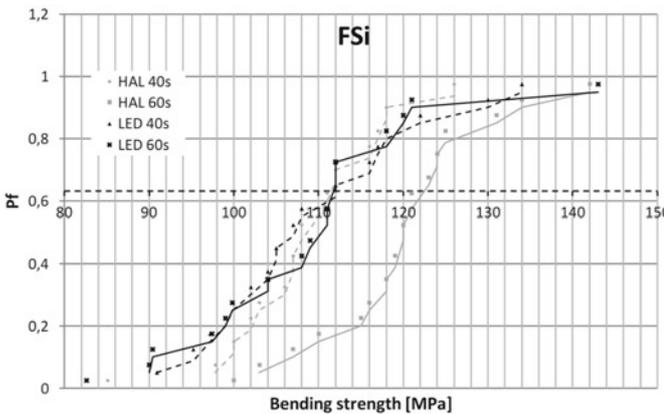


Fig. 7. Unreliability function of FSi composite with regards to flexural strength at different photopolymerization procedures

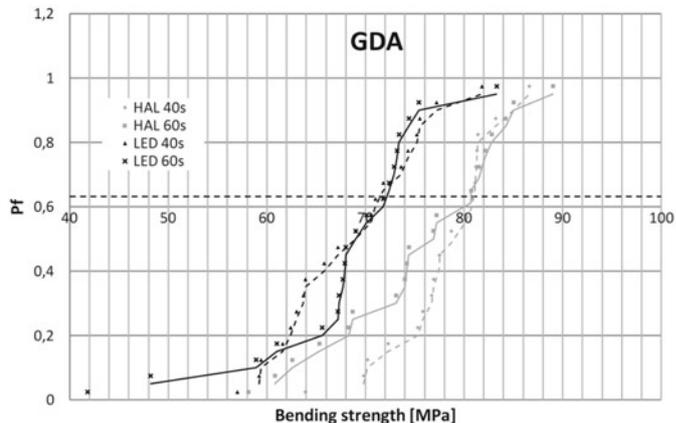


Fig. 8. Unreliability function of GDA composite with regards to flexural strength at different photopolymerization procedures

For the studied group of conventional polymers based on methacrylates, regularities described above are also valid in the aspect of reliability (tab. 6, fig. 8, fig. 9, fig. 10), although it is not so unequivocal. Switching from halogen to LED lamp has a clear and negative impact on the characteristic strength only in case of GDA material. For GDP material this effect is insignificant. In both cases the impact of pro-

longed exposure time has not been noticed. In case of H material, both changing the lamp as well as extending the exposure time, reduced the characteristic flexural strength. It is worth noticing that the Weibull modulus  $m$  for H material has a low value. In the literature the low Weibull modulus is interpreted as a result of large scatter of structural defects in the specimens, equal to its low reliability [20, 23].

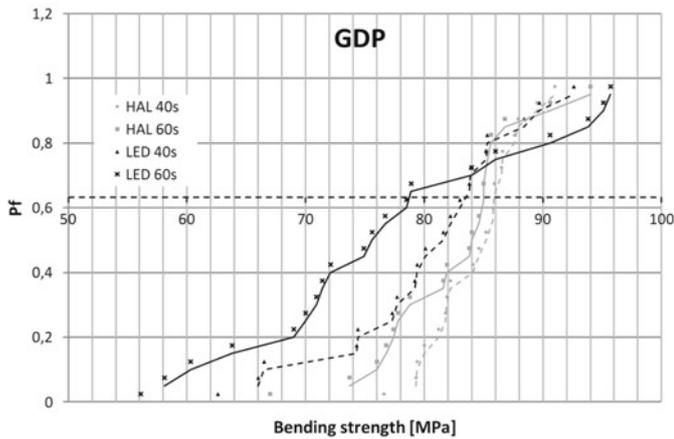


Fig. 9. Unreliability function of GDP composite with regards to flexural strength at different photopolymerization procedures

In comparison with conventional methacrylates-based composites, FSi material based on siloranes can be favorably distinguished. A high characteristic strength value (the highest of all studied materials)  $\sigma_0 = 112$  to  $122$  MPa (tab. 6) and high Weibull modulus value  $m = 12.31$  were observed, which gives a basis for a quite high rating of reliability of this material. Unreliability function curve (fig.7) indicates a rapid increase of failure probability due to flexural strength occurs above  $100$  MPa, while for conventional composites it occurs already at  $60$ – $90$  MPa. It can result from difference in polymerization shrinkage, which as well know, is a cause of initiation of internal residual stresses in the material. The polymerization shrinkage of Filtek Siloran material is estimated at  $\leq 1.0\%$  [12], while polymerization shrinkage of methacrylates-based composites is in a range of  $2.0$  –  $3.0\%$  [29,33]. Additionally, it has been indicated that in case of FSi composite the extension of exposure time by halogen lamp from  $40$  to  $60$  seconds results in the increase of characteristic strength, while it has no impact on reliability (Weibull modulus). It has been demonstrated that switching to LED lamp has now impact on the strength of FSi composite.

The conducted studies have indicated that for some testing groups (GDA material – group LED 40 and LED 60, GDP material – group LED 60, H material – group HAL 60 and LED 60) there are low values of coefficient of determination (below  $0.95$ ). Some authors suggest that it can be caused by heterogeneity of the material properties in specimen as a result of imperfect photopolymerization procedure, consisting in overlapping exposure of the specimens surface by the light beam of the lamp [19, 28], thus producing double exposed areas in the specimens.

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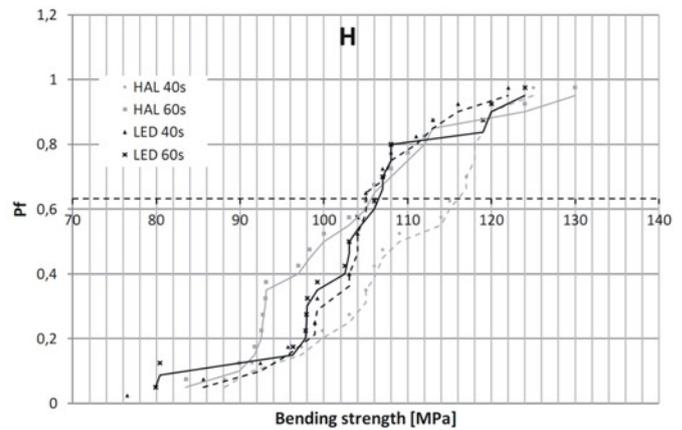


Fig. 10. Unreliability function of H composite with regards to flexural strength at different photopolymerization procedures

## 5. Conclusions

1. It has been demonstrated that in 3-point bending test conditions a change of the type of photopolymerization lamp from halogen (HAL) to LED brings the following results:
  - in case of FSi composite with low polymerization shrinkage it has no impact on the characteristic composite strength, while it has an impact on the increase of its reliability (specified by Weibull modulus);
  - in case of conventional composites based on methacrylate compounds (GDA, GDP, H) a reduction of characteristic strength occurs, while reliability of the material increases (increase of Weibull modulus).
2. It has been concluded that the extension of polymerization time from  $40$  to  $60$  seconds:
  - in case of Filtek Siloran (FSi) with use of halogen lamp effects in the increase of strength, however it does not change composite reliability. In case of LED lamp it has no impact on the strength and only insignificantly increases reliability;
  - in case of conventional composites based on methacrylates (GDA, GDP, H) with the use of both halogen and LED lamp has no impact on the strength, however it significantly improves material reliability.
3. It has been concluded that the applied method for analysis of the study's results, consisting in application of Weibull distribution relating to 3-point bending test, expands possibilities of preliminary assessment of operational usability of the new dental materials.

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**Agata M. NIEWCZAS, DMD Ph.D.**

Department of Conservative Dentistry  
Medical University of Lublin,  
Karmelicka 7 Str., 20-081 Lublin,  
e-mail: agatan117@wp.pl

**Daniel PIENIAK, Ph.D. (Eng.)**

Department of Applied Mechanics,  
Main School of Fire Service, Warsaw  
52/54 J. Słowackiego Str., 01-629 Warsaw  
e-mail: dpieniak@sgsp.edu.pl

**Paweł OGRODNIK, Ph.D. (Eng.)**

Department of Applied Mechanics,  
Main School of Fire Service, Warsaw  
52/54 J. Słowackiego Str., 01-629 Warsaw  
e-mail: pogrodnik@sgsp.edu.pl

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