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Problems associated with photopolymerization in dentistry

SUMMARY

Of Ph.D. Thesis

for awarding the educational and scientific degree

„DOCTOR“

SCIENTIFIC SPECIALTY

Therapeutic dentistry

SCIENTIFIC ADVISORS:

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2. Prof. Dr. Vladimir Panov, D.Sc.

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The dissertation contains 197 standard pages and is illustrated with 24 tables and 45 figures. The bibliography consists of 178 sources, of which 4 in Cyrillic and 174 in Latin.

The public defense of the Ph.D. Thesis will take place on June 8, 2021 in the Auditorium "Assoc. Dimitar Klisarov " at the Faculty of Dental Medicine of the Medical University of Varna "Prof. Dr. Paraskev Stoyanov", in front of a a scientific council consisting of:

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Prof. Dr. Radosveta Vasileva, MD, PhD – external member

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Ass. Prof. Dr. Miglena Balcheva, MD, PhD – reserve internal member

The materials on the defense are available in the Scientific Department of MU - Varna and are published on the website of MU - Varna.

Note: In the abstract the numbers of the formulas correspond to the numbers in the Ph.D. Thesis.

CONTENTS

LIST of ABBREVIATONS

BFC - bulk fill light cured composite for posterior restorations
FC - universal flowable highly filled light cured composite
UC - universal nanohybrid light cured composite
RBC - resin based composites
LCU - light curing unit
HV - Vickers hardness
LED - light-emitting diode
d - layer thickness, mm
E_L - energy required for the adequate polymerization of a 2 mm composite layer, J/cm^2
I - light intensity, mW/cm^2
P - power, W
S - surface area, cm^2
τ, t - irradiation time, s
x_1 - light intensity
x_2 - irradiation time
x_3 - layer thickness
Y_1 - top surface hardness
Y_2 - bottom surface hardness

INTRODUCTION

The introduction of light cured resin based composites (RBCs) is a revolutionary step in restorative dentistry because it allows clinicians to determine the beginning of the polymerization process. The reason for their wide application in everyday practice are on the one hand the increased aesthetic requirements of patients, and on the other hand the disadvantages of the amalgam such as bad aesthetics, galvanic current flow and corrosion, staining of hard dental tissues, soft tissue tattoos, release of mercury vapor and others. During the last decade there have been a lot of hard work on the improvement of light cured RBCs. Evidence of this is the improved properties of conventional composites, the creation of universal flowable composites with a high filler content and bulk fill composites, which allow up to 5 mm layer thickness.

In order to carry out the process of photopolymerization of dental composites, a source of blue visible light with a wavelength in the range of 400-520 nm is required. Modern LED light curing units (LCUs) have very good characteristics and have a number of advantages over other types of LCUs such as: the ability to polymerize composites using all types of photoinitiator systems; high light intensity; availability of compact wireless models with an exceptional battery life; affordable price; small heat generation. Due to the many advantages and virtually no disadvantages, LED LCUs have established themselves as the most reliable and preferred polymerization devices by dentists around the world.

Despite the daily placement of composite restorations and the presence of LCUs in any dental practice, the level of knowledge of dentists about the main factors of the photopolymerization process - light intensity, irradiation time, layer thickness, distance and direction of the LCU's tip, etc., is not high.The poor awareness of the work with LCUs and light polymerization factors can lead to an incorrect curing protocol, which in turn can lead to an incomplete polymerization of the material with all the adverse consequences: increased risk of restoration fracture, reduced hardness and wear resistance, elution of residual monomers, reduced adhesive bonding strength and faster color change.

This gives grounds for the present dissertation to be focused on this part of the problems of photopolymerization - by conducting research using conventional and newly developed methods to contribute to a higher level of knowledge about working with LCUs and to give practical guidelines for better control of the polymerization factors. This, in the end, will ensure higher quality and longevity of the composite restorations, which in turn will improve the dental health of patients.

AIM OF THE DISSERTATION:

To study the problems related to light curing in dentistry by analyzing the factors influencing the process of photopolymerization of dental composites.

TASKS:

1. To investigate the relationship between the light intensity of wireless LED LCUs and their battery charge.
2. To investigate the light intensity of LED LCUs after a different period of use and to establish the relationship between the time of use of the devices and their light intensity.
3. To study the influence of the factors of the photopolymerization process - light intensity, irradiation time and layer thickness on the hardness of dental composites from three different groups:
    a. Universal nanohybrid light cured composite;
    b. Bulk Fill light cured composite;
    c. Universal flowable light cured composite with a high filler content.
4. To optimize the parameters of the process of photopolymerization of the studied dental composites.
    a. To develop recommended light curing modes of the studied dental composites;
    b. To make recommendations for efficient polymerization for each of the studied composites.

MATERIALS and METHODS

MATERIALS and METHODS
for TASK 1
Investigation of the relationship between the light intensity of wireless LED LCUs and their battery charge

The aim of the present task is to evaluate the stability of the light intensity of ten different brands of wireless LED LCUs by measuring it from a fully charged to a fully discharged battery. A comparative analysis of the actual light intensity of the LCUs with that, specified by the manufacturers, was made. Ten new different fully-charged wireless LED LCUs were used (Table 1). Light intensity was measured with a digital radiometer LM1 (*Woodpecker, China*) (*fig. 1*). The light guide tip was placed in contact with the radiometer sensor at an angle of 90^{o}. Orange glasses ("*blue blockers*") were used to protect the operator from eye damage, caused by the blue visible light. (*fig. 2*). To reproduce the clinical situation measurements were made every 10 curing cycles of 20 s each in continuous mode of operation until the LCU's battery was completely discharged.

Fig. 1. Digital Radiometer LM-1, Woodpecker, China

Fig. 2. Protective orange glasses "blue blockers".

Table 1.
Technical data of LED LCUs.

№	**Model LED LCU**	**Brand**	**Light intensity, mW/cm^2**	**Wavelength, nm**
1	*Xlite 4*	ThreeH, China	800	385-515
2	*Bluephase N*	Ivoclar Vivadent, Lichtenstein	1200	385-515
3	*D-Light Duo*	GC, Japan	1200	400-480
4	*LY-C240*	BDMED, China	1200	420-480
5	*OSA-F686C*	Osaka Dental, China	1200	440-480
6	*Demi Plus*	Kerr, USA	1200	450-470
7	*I-LED 2500*	Woodpecker, China	1300	420-480
8	*Elipar Deep Cure S*	3M ESPE, USA	1470	430-480
9	*CV-215*	Cicada Dental, China	1500	430-480
10	*SK-L029A*	Spark Dental, China	2200	385-430

In Table 1, the light intensity values by specification refer to continuous mode of operation of the LCUs, not taking into account the turbo-modes (3 s) with higher intensity in some models. D-Light Duo have only one polymerization mode of 10 s, so two cycles of 10 s are counted as one cycle of 20 s.

For each LCU, the number of curing cycles of 20 s until the battery is completely discharged is determined. With the use of Microsoft Excel software, the change of the light intensity is expressed by increasing the number of polymerization cycles (N) and by decreasing the battery life (%).

MATERIALS and METHODS for TASK 2

Investigation of the light intensity of LED light curing units after different periods of use to establish the relationship between the time of use of the devices and their light intensity.

The aim of the present paper is to study the change in light intensity of light curing units after different periods of intensive use. Ninety four regularly used and fully charged LED LCUs, aged between 1 and 10 years were examined, 68 of which are mounted on the dental unit and 26 are wireless (*table 2*). The devices are located on the territory of the Faculty of Dental Medicine at Medical University of Varna (Bulgaria), as well as in various dental offices in Varna.

Table 2.
Technical data of LED LCUs.

Group	**Number of LCUs**	**Model**	**Brand**	**Light intensity, mW/cm^2**	**Wavelength, nm**
1	19	*DB-686 DELI*	Coxo Medical Instruments, China	>1600	420-480
1	1	*SK-L029A*	Spark Dental, China	2200	435-480
1	3	*Smart Xpress*	Bluedent, Bulgaria	1700	410-490
2	21	*Masterdent*	Vigodent, Bulgaria	>700	400-480
2	9	*Minident*	Vigodent, Bulgaria	>700	420-480
3	40	*LD Max*	Gnatus, Brazil	700	440-480

The light intensity was measured with a digital radiometer (LM-1, Woodpecker, China), which allows measuring LCUs' output from 0 to 3500 mW/cm^2 (*fig. 1*). Before the start of the measurements, LCUs' tips were cleaned of contamination and stuck hard pieces of composite. LCUs were used in conventional mode with maximum intensity of the light. The light guide tip was placed in contact with the radiometer sensor at an angle of 90^o, three measurements were made for each device and the mean values were recorded. Orange glasses ("*blue blockers*") were used to protect the operator from eye damage, caused by the blue visible light.

The LCUs are divided into three groups depending on their time of use. The first group includes 23 devices from three models of different brands, measurements of which were made after 1, 3 and 5 years of use. The second group is composed of 31 devices from two models of the same brand, measurements of which were made after 7 and 9 years of use. In the third group 40 devices from one model of the same brand are included, measurements of which were made after 10 years of use.

The results are analyzed statistically and presented graphically using *Microsoft Excel* software.

MATERIALS and METHODS for TASK 3

Influence of the factors of the photopolymerization process on the hardness of dental composites

3.1. Materials

To perform the experiment, 3 dental composites from different groups were used:

1) Universal nanohybrid light cured composite (UC) - *Evetric* (Ivoclar Vivadent, Lichtenstein),

2) Nanohybrid bulk fill light cured composite (BC) for posterior restorations which allows layer thickness up to 5 mm - *Filtek One Bulk Fill Restorative* (3M, USA) and

3) Universal nanofilled flowable light cured composite (FC) indicated for restoring all cavity classes - *G-aenial Universal Flo* (GC, Japan).

All composites are of the same shade - A2, but have a different composition and ratio between the organic matrix and filler (*table 3*).

Table 3.
Composition of the composites used.

Composite	Composition: Component	Composition: Amount	Shrinkage	Density
UC ***Evetric*** [1*, 2*] Matrix/filler ratio wt %: 19-20/80-81	Matrix: BIS-GMA (Bisphenol A glycydil dimethacrylate) UDMA (Urethane dimethacrylate) Bis-EMA (Bisphenol A polyethethylene glycol dimethacrylate) Fillers: Barium glass, Ytterbium Fluoride (YbF3) Mixed oxydes and prepolymers 40nm-3μm	3-10%; 10-25%; 3-10%	1.5%	2.10 g/cm^3
BC ***Filtek One Bulk Fill Restorative*** [3*, 4*] Matrix/filler ratio wt %: 23.5/76.5	Matrix: AUDMA (Aromatic Urethane Dimethacrylate) DDDMA (1,12-Dodecane Dimethycrylate) UDMA (Urethane dimethacrylate) Fillers: Silane Treated Ceramic, Silane Treated Silica, Ytterbium Fluoride (YbF3), Silane Treated Zirconia	10-20% <10% 1-10%	1.8%	1.9 g/cm^3

ТФК ***G-aenial Universal Flo*** [5*, 6*] Matrix/filler ratio wt %: 31/69	Matrix: UDMA (Urethane dimethacrylate) Bis-EMA (Bisphenol A polyethethylene glycol dimethacrylate) Dimethacrylate component Fillers: Silicon dioxide (16 nm), Strontium glass (200 nm), pigments	10-20% 5-10% 5-10%	3.95 %	-
Note: 1*Objelean AC et al, 2015. 2*Safety data sheet. Evetric. 3*Safety data sheet. Filtek One Bulk Fill Restorative. 4* Technical Product Profile. Filtek One Bulk Fill Restorative. 5* Safety data sheet. G-aenial Universal Flo. 6* Technical Manual. G-aenial Universal Flo.				

Table 4.
Mechanical properties of the composites used.

Composite	Tensile strength, MPa	Compressive strength, MPa	Flexural stength MPa	Modulus of elasticity , GPa	Hardness on top surface, HV
UC ***Evetric***	40 [1*]	263 [1*]	94.5 [1*]	12.2 [7*]	62 [1*]
BC ***Filtek One Bulk Fill Restorative***	55.74 [8*]	347 [9*]	183 [9*]	10.6 [9*]	55.2 (1500 mW/cm^2/20 s) 52.0 (1000 mW/cm^2/20 s) 53.2 (600 mW/cm^2/40 s) [9*]
FC ***G-aenial Universal Flo***	-	425 [10*]	167 [11*]	7.9 [11*]	49.01 (1200 mW/cm^2/20 s) [12*] 48.54 (700 mW/cm^2/40 s) [13*]

Note: 1*Objelean AC et al, 2015. 7*Azmi MM et al, 2017. 8*Abdulmajeed A et al, 2019. 9*Cowen M et al, Dental Advisor. 10*Estelite Universal Flow Brochure. 11*G-aenial Universal Flo. Research and physical properties. 12*Al Sunbul A et al, 2016. 13*Jang JH et al, 2015.

3.2. Preparation of the samples

Composite samples with a cylindrical shape, diameter of 5 mm and thickness of 2, 3 and 4 mm were made for each composite (*fig. 3*). Polyurethane matrices, glass slides and transparent celluloid strips were used for the preparation of the samples. The material was placed in a single layer. The samples were polymerized with LED LCU *Curing Pen* (Eighteeth, China) with a wavelength of 385-515 nm (*fig. 4*) for 20, 40 or 60 s with a light intensity of 600, 1000 or 1500 mW/cm^2 in conventional curing mode. The distance between the LCU's tip and the top surface of the composite was 1 mm, which is the thickness of the glass slide.

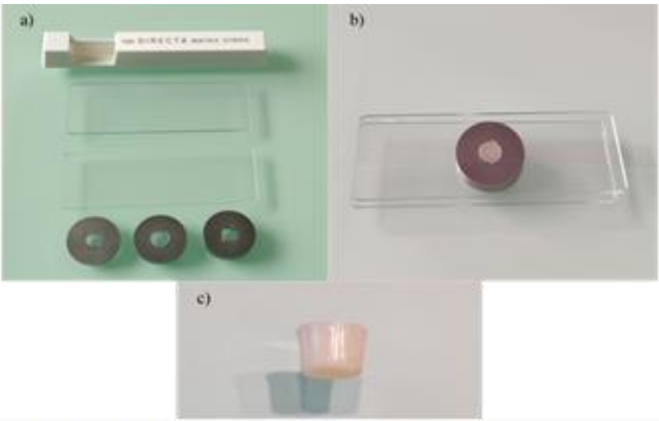


Fig. 3. Polyurethane matrices, glass slides and cellulose strips - a), filling the matrix with composite and pressing with a slide - b) and polymerized and removed sample, ready for storage for 24 hours in a dry dark container - c).

Fig. 4. LED Light curing unit Curing Pen (Eighteeth).

For each combination of parameters (light intensity, curing time and layer thickness) 3 samples were made (*table 5*) and stored in dry dark containers at room temperature for 24 hours, after which the Vickers microhardness measurements were performed.

Table 5.
Experiment plan.

Light Curing Unit „*Eighteeth CuringPen*”										
Light intensity, mW/cm²	**600**			**1000**			**1500**			**Total number of samples**
Time, s.	**20**	**40**	**60**	**20**	**40**	**60**	**20**	**40**	**60**	
Composite UC *Evetric*	**Number of samples**									
Thickness, 2 mm	3	3	3	3	3	3	3	3	3	27
3 mm	3								3	6
4 mm	3		3			3	3	3	3	18
BC *Filtek One Bulk Fill Restorative*										

Thickness, 2 mm	3	3	3	3			3		3	18
3 mm	3								3	6
4 mm	3		3			3	3	3	3	18
FC *G-aenial Universal Flo*										
Thickness, 2 mm	3	3	3	3			3		3	18
3 mm	3								3	6
4 mm	3		3			3	3	3	3	18
	Total number of samples									**135**

3.3. Microhardness measurements

Vickers microhardness was measured with a ZHVμ-S microhardness tester (Zwick / Roell, Germany). With a load of 50 gr for 10 s, 5 measurements were made (*Fig. 5 and Fig. 6*) on the top and bottom surface of each sample and the mean values were recorded.

On 9 samples (600 mW/cm^2 /2mm /20 s, 1000 mW/cm^2 /2mm /40 s and 1000 mW/cm^2 /2mm /60 s) 4 additional measurements were made within 28 days - on the 7th , 14th, 21st and 28th day. Throughout the study period, the samples were stored in dry dark containers at room temperature.

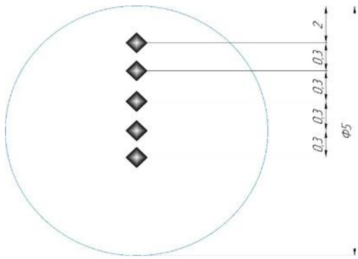


Fig. 5. Test scheme.

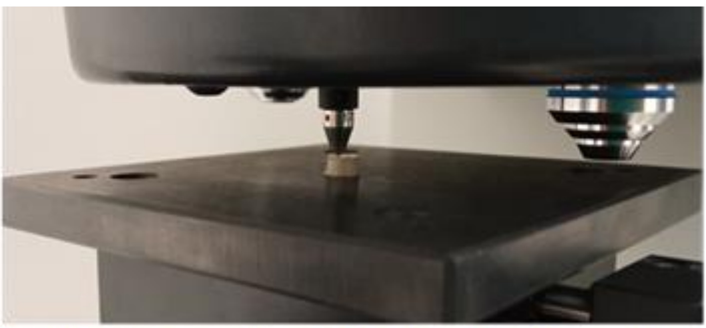


Fig. 6. Application of a base load F 50 gr with a duration of 10 s on a sample of UC Evetric.

The results are expressed in tabular and graphical form using *Microsoft Excel* software, and the influence of the factors is examined by analysis of variance (ANOVA).

The hardness measurements were performed with the assistance of Eng. Vladimir Todorov, Ph.D. from Technical University of Gabrovo.

MATERIALS and METHODS for TASK 4

Optimization of the parameters of the process of photopolymerization of dental composites

4.1. Regression analysis

An optimization of the three parameters of the photo-polymerization process – light intensity *I*, irradiation time *t* and layer thickness *d* was caried out. The aim was to obtain a composite layer with a certain thickness, having maximum micro-hardness on the top surface (HVmax) and micro-hardness on the bottom surface - 80% of Hvmax [Price RB et al, 2003; Yap AU et al, 2003; Bouschlicher MR et al, 2004], in given intensity values and irradiation times, specific for each LCU.

Table 6

Governing factors and their levels

Governing factors					
Natural $\tilde{x}_i$	**Coded** x_i	**Factor levels**			
		Coded			
		For the first factor			
		-1	-0.1111		1
		For the rest of the factors			
		-1		0	1
		Natural			

Intensity I [mW/cm^2] $\tilde{x}_1$	x_1	600	1000		1500
Time t [s] $\tilde{x}_2$	x_2	20		40	60
Thickness d [mm] $\tilde{x}_3$	x_3	2		3	4

The governing factors, namely intensity *I*, irradiation time *t* and layer thickness *d* as well as their levels, are listed in *table 6*. The factors, measured in natural physical units, are marked with $\tilde{x}_i$ and have different dimensions. In order to eliminate the experimental plan's dependence from the dimensions, the factors $\tilde{x}_i$ are transformed into a coded form x_i through dependence:

$$x_i = \left(\tilde{x}_i - \tilde{x}_{i,0}\right) / \left|\tilde{x}_{i,max} - \tilde{x}_{i,0}\right| \qquad (3)$$

The objective functions are: Y_1, HV – hardness on the top surface of the composite layer and Y_2, HV – hardness on the bottom surface. A planned experiment for the three investigated composites is carried out. The experimental design is shown in *table 7*.

Regression analyses of the obtained experimental results for each composite were carried out through *QStatLab* software.

For the objective functions Y_i, $i=1,2$, polynomials from second order were chosen since the governing factors were changed of three levels:

$$Y_k\left(\{X\}\right) = a_0 + \sum_{i=1}^{m} a_i x_i + \sum_{i=1}^{m-1} \sum_{j=i-1}^{m} a_{ij} x_i x_j + \sum_{i=1}^{m} a_{ii} x_i^2, k = 1,2 \qquad (4)$$

к where $\{X\} = [x_1\ x_2]^T \in \Gamma_x$ is the vector of the governing factors, Γ_x is the admissible space of the governing factors and m is their number.

For each of the composites, regression models were created for the objective functions Y_1 - hardness on the top surface of the composite layer and Y_2 - hardness on the bottom surface.

Using regression models, for UC *Evetric* optimizations were made in nine variations of the governing factors intensity (x_1) and irradiation time (x_2). As a condition for optimization, for each composite the average value of the micro-hardness on the top surface obtained in the experiment was accepted, and for the micro-hardness on the bottom surface - 80% of the micro-hardness on the top surface.

Table 7
Experiment plan.

№	Composite type						UC		BC		FC	
	Governing factors						Y_1	Y_2	Y_1	Y_2	Y_1	Y_2
	Coded			Natural								
	x_1	x_2	x_3	I, mW/cm^2	t, s	d, mm	HV top	HV bottom	HV top	HV bottom	HV top	HV bottom
1	-1	-1	-1	600	20	2	42.0	33.5	59.1	55.8	42,4	37,9
2	1	-1	-1	1500	20	2	52.4	42.9	61.7	60.2	50,0	46,3
3	-1	1	-1	600	60	2	45.9	41.1	61.8	61.1	47,5	45,5
4	1	1	-1	1500	60	2	57.8	51.3	68.4	67.5	49,9	47,7
5	-1	-1	1	600	20	4	45.0	12.2	57.9	45.4	42,9	13,1
6	1	-1	1	1500	20	4	58.9	26.1	61.7	55.3	45,0	27,0
7	-1	1	1	600	60	4	49.3	32.7	60.3	57.5	45,3	31,7
8	1	1	1	1500	60	4	62.7	45.0	67.2	65.3	48,1	42,6
9	-0.1111	-1	-1	1000	20	2	54.1	42.2	62.2	60.3	47,7	42,3
10	-0.1111	1	1	1000	60	4	56.9	35.8	65.1	61.6	45.8	37,6
11	-1	0	-1	600	40	2	42.4	38.2	63.8	60.3	45,3	43,5
12	1	0	1	1500	40	4	61.7	38.4	65.3	62.5	45,5	36,5
13	-1	-1	0	600	20	3	44.3	22.2	59.2	51.8	46,1	29,9
14	1	1	0	1500	60	3	59.3	48.7	69.1	67.3	51,1	47,1

4.2. Calculation of the parameters of photo-polymerization process with MatLab software.

The abovementioned condition for optimization led to incorrect solutions of the equations of regression analysis for some regimes of UC *Evetric*. These incorrect solutions were referred to the light curing parameters in which the values of the micro-hardness on the top surface were less than the acceptable ones. In other words, the difference in the micro-hardness between the top and bottom surfaces was larger than 20%. For that reason, *MatLab* software based algorithm was developed to calculate the micro-hardness on the top and bottom surfaces as well as the layer thickness, which met the requirement for maximum micro-hardness and 20% difference.

With the use of the specially designed program for each of the studied composites maximum hardness on top surface was calculated, 80% hardness on the lower surface and layer thickness were also calculated, which guarantee it for variations of irradiation time of 20, 40 and 60 s and with intensity of the LCUs studied in tasks 1 and 2, which is in the range 600-1500 mW/cm^2.

RESULTS and DISCUSSION on TASK 1

Investigation of the relationship between the light intensity of wireless LED LCUs and their battery charge

In the present study, LCUs were divided into two groups depending on the light intensity. The first group includes 6 models with intensity lower than 1200 mW/cm^2, and the second group consists of 4 models with intensity higher than 1200 mW/cm2.

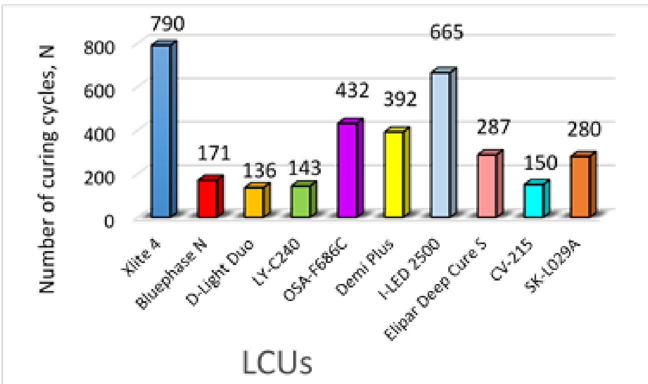


Fig. 7. Battery life of the LCUs, expressed by the number of curing cycles.

In *fig. 7* the battery life of the tested LCUs is shown, expressed by the number of polymerization cycles. It can be clearly seen that there is a big difference in the battery life of the devices in the two groups, but no definite dependence was found either on the light intensity or on the wavelength range.

During the investigation of the light intensity of the 10 LCUs it was found that for 6 of them (LY-C240, SK-L029A, CV-215, OSA-F686C, Xlite 4, D-Light Duo) the actual intensity is lower than the one specified by the manufacturer, and for 4 of them (SK-L029A, CV-215, Xlite 4, OSA-F686C) the intensity decreases with battery discharge, respectively increases with the number of curing cycles. This proves that for some LED LCUs the operating time and battery charge affect the intensity of the light emitted by them. (*fig. 8. u fig. 9.*)

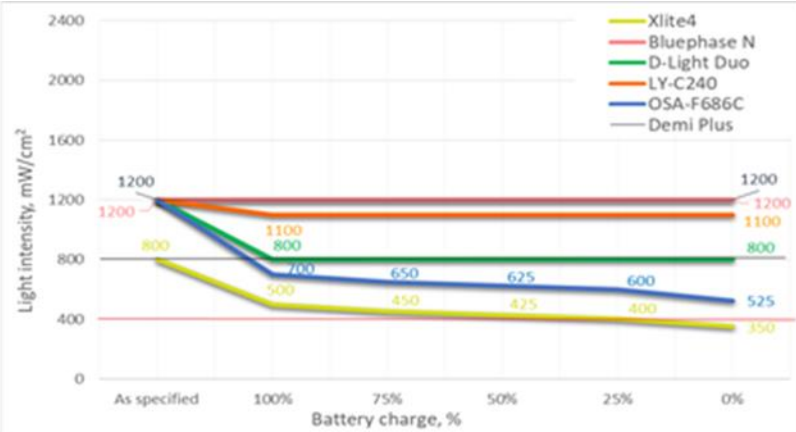


Fig. 8. Change in light intensity of LCUs ($I_L < 1200\ mW/cm^2$) with decrease of battery charge.

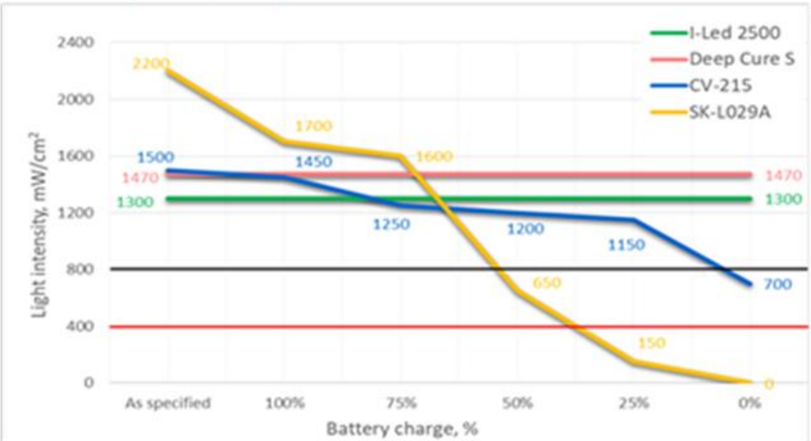


Fig. 9. Change in light intensity of LCUs ($1200 < I_L < 2200\ mW/cm^2$) with decrease of battery charge.

The present study shows that for 60% of the devices tested, the actual light intensity does not correspond to that specified by the manufacturer. For 40% of the units the intensity decreases when battery gets discharged, and for some it falls below the minimum required 400 mW/cm^2 (to achieve adequate polymerization of RBCs the light intensity of the LCUs must be at least 400 mW/cm^2 with 60 seconds irradiation time [Rueggeberg FA et al, 1994]) .

Conclusions:

- *For some devices (LY-C240, SK-L029A, CV-215, OSA-F686C, Xlite4, D-Light Duo), the light intensity is lower than that specified by the manufacturer, which may cause incorrect determination of the optimum polymerization time.*
- *For six of the LCUs tested - Bluephase N, D-Light Duo, LY-C240, Demi Plus, I-LED 2500 and Elipar Deep Cure S, the light intensity is stable and independent of the battery life.*
- *For the rest of models (SK-L029A, CV-215, Xlite4, OSA-F686C), the battery discharge causes a decrease in the light intensity. Recommendations have been given for the effective use of these LCUs to obtain high quality restorations.*
- *Dentists need to periodically measure the light intensity of LCUs and regularly recharge them, especially for the battery-dependent models.*
- *In order for this research to be completed, further longer and more complex studies are needed to provide information on the stability of the light intensity of LCUs as they age.*

RESULTS and DISCUSSION on TASK 2

Investigation of the light intensity of LED light curing units after different periods of use to establish the relationship between the time of use of the devices and their light intensity.

- ***LCUs from group 1***

In the first group of LCUs (24 devices from three different brands) the measurements were made after 1, 3 and 5 years of use. It is noteworthy that for only one device the light intensity measured was lower than 400 mW/cm^2, which makes it unusable. At the same time, however, all devices showed a decrease in intensity varying from 2% to 83%. On the one hand this difference is due to the difference in the models and brands, and on the other hand - to the different intensity of use (the more curing cycles one LCU performs - the more its light intensity decreases). The comparative analysis of the average decrease after 3 years of use, shows that it is the smallest for Smart Xpress (Bluedent) - 0%, followed by DB-686 DELI (Coxo) - 13.2%, and the largest for SK-L029A (Spark Dental) - 37.5%. This sequence is preserved also after 5 years of operation, respectively Smart Xpress - 16.2%, DB-686 DELI - 20.1%, and SK-L029A - 52.3%. (*fig. 10, 11. and 12.*)

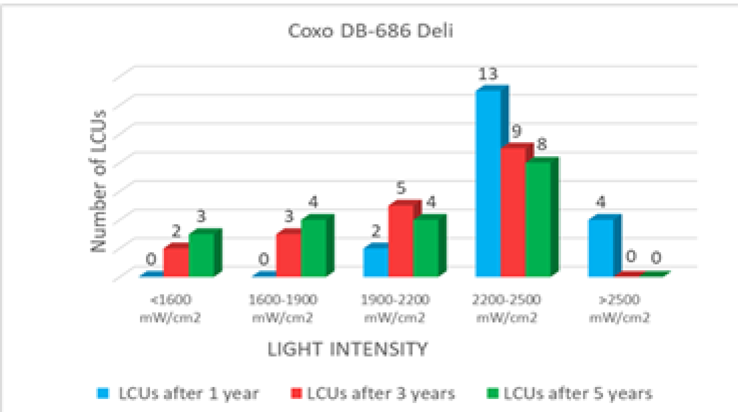


Fig. 10. Measurement of the light intensity of LCU DB-686 Deli (Coxo) after 1, 3 and 5 years of use.

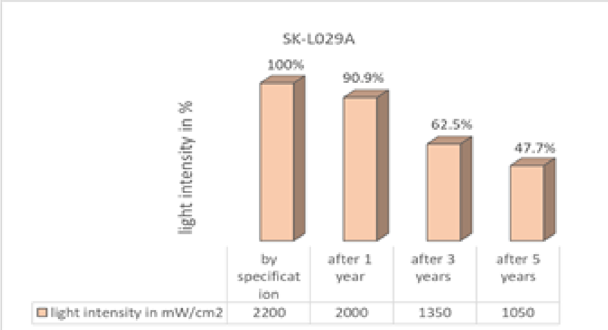


Fig. 11. Change in light intensity of LCU SK-L029A (Spark Dental) during 5 years period of use.

Fig. 12. Change in light intensity of LCU Smart Xpress (Bluedent) during 5 years period of use.

- ***LCUs from group 2***

The second group of LCUs includes 30 devices from 2 different models of the same brand - Minident and Masterdent of Vigodent, tested respectively after 7 years and 9 years of use. It should be noted that the light intensity of all tested LCUs is between 800 mW/cm^2 and 1300 mW/cm^2. None of the devices has intensity below the required minimum of 400 mW/cm^2 and none of them has intensity less than 700 mW/cm^2, as specified by the manufacturer. These results confirm once again the thesis that the model and frequency of use are of great importance for the reduction of light intensity with the progression of the clinical age of the devices.

- ***LCUs from group 3***

The most drastic change in light intensity was found in the LCUs of the third group - LD Max (Gnatus) - 31 out of 40 devices or 77.5% are unusable because the light intensity is below the minimum required 400 mW/cm^2 (*fig. 13.*)

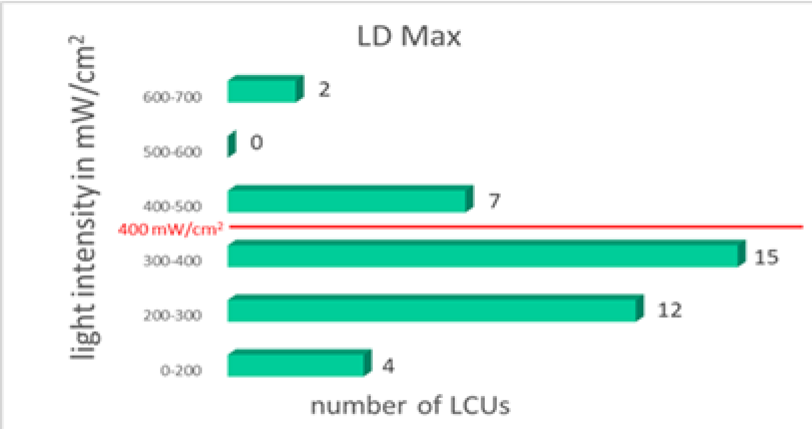


Fig. 13. Measurement of the light intensity of LCU LD Max (Gnatus) after 10 years of use.

The present study shows that dentists should be well acquainted with the characteristics of their LCUs and periodically check them with a radiometer, because as the clinical age of the devices increases, their light intensity decreases. The measurement is necessary for two reasons: first, because the bright blue light from the LCU can have a very low intensity and second, because the surface of the composite may be seemengly hard, but at the bottom of the restoration the degree of monomer-polymer conversion may be low. In some cases, the drop in the intensity is insignificant, but in others it is drastic and requires the devices to be repaired or replaced 34% -

approximately 1/3 of the LCUs investigated in the present study are unusable after 10 years of use.

Conclusions:

- *Regardless of the type and model of LCUs, there is a direct relationship between the time of use and light intensity - the longer the period of operation of a device is and the more used it is, the lower its intensity is.*
- *The decrease in the light intensity with increase of the clinical age of the devices is different for different models, as well as for different devices of the same model.*
- *Approximately 1/3 or 34%% of the studied LCUs with a 10-year period of use have a light intensity lower than the required minimum of 400 mW/cm^2, which makes them unusable.*
- *Dentists should regularly monitor and measure the light intensity of their LCUs, especially with increase of their period of use to ensure the longevity of the restorative procedures.*

RESULTS and DISCUSSION on TASK 3

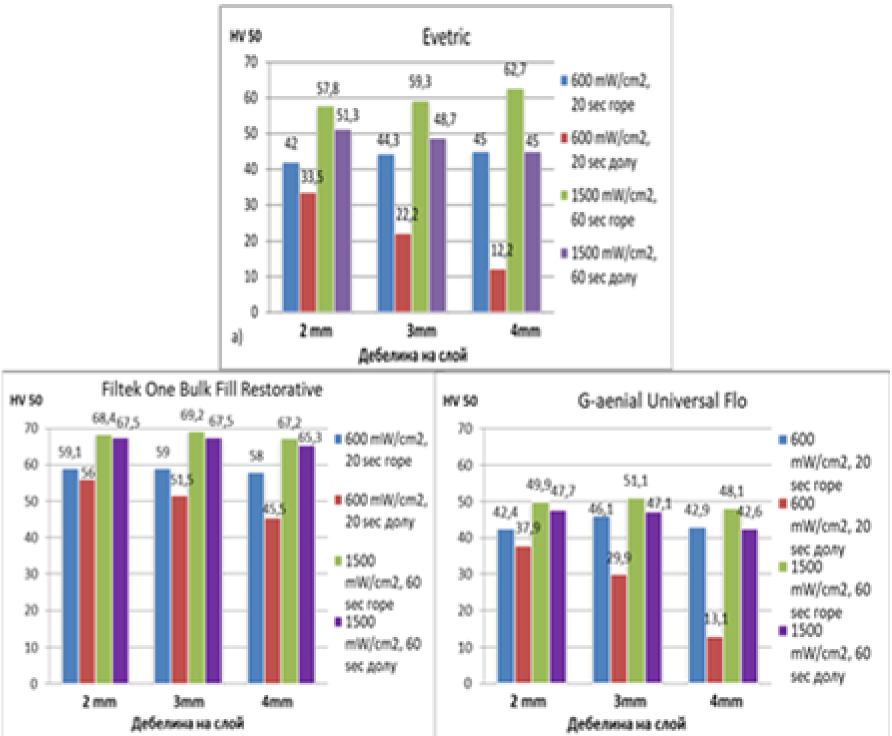
Influence of the factors of the photopolymerization process on the hardness of dental composites

In the present task the influence of the three main factors of photopolymerization - light intensity, irradiation time and layer thickness, on the hardness of the three studied composites is studied.

A guideline to determine if the bottom of a resin composite is adequately cured, it has been suggested that there should be no more than a 20% difference between the maximum hardness on the top of the composite and that on the bottom [Price RB et al, 2003; Yap AU et al, 2003; Bouschlicher MR et al, 2004]. In addition, a bottom-to-top KHN ratio of 80% has been reported to correspond to a bottom-to-top degree-of-conversion ratio of 90% [Price RB et al, 2003].

3.1. Hardness of the studied composites

Under the same polymerization conditions, different Vickers microhardness values were obtained for the three composites - FC *G-aenial Universal Flo* with the lowest hardness (42.4-51.1 HV on the top surface/ 13.1-47.1 HV on the bottom), followed by UC *Evetric* (42-62.7 HV/ 12.2-51.3 HV), and BC *Filtek One Bulk Fill Restorative* with the biggest (58-69.2 HV/ 45.5-67.5 HV) (*fig. 13*). The obtained



Фиг. 13. Hardness of the studied composites obtained under the same light curing conditions.

results can be explained by the differences in the composition of the materials - the organic matrix/filler ratio on the one hand, and on the other - the type of inorganic filler particles. In FC *G-aenial Universal Flo*, whose viscosity is the lowest, the weight ratio of matrix/filler is 31/69%. The high content of organic matrix in the composition determines its relatively low HV values.

BC *Filtek One Bulk Fill Restorative* is the composite with the highest hardness, although it contains a larger amount of organic matrix than UC *Evetric* - the matrix/filler ratio for the first is 23.5/76.5%, and for the second - 19-20/80-81%. These results can be explained by the differences in the composition of the inorganic component - in BC *Filtek One Bulk Fill Restorative* are incorporated ceramic and zirconium particles that have high hardness. These fillers are not found in the composition of UC *Evetric*, but on the other hand there are prepolymers that are characterized by lower hardness.

When comparing the difference in hardness between the top and bottom surface of the three composites, it was found that in UC *Evetric* a difference above the permissible 20% occurs in 41% of the tested samples. For FC *G-aenial Universal Flo* a better polymerization of the material is observed in its entire volume even with a greater layer thickness of 3 and 4 mm - the difference in the hardness between the top and bottom surface is over 20% in 29% of the cases. Extremely close values of HV on the top and bottom surfaces are obtained with BC *Filtek One Bulk Fill Restorative*, as only 7% of the tested samples gave a difference in hardness greater than 20%.

The analysis of the results showed that different photopolymerization factors affect the hardness on both surfaces of the composites in different ways.

At constant irradiation times and layer thickness, as the light intensity increases, the hardness increases approximately evenly on both surfaces for all three composites (*fig. 14.*). The increase in hardness is most pronounced for UC *Evetric*, followed by FC G-*aenial Universal Flo*, and the weakest is the effect of intensity for BC *Filtek One Bulk Fill Restorative*.

Increasing the irradiation time at unchanged light intensity and layer thickness leads to an increase in the hardness mainly on the bottom surface of the three materials (*fig. 15*). Curing time has the strongest influence on the hardness on the bottom surface of UC *Evetric* and FC *G-aenial Universal Flo*.

At constant light intensity and irradiation time, however, the change in layer thickness has a similar effect on the hardness on the top surface - it remains almost unchanged, but affects in a different way the hardness on the bottom surface (*fig. 16).* In UC *Evetric*, the increase in thickness leads to a big decrease in the hardness on the bottom of the restoration. In BC *Filtek One Bulk Fill Restorative* the opposite is observed - the increase in the thickness of the layer does not lead to a change in the hardness on the bottom surface. FC *G-aenial Universal Flo* occupies a middle ground between the other two composites, because with low intensity and short curing time the hardness on the bottom of the material decreases significantly with increasing thickness, and at high intensity and prolonged polymerization time the hardness remains relatively unchanged.

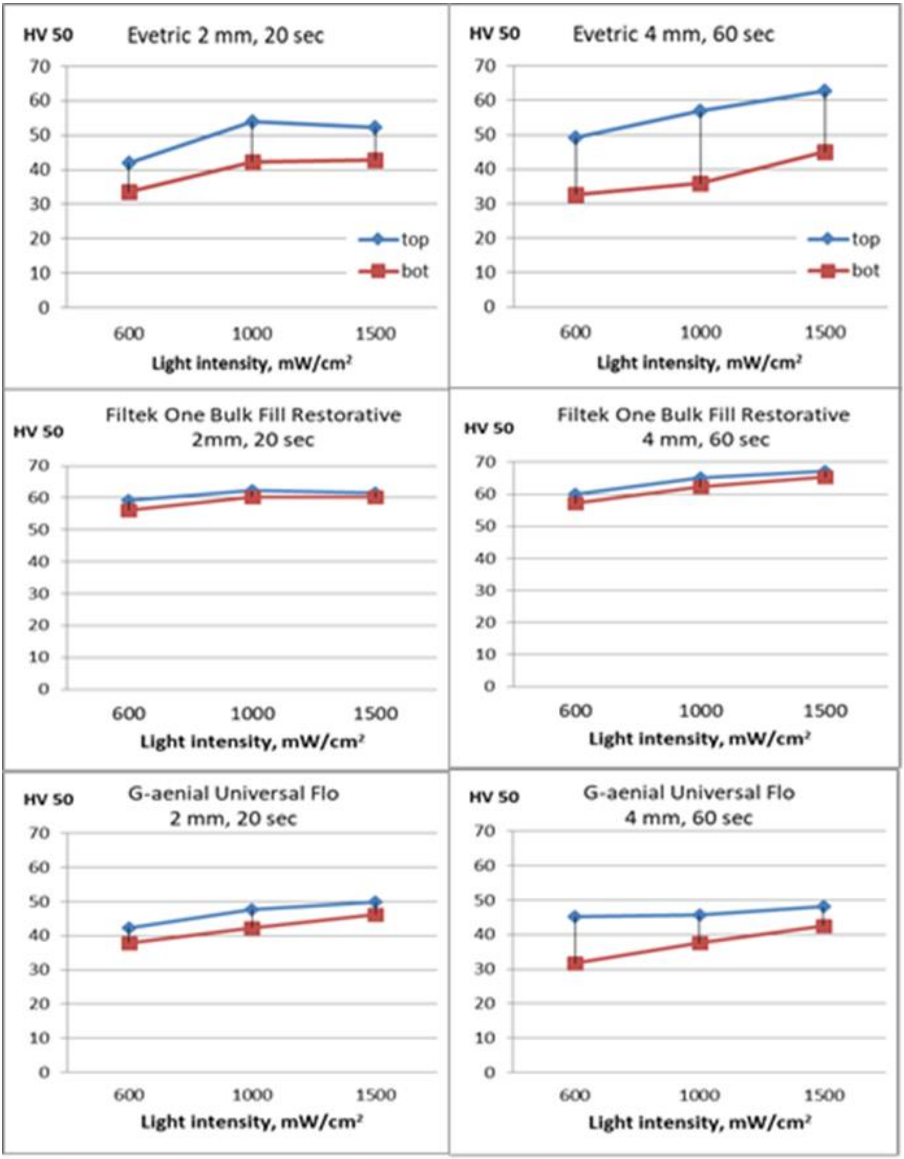


Fig. 14. Influence of light intensity on the hardness of the studied composites at a layer thickness and irradiation time of 2 mm/20 s and 4 mm/60 s, respectively.

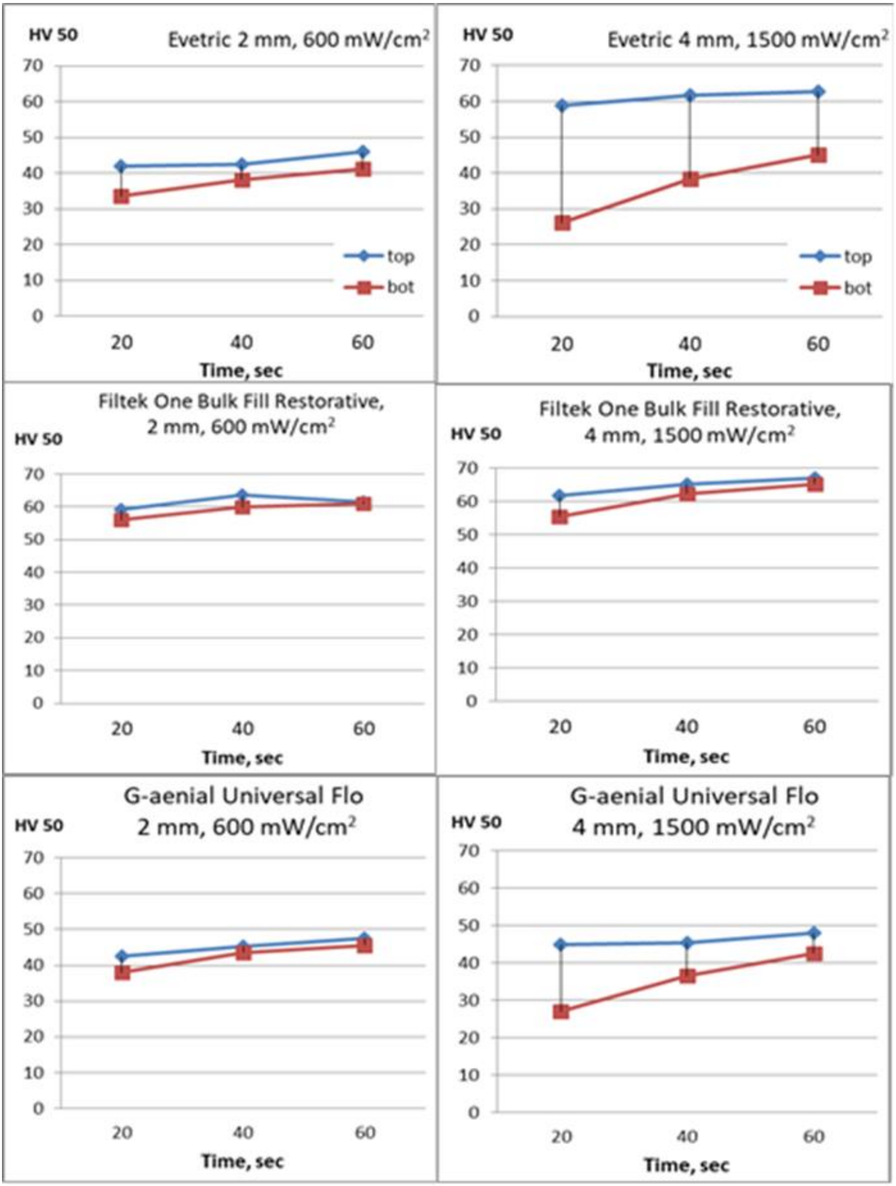


Fig.. 15. Influence of irradiation time on the hardness of the studied composites at layer thickness and light intensity 2 mm/ 600 mW / cm2 and 4 mm/ 1500 mW / cm2, respectively.

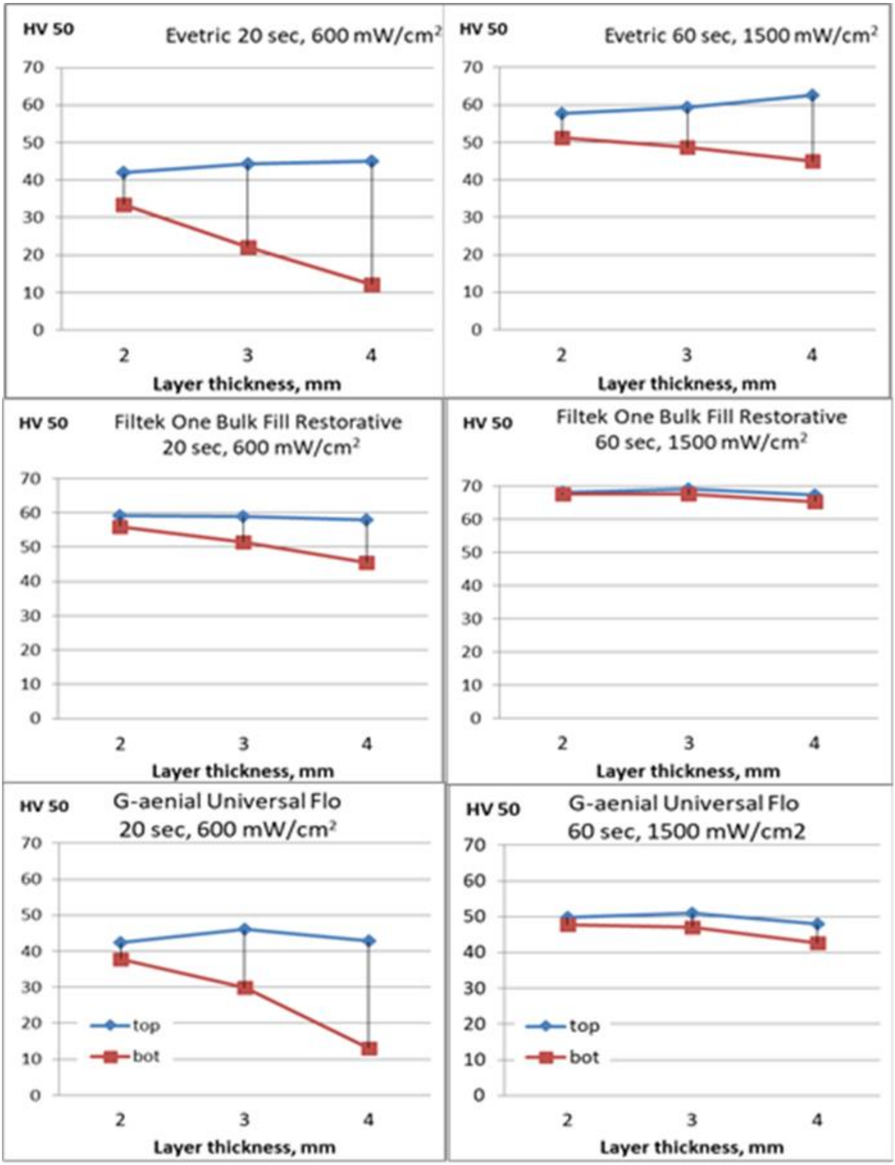


Fig. 16. Influence of the layer thickness on the hardness of the studied composites at light intensity and irradiation time of 600 mW/cm² / 20 s and 1500 mW/cm² / 60 s, respectively.

The results of the 4 additional Vickers microhardness measurements of UC *Evetric* in an interval of 28 days showed that the behavior of the material is the same in all three combinations of parameters - up to the 7th day there was an increase in hardness on both surfaces - between 11% and 20% for the top surface and 9% and 18% for the bottom surface. This higher hardness was maintained for a period of 14 days - until the 21st day, after which the last measurement on the 28th day the hardness of the material decreased almost to its original values.

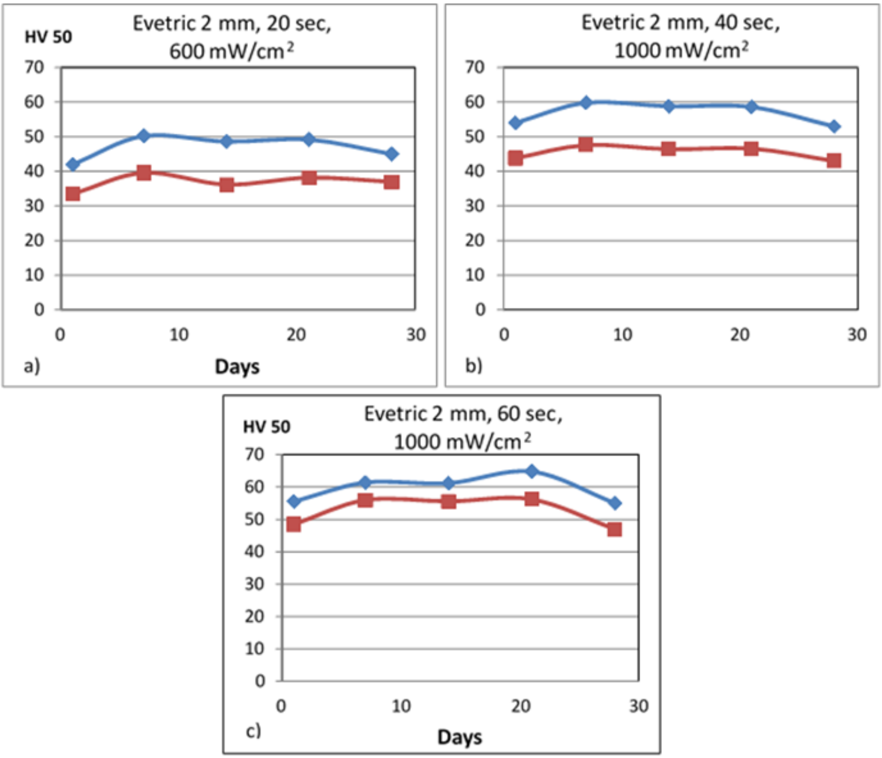


Fig. 17. Influence of the factor aging on the hardness of UC Evetric at a layer thickness of 2 mm and light intensity / irradiation time: 600 mW/cm² / 20 s - a), 1000 mW/cm² / 40 s - b) and 1000 mW/cm² / 60 s - c).

The increase in hardness during the first week confirms the results of Yilmaz EÇ et al, 2016 and Yilmaz EÇ et Sadeler R, 2018, and this is probably due to the ongoing polymerization process throughout the volume of the composite. On the other hand, some authors [Deliperi S, 2002; van Dijken JW et Pallesen U, 2011; Subbiya A et al, 2015; Baroudi K et Mahmoud S, 2015] has proven that polymerization shrinkage leads to stresses in the composite restoration, which will further affect the hardness. Over time, the relaxation of internal stresses begins and this leads to a corresponding decrease in hardness. **Further research is needed to prove this hypothesis.**

3.2. Influence of the factors of the photopolymerization process on the hardness of the studied composites

The influence of the factors of the photopolymerization process - light intensity, irradiation time and layer thickness was studied by ANOVA (analysis of variance). This method, on the one hand, confirms the influence of each factor on the hardness of UC *Evetric*, established by *Excel* software, and on the other hand - allows to determine the significance of individual factors and their mutual influence.

From the graphs of *fig. 18* can be seen that the most significant factor for the hardness on the top surface of UC *Evetric* is the light intensity (x1), followed by the layer thickness (x3) and the irradiation time (x2). Therefore, the intensity of the LCU has the greatest influence on the hardness on the top surface of the composite restoration. Maximum hardness on the top surface is obtained at maximum values of the three parameters - intensity, time and layer thickness (1500 mW/cm^2 /60 s /4 mm), and minimum - at minimum values of the parameters (600 mW/cm^2 /20 s /2 mm).

The most significant factor in BC *Filtek One Bulk Fill Restorative* is the light intensity, followed by the irradiation time and

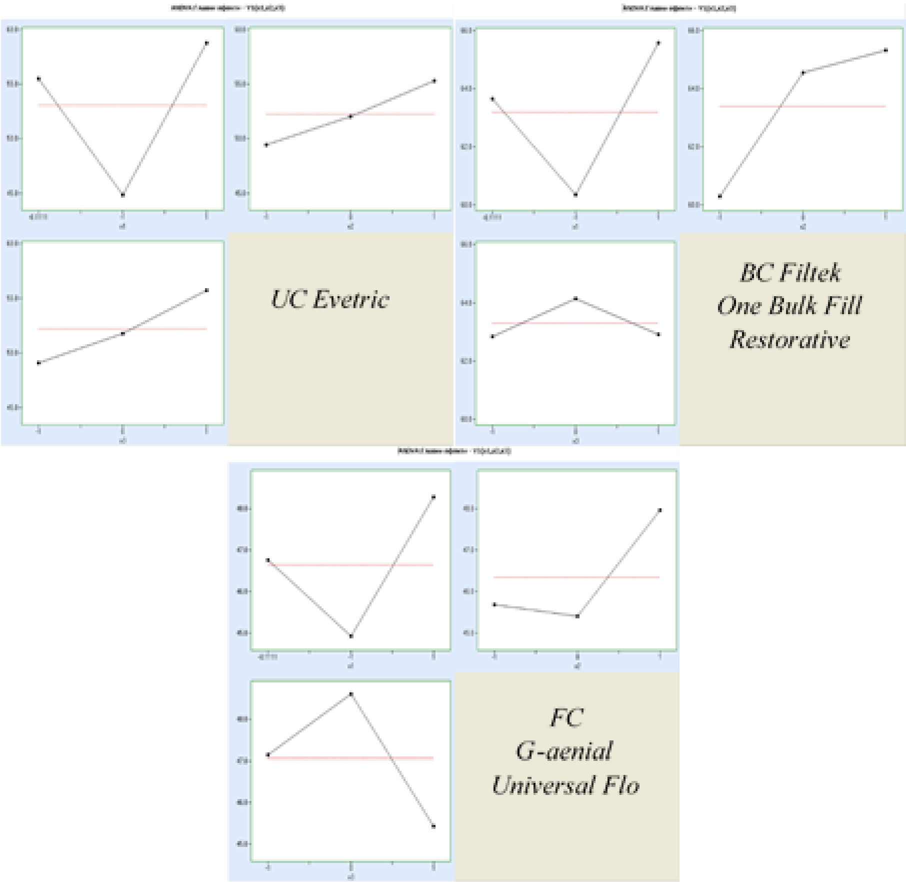


Fig. 18. Influence of the individual factors of photopolymerization on the hardness on the top surface of the studied composites.

the layer thickness (*fig. 18*). Maximum surface hardness is obtained when the light intensity and irradiation time have the highest values (1500 mW/cm^2 and 60 s) at an average layer thickness of 3 mm. Working with the minimum values of the three factors (600 mW/cm^2, 20 s and 2 mm) ensures minimum hardness on the top surface.

For FC *G-aenial Universal Flo*, similar to UC *Evetric*, the most significant factor for top surface hardness is the intensity, followed by thickness and time (*fig. 18*). But in contrast, individual factors have different effects on the objective function. Maximum hardness on the top surface is obtained with maximum values of the intensity and irradiation time (1500 mW/cm^2 and 60 s) and with an average layer thickness of 3 mm. The minimum hardness is obtained by working with a minimum intensity of 600 mW/cm^2, an irradiation time of 40 s and a maximum layer thickness of 4 mm.

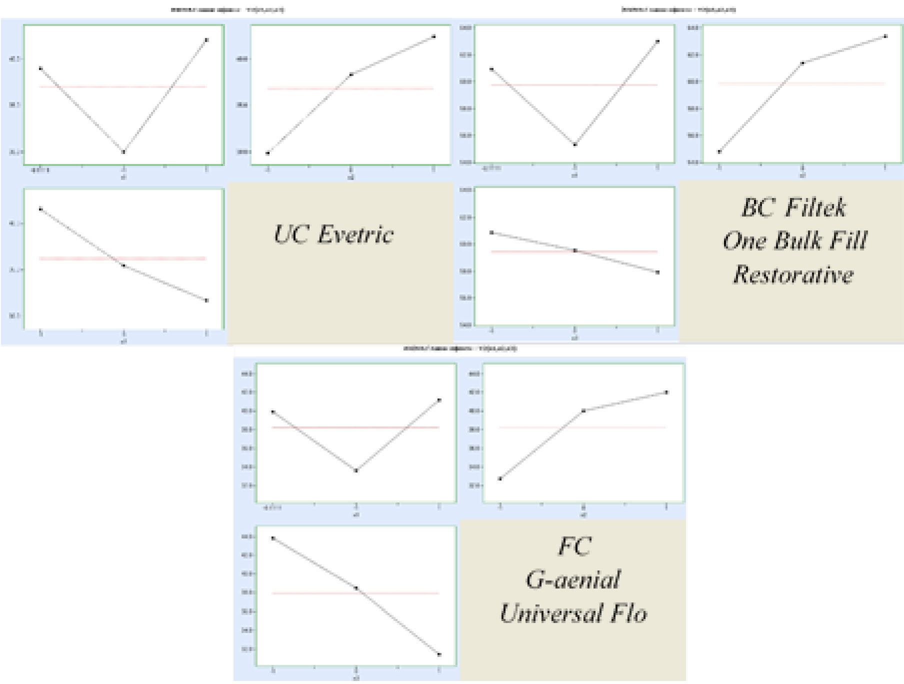


Fig. 19. Influence of the individual factors of photopolymerization on the hardness on the top surface of the studied composites.

Regarding the influence of the individual factors on the hardness on the bottom surface of the UC *Evetric* restoration, the most significant factor is the layer thickness (x3), followed by the irradiation time (x2) and the light intensity (x1) (*fig. 19*). Maximum/minimum hardness on the bottom surface is obtained with maximum/minimum values of the first two parameters - intensity and time (1500 mW/cm^2 and 60 s / 600mW/cm^2 and 20 s), and the values of the third parameter - layer thickness are respectively minimum/maximum (2 mm/4 mm).

For the hardness on the bottom surface of BC *Filtek One Bulk Fill Restorative* (*fig. 19.*), however, the most significant factor is time, followed by light intensity and layer thickness. Maximum/minimum hardness on the bottom surface is obtained with maximum/minimum values of intensity and time (1500mW/cm^2 and 60 s / 600 mW/cm^2 and 20 s) and with minimum/maximum layer thickness, respectively 2 mm/4 mm.

For the hardness on the bottom surface of FC *G-aenial Universal Flo*, the thickness of the layer is of the greatest importance, followed by the irradiation time and the light intensity. Here, as with UC *Evetric*, maximum/minimum hardness on the bottom surface is obtained with maximum/minimum values of the first two parameters - intensity and time (1500 mW/cm^2 and 60 s / 600 mW/cm^2 and 20 s), while the values of the third parameter - layer thickness are minimum/maximum (2 mm /4 mm).

Conclusions:

- *The present study showed that BC Filtek One Bulk Fill Restorative has the highest hardness (65 +/- 4 HV), followed by UC Evetric (56 +/- 4 HV), and FC G -aenial Universal Flo with the lowest(47 +/- 4 HV).*
- *BC Filtek One Bulk Fill Restorative is the composite with the highest hardness and the smallest difference in hardness between the top and bottom surface (1-22%) regardless of the layer thickness. This shows the superiority of bulk fill over conventional composites in terms of the degree of convertion.*
- *UC Evetric occupies an intermediate position in terms of hardness compared to BC and FC. In this composite the difference in hardness between the top and bottom surface is the largest (11-73%). When light curing a layer with 4 mm thickness, the*

difference in hardness exceeds 20% in all combinations of intensity and time.

- *The hardness of FC G-aenial Universal Flo is the lowest compared to the other two composites in all curing modes. This should be taken into account when restoring extensive defects on the occlusal surface of posterior teeth, in patients with strong masticatory muscles and/or parafunctions. The difference in hardness between the top and bottom surface vary between 4-69%, and the large difference is observed when working with low intensity, short irradiation time and large layer thickness.*
- *Regardless of the dental composite, the intensity of light affects the hardness evenly in the entire volume of the material, and the irradiation time mainly affects the hardness on the bottom part of the composite layer.*
- *As the layer thickness increases, the hardness on the top surface of the composite does not change. The effect on the hardness on the bottom surface varies depending on the type of the composite - from a slight change in BC Filtek One Bulk Fill Restorative to a strong decrease in UC Evetric.*
- *The most significant factor influencing the top surface hardness of all three composites is the light intensity, followed by the layer thickness and the irradiation time for UC Evetric and FC G-aenial Universal Flo. For BC Filtek One Bulk Fill Restorative, the irradiation time is in second place, followed by the layer thickness.*
- *The light curing parameters that provide maximum /minimum hardness on the top surface are different for the examined dental composites:*
    - *For UC Evetric maximum/minimum hardness is obtained with maximum/minimum values of the three parameters - intensity, time and layer thickness (1500 mW/cm^2, 60 s and 4 mm / 600 mW/cm^2, 20 s и 2 mm).*
    - *For BC Filtek One Bulk Fill Restorative, maximum hardness on the top surface is obtained with the highest values of intensity and time (1500 mW/cm^2 and 60 s) and an average layer thickness of 3 mm. Working with the*

minimum values of the three factors (600 mW/cm^2, 20 s and 2 mm) guarantees minimum hardness.

  - *For FC G-aenial Universal Flo, the maximum hardness on the top surface is obtained with maximum values of intensity and time (1500 mW/cm^2 and 60 s) and an average layer thickness of 3 mm. Minimum hardness is obtained when working with a minimum intensity of 600 mW/cm^2, an irradiation time of 40 s and a maximum layer thickness of 4 mm.*

- *The parameters of light curing, which provide maximum/minimum hardness on the bottom surface are identical for the studied dental composites - maximum/minimum values of intensity and time (1500 mW/cm^2 and 60 s / 600 mW/cm^2 and 20 s) and minimum/maximum layer thickness of 2 mm/4 mm.*
- *For all composites, the highest hardness of the restoration can be obtained by combining the highest intensity, the longest duration and the smallest layer thickness.*
- *Uneven hardness of UC Evetric was found for a 28-day period of time - increase by 9-20% until the 7th day, maintaining this higher hardness until the 21st day and decrease to the initial values on the 28th day.*

RESULTS and DISCUSSION on TASK 4

Optimization of the parameters of the process of photopolymerization of dental composites

For each composite, layer thickness and hardness on the top and bottom surfaces were calculated for 21 light curing modes with light intensity in the range 600-1500 mW/cm^2 and irradiation times of 20, 40 and 60 s.

4.1. UC Evetric

The regression models for the objective functions Y_1 - hardness on the top surface of the composite layer and Y_2 - hardness on the bottom surface of UC *Evetric* are shown in *formulas (5)* and *(6)*:

$$Y_1 = 56.266 + 6.402x_1 + 1.5325x_2 + 2.0025x_3 - 4.458x_1^2 \qquad (5)$$

$$Y_2 = 35.936 + 6.118x_1 + 6.459x_2 - 6.991x_3 + 1.189x_1x_3 + 3.089x_2x_3 \qquad (6)$$

Using regression models, for UC *Evetric* optimizations were made in nine variations of the governing factors intensity (x_1) and irradiation time (x_2) in order to obtain a maximum hardness on the top surface of the composite, a hardness on the bottom surface equal to 80% of that on the top surface, and a layer thickness that provides them. As a condition for the optimization of UC *Evetric* is accepted hardness on the top surface 56 +/- 4 HV, and hardness on the bottom surface - 80% of 56 +/- 4 HV.

Table 8.

Optimal regimes for light curing of UC Evetric obtained by regression analysis.

№	**I mW/cm²**	**t s**	**d mm**	**HV Top**	**HV Bottom**	**Note**
1	1500	60	3,10	60,0	48,0	Regimes that meet the requirement of max HV 56 +/- 4 on the top surface and HV on the bottom surface ≥ 80%.
2	1500	40	3,05	58,3	41,6	
3	1500	20	2.35	55.3	41.6	
4	1000	60	2,85	57,0	43,0	
5	1000	40	2,15	53,9	41,6	
6	1000	20	Incorrect solution			HV difference between top and bottom surface is larger than 20%.
7	600	60	Incorrect solution			HV values on the top surface are lower than the acceptable 56 +/-4.
8	600	40	Incorrect solution			
9	600	20	Incorrect solution			

It can be seen that in regimes with lower intensity (600 mW/cm^2 and 1000 mW/cm^2/20 s) incorrect solutions are obtained, referring to the lower microhardness values or HV difference between the top and bottom surfaces larger than 20%. This necessitated the application of a new approach for the optimization of the parameters and the development of a program for their calculation through the software product MatLab.

With the help of the newly designed program, maximum micr-hardness on the top surface, 80% micro-hardness on the bottom surface and the layer thickness, which guarantees it, are calculated for variations of irradiation time and LCU intensity. The results obtained are shown in *table 9*.

Table 9.
Parameters of light curing of UC Evetric.

LCUs used in task 1 and task 2	**№**	**Intensity mW/cm^2**	**Times**	**Thickness mm**	**Hardness, HV 50**	
					Top	**Bottom**
Xlite4 (ThreeH, China)	1	600	20	2.09	42.05	33.64
	2	600	40	2.33	44.07	35.26
	3	600	60	2.81	46.56	37.25
OSA-F686C (Osaka Dental,China)	4	700	20	1.97	45.00	36.00
	5	700	40	2.19	46.97	37.58
	6	700	60	2.62	49.36	39.48
D-Light Duo (GC, Japan)	7	800	20	1.88	47.57	38.05
	8	800	40	2.08	49.49	39.59
	9	800	60	2.46	51.80	41.44
LY-C240 (BDMED, China)	10	1000	20	1.78	51.53	41.22
	11	1000	40	2.95	53.40	42.72
	12	1000	60	2.31	55.64	44.51
Demi Plus (Kerr, USA) *Bluephase N* (Ivoclar Vivadent, Lichtenstein)	13	1200	20	1.76	53.96	43.17
	14	1200	40	1.98	55.86	44.69
	15	1200	60	2.39	58.21	46.57
I-LED 2500 (Woodpecker, China)	16	1300	20	1.85	54.61	43.69
	17	1300	40	2.06	56.57	45.26
	18	1300	60	2.54	59.07	47.26
Elipar Deep Cure S (3M ESPE, USA); *CV-215* (Cicada Dental, China); *SK-L029A* (Spark Dental, China); *Smart Xpress* (Bluedent, Bulgaria)	19	1500	20	2.07	54.82	43.85
	20	1500	40	2.39	56.98	45.59
	21	1500	60	3.17	60.08	48.06

DB-686 DELI (Coxo, China)						

The data in *table 9* can be classified into three groups. The first group includes the curing modes that do not provide the necessary microhardness on the top and bottom surfaces, which is a sign of inadequate polymerization. These are the regimes with intensity of 600-700 mW/cm^2 for all irradiation times and intensity of 800 mW/cm^2 for 20 and 40 s curing. The second is the boundary group with two modes: 800 mW/cm^2/60 s and 1000 mW/cm^2/20 s, which provide hardness close to the lower limit. Taking into account the microhardness increase of the composite over time, these regimes can be considered acceptable. The third group includes all modes with intensity of 1000 mW/cm^2 for time 40 and 60 s, as well as those over 1000 mW/cm^2. They guarantee maximum micro-hardness on the top surface and 80% HVmax on the bottom surface at the calculated thickness of the composite

Our results show that in this case it is possible to work with a layer thickness of up to 2.39 mm for 1200 mW/cm^2, 2.54 mm for 1300 mW/cm^2 and 3.17 mm for 1500 mW/cm^2. On the other hand, the manufacturer recommends 2 mm composite layer to be light cured for 20 s with LCU intensitiy between 500 and 1000 mW/cm^2 and for 10 s with intensity above 1000 mW/cm^2. The results, obtained by us, disprove these recommendations, as observance of the specified parameters would not lead to satisfactory micro-hardness of the material.

4.2. BC Filtek One Bulk Fill Restorative

For BC *Filtek One Bulk Fill Restorative* the regression models for the objective functions Y_1 and Y_2 are of the following type:

$$Y_1 = 64.55 + 2.327x_1 + 2.256x_2 - 0.704x_3 - 1.866x_2^2 + 1.003x_1x_2 \qquad (10)$$

$$Y_2 = 59.113 + 3.596x_1 + 4.170x_2 - 2.740x_3 + 1.162x_1x_3 + 1.397x_2x_3 \qquad (11)$$

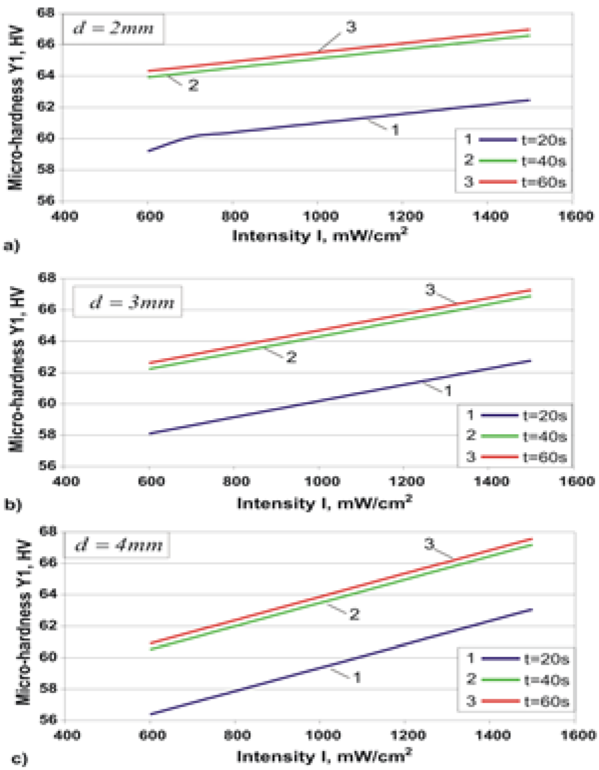
As a condition for the optimization of BC *Filtek One Bulk Fill Restorative* is accepted hardness on the top surface 65 +/-4 HV, and hardness on the bottom surface - 80% of 65 +/-4 HV. The results obtained are shown in *table 10.*

It can be seen that only at I=600 mW/cm^2 and t=20 s there is a

Table 10.
Light curing parameters of BC Filtek One Bulk Fill Restorative.

№	Intensity mW/cm^2	Time s	Thickness mm	Hardness, HV 50		Y_2/Y_1
				Top, Y_1	Bottom, Y_2	
1	600	20	3.86	56.63	45.30	0.80
2	600	40	4.72	59.29	48.81	0.82
3	600	60	8.36	53.47	46.27	0.86
4	700	20	4.03	57.08	46.94	0.82
5	700	40	4.99	59.78	49.07	0.82
6	700	60	9.30	53.78	46.33	0.86
7	800	20	4.23	57.58	47.06	0.82
8	800	40	5.31	60.35	49.31	0.82
9	800	60	10.58	54.08	46.20	0.85
10	1000	20	4.71	58.78	47.26	0.80
11	1000	40	6.16	61.72	49.66	0.80
12	1000	60	15.35	54.61	44.70	0.82
13	1200	20	5.34	60.34	47.38	0.78
14	1200	40	7.50	63.66	49.73	0.78
15	1200	60	32.66	54.75	36.13	0.66
16	1300	20	5.73	61.32	47.39	0.77
17	1300	40	8.51	65.03	49.57	0.76
18	1300	60	91.26	53.28	The results have no physical sense	
19	1500	20	6.79	63.89	47.27	0.74
20	1500	40	12.07	69.59	48.39	0.70
21	1500	60	The results have no physical sense			

thickness of 3.86 mm in the range 2-4 mm, where the bottom/top micro-hardness ratio is equal to 0.8 and the accuracy of the calculated hardness (56.63 HV on top and 45.30 HV on bottom) is guaranteed. For all other "intensity - time" combinations in the table, the calculated limit thickness is larger than the upper limit of the range 2-4 mm. In this composite, for all combinations in the table (except for the first), the bottom/top micro-hardness ratio is higher or lower than 0.8. For the calculated thicknesses larger than 4 mm, i.e. outside the defined range 2-4 mm, the accuracy of the calculated hardness is not guaranteed, as the regression models are valid only for the intervals in which the governing factors are changed.



Фиг. 20. Dependence of the microhardness on the top surface on the intensity, time and thickness.

The graphs in *fig. 20* show the dependence of the microhardness Y_1 on the top surface on the intensity, irradiation time and thickness. It can be clearly seen that for the three thicknesses (2, 3 and 4 mm) the increase of the irradiation time over 40 s is practically unnecessary, as the micro-hardness on the top surface increases insignificantly. Using *fig. 20* and according to the specific conditions, an optimal combination of photo-polymerization parameters can be selected to ensure the minimum allowable hardness of 61 HV (the blue horizontal line of all graphs).

For comparison, the manufacturer recommends the irradiation time for 4 mm layer to be 40 s with intensity in the range 550-1000 mW/cm^2 and 20 s with intensity above 1000 mW/cm^2 [Technical Product Profile. Filtek One Bulk Fill Restorative]. The results of our study show that a satisfactory micro-hardness can not be obtained by polymerization for 40 s with intensity of 550-700 mW/cm^2 and for 20 s with intensity of 1000-1250 mW/cm^2.

The parameters of photo-polymerization of BC *Filtek One Bulk Fill Restorative* are summarized in *table 11*. These parameters provide the minimum allowable hardness 61 HV on the top surface and hardness on the bottom surface - 80% of that of the top for layer thickness between 2-4 mm.

Table 11.

Optimal parameters for light curing of BC Filtek One Bulk Fill Restorative.

№	**Inensity mW/cm^2**	**Time s**	**Layer thickness mm**
1	I>1000	20	2
2	600-1500	40	2
3	I>1000	20	3
4	600-1500	40	3
5	I>1250	20	4
6	700-1250	40	4
7	I<700	60	4

4.3. FC G-aenial Universal Flo

The regression models for the objective functions Y_1 and Y_2 of FC *G-aenial Universal Flo* have the following form:

$$Y_1 = 46.762 + 1.715x_1 + 1.054x_2 - 1.326x_3 - 0.766x_1x_3 + 0.730x_1x_2x_3 \quad (13)$$

$$Y_2 = 37.091 + 4.214x_1 + 5.271x_2 - 7.769x_3 + 1.994x_1x_3 + 3.185x_2x_3 \quad (14)$$

As a condition for the optimization of FC *G-aenial Universal Flo* is accepted hardness on the top surface 47 +/-4 HV, and hardness on the bottom surface - 80% of 47 +/-4 HV. The results obtained are shown in *table 12.*

The data analysis shows that all light curing regimes satisfy the requirement for the surface micro-hardness. It is noteworthy that two modes lead to lowest hardness on the top surface – regime 1, which is characterized by the lowest parameters intensity and time, and regime 21 - with the highest parameters. The other combinations of parameters provide HVmax in the range 44.46 - 47.50 HV**.** It is noteworthy that the maximum micro-hardness values of 46-48 HV on the top surface are obtained at layer thicknesses between 2.51-4.77 mm

According to the recommendations of the manufacturer, 1.5 mm layer should be cured for 20 s with LCU intensity of 700 mW/cm^2 and for 10 s with intensity of 1200 mW/cm^2 [Technical Manual. G-aenial Universal Flo]. The results of our study confirm that if these guidelines are followed, a satisfactory hardness or degree of polymerization of FC *G-aenial Universal Flo* could be obtained.

Table 12.

Light curing parameters of FC *G-enial Universal Flo.*

LCU	**№**	**Intensity mW/cm²**	**Time s**	**Thickness mm**	**Hardness, HV 50**	
					Top	**Bottom**
Xlite4 (ThreeH, China)	1	600	20	2.42	43.89	35.11
	2	600	40	2.66	45.24	36.19
	3	600	60	3.23	45.81	36.65
OSA-F686C (Osaka Dental,China)	4	700	20	2.44	44.46	35.57
	5	700	40	2.71	45.64	36.51
	6	700	60	3.37	46.00	36.80
D-Light Duo (GC, Japan)	7	800	20	2.46	45.02	36.02
	8	800	40	2.77	46.02	36.82
	9	800	60	3.54	46.15	36.92
LY-C240 (BDMED, China)	10	1000	20	2.51	46.09	36.87
	11	1000	40	2.91	46.68	37.35
	12	1000	60	4.01	46.29	37.03
Demi Plus (Kerr, USA)	13	1200	20	2.57	47.06	37.65

Bluephase N (Ivoclar Vivadent, Lichtenstein)	14	1200	40	3.11	47.16	37.73
	15	1200	60	4.77	46.01	36.81
I-LED 2500 (Woodpecker, China)	16	1300	20	2.61	47.50	38.00
	17	1300	40	3.24	47.29	37.84
	18	1300	60	5.37	45.58	36.46
Elipar Deep Cure S (3M ESPE, USA); *CV-215* (Cicada Dental, China) *SK-L029A* (Spark Dental, China); *Smart Xpress* (Bluedent, Bulgaria); *DB-686 DELI* (Coxo, China)	19	1500	20	2.72	48.22	38.58
	20	1500	40	3.61	47.19	37.75
	21	1500	60	7.63	43.22	34.58

Conclusions:

- *In the present study, optimization of the parameters of photo-polymerization process of dental composites from three different groups was conducted using regression analysis.*
- *For all composites, regression models for the micro-hardness on top and bottom surfaces of the composite layer were established.*
- *Both the layer thickness and the micro-hardness on the samples top and bottom surfaces of each composite were calculated for 21 modes of light curing varying with the light intensity in the range 600-1500 mW/cm^2 and irradiation time – 20, 40 and 60 s.*
- *It is established that for UC Evetric the required micro-hardness of 56+/-4 HV on the top surface is not provided by modes with intensity below 800 mW/cm^2. When the intensity is equal to 800 mW/cm^2, the required microhardness is guaranteed only at irradiation of 60 s. For LCUs with intensity in the range 1000-1500 mW/cm^2 it is possible to work with all irradiation times. The maximum values of intensity and time ensure successful polymerization of a layer with a thickness of 3 mm.*
- *Recommended modes for BC Filtek One Bulk Fill Restorative are developed, which provide microhardness on the top surface, higher than the minimum allowable (61 HV), when working with layer thickness of 2-4 mm. For layer thicknesses above 4 mm,*

modes with intensity of 1000-1500 mW/cm^2 and irradiation time of 20 and 40 s guarantee the required top surface micro-hardness and bottom/top micro-hardness ratio less than 0.8. Using this composite, layer with a thickness of 5-7 mm can be successfully polymerized.

- *It is established that in FC G-aenial Universal Flo all studied curing regimes meet the hardness requirement, but to ensure its maximum value of 46-48 HV, the layer thickness should not exceed 5 mm.*
- *It is found that the photo-polymerization guidelines only of the FC manufacturer guarantee the required hardness, while the recommended regimes for UC and BC do not satisfy this requirement.*
- *Tables with recommended light curing regimes are developed for the three types of composites, which guarantee high microhardness of the composite filling. They are designed to facilitate the work of dentists in dental offices.*

Conclusion

Along with the exceptional opportunities that photo polymerization provides to dentists, there are a number of problems, the main group of which is related to the incomplete polymerization of dental composites. This, on the one hand, is due to the lack of information of dentists about the characteristics, operation and maintenance of light curing units, and on the other - the low level of knowledge and control over the main factors of photopolymerization.

In the present dissertation it was found that in some LED light curing units the light intensity is lower than specified by the manufacturer, and in some wireless models the discharge of the battery is the reason for its reduction. An inverse relationship between the service life of ligh curing units and their light intensity was also found - the longer and more intensively a unit is used, the lower its intensity is. Due to the stated facts, we recommend dentists to regularly measure the light intensity of their light curing units with a radiometer, especially by increasing the time of their use to ensure higher quality and longevity of their composite restorations.

The results of microhardness tests of dental composites show that the manufacturer's light curing guidelines do not always meet the requirement for high hardness. In these cases, a sufficient degree of polymerization of the composite can not be achieved, which in turn leads to reduced hardness - a prerequisite for reduced wear resistance of the restoration. Therefore, in the dissertation are developed tables with recommended light curing modes for three main types of composites - universal nanohybrid light cured composite *Evetric*, bulk fill light cured composite for posterior restorations *Filtek One Bulk Fill Restorative* and universal flowable light cured composite *G-aenial Universal Flo*. The use of these modes allows the achievement of high hardness of the restorations.

The obtained results in the dissertation, the recommendations made regarding the operation and maintenance of light curing units, as well as the developed recommended regimes for light curing of dental composites are intended for application in dental offices. They will facilitate the daily work of dentists and will guarantee high quality composite restorations.

CONTRIBUTION

SCIENTIFIC AND APPLIED CONTRIBUTIONS

Original

1. An inverse relationship was established between the service life of LED light curing units and their light intensity - the longer and more intensively a unit is used, the lower its intensity is.
2. The importance of the factors of the photopolymerization process was established - light intensity, irradiation time and layer thickness on the hardness of three types of dental composites.
3. The parameters of light curing were established - light intensity, irradiation time and layer thickness, which provide maximum/minimum hardness of the studied composites.
4. Uneven hardness of UC *Evetric* was found for a 28-day period of time - increase by 9-20% by day 7, maintaining higher hardness by day 21 and decrease to the initial values on day 28.
5. Optimization by regression analysis of the parameters of the photopolymerization process was made - light intensity, irradiation time and layer thickness of the studied dental composites.
6. Light curing regimes were calculated and established, which guarantee maximum hardness of the composites at the respective layer thickness.

Confirmatory

1. It was confirmed that BC *Filtek One Bulk Fill Restorative* has the highest hardness (65 +/- 4 HV), followed by UC *Evetric*

(56 +/- 4 HV), and FC *G -aenial Universal Flo* with the lowest (47 +/- 4 HV).

APPLIED CONTRIBUTIONS

Original

1. It was found that not all light curing units have a stable light intensity - in some of them the intensity is lower than specified by the manufacturer, and the discharge of the battery leads to its reduction.
2. It was established that the instructions for light curing of the manufacturers do not always guarantee the optimal hardness for the studied dental composites.
3. Tables with recommended light curing regimes have been created for the three types of tested composites, which guarantee the necessary hardness and are designed to facilitate the work of dentists.

PUBLICATIONS ON THE TOPIC OF THE DISSERTATION

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2. Georgiev G, Dikova T, Panov V. Development of devices for photo polymerization of dental composites. MATERIALS SCIENCE "NONEQUILIBRIUM PHASE TRANSPHORMATIONS". 2019;1(3):139-42.
3. Georgiev G, Panov V, Dikova T. Investigation of light intensity of wireless LED light curing units. J Techn Univ Gabrovo. 2020;60:40-45.

PARTICIPATION IN SCIENTIFIC FORUMS

1. Georgiev G, Panov V, Dikova T. Development of Devices for Photo polymerization of Dental Composites. 29th Annual Assembly of IMAB, 9-12 May 2019, Varna, Bulgaria.
2. Georgiev G, Dikova T, Panov V. Development of devices for photo polymerization of dental composites. V INTERNATIONAL SCIENTIFIC CONFERENCE: MATERIALS SCIENCE "NONEQUILIBRIUM PHASE TRANSPHORMATIONS". 09-12.09.2019, Varna, Bulgaria.
3. Georgiev G, Panov V, Dikova T. Investigation of the light intensity of LED light curing units after different periods of use. Jubilee 30-th Annual Assembly of IMAB, 18-21 October 2021. Varna, Bulgaria.

Application 6

RECOMMENDATIONS and RECOMMENDED REGIMES for EFFICIENT LIGHT CURING OF DENTAL COMPOSITES

This application provides recommendations and recommended regimes for light curing of the three studied composites, which provide:

1) maximum hardness on the top surface,

2) hardness on the bottom surface equal to 80% of that on the top surface and

3) layer thickness, which guarantees it for variations of the irradiation time of 20, 40 and 60 s and at light intensity in the range 600-1500 mW/cm^2.

1. Universal nanohybrid light cured composite *Evetric*

1.1. Reccomendations for efficient light curing:

- Not all curing modes with intensity in the range of 600-1500 mW/cm^2 and irradiation time of 20-60 s provide the necessary hardness on the top surface of UC *Evetric*.
- It is not recommended to use LCUs with an intensity below 800 mW/cm^2.
- For LCUs with an intensity of 800 mW/cm^2, it is necessary to light cure the restoration for 60 s to ensure the required hardness.
- For LCUs with intensity in the range 1000-1500 mW/cm^2 it is possible to work with all irradiation times.
- Increasing the irradiation time from 20 to 60 s leads to an increase of the compsite layer thickness and the hardness.
- When working with maximum values of intensity and time (1500 mW/cm^2 and 60 s), a layer with 3 mm thickness and 60 HV hardness on the top surface can be successfully polymerized.

1.2. Reccomended regimes for UC *Evetric*:

Reccomended regimes for light curing of U Evetric.

№	Intensity mW/cm^2	Time s	Layer thickness mm	Hardness, HV 50	
				Top	Bottom
1	800	60	2.46	51.80	41.44
2	1000	20	1.78	51.53	41.22
3	1000	40	2.95	53.40	42.72
4	1000	60	2.31	55.64	44.51
5	1200	20	1.76	53.96	43.17
6	1200	40	1.98	55.86	44.69
7	1200	60	2.39	58.21	46.57
8	1300	20	1.85	54.61	43.69
9	1300	40	2.06	56.57	45.26
10	1300	60	2.54	59.07	47.26
11	1500	20	2.07	54.82	43.85
12	1500	40	2.39	56.98	45.59
13	1500	60	3.17	60.08	48.06

2. Bulk fill light cured composite for posterior restorations *Filtek One Bulk Fill Restorative*

2.1 Reccomendations for efficient light curing:

- When light curing BC *Filtek One Bulk Fill Restorative* it is not necessary to irradiate for 60 s, as increasing the time above 40 s does not lead to a significant increase in the hardness on the top surface.
- When working with a layer thickness in the range of 2-4 mm, the light curing regimes can be selected from the *table 4.4.*
- When working with a layer thickness greater than 4 mm, the light curing regimes need to be selected from *table 4.7.*
- In *table 4.7.* shows the maximum layer thickness at which the condition for high hardness on the top surface and hardness on the bottom surface equal to 80% of that of the top surface is satisfied.

2.2. Reccomended regimes for *Filtek One Bulk Fill Restorative*:

Table 4.7.
Reccomended regimes for light curing of BC *Filtek One Bulk Fill Restorative with layer thickness bigger than 4 mm.*

№	Intensity mW/cm^2	Time s	Layer thickness mm	Hardness, HV 50	
				Top	Bottom
1	800	40	5.31	60.35	49.31
2	1000	40	6.16	61.72	49.66
3	1200	20	5.34	60.34	47.38
4	1200	40	7.50	63.66	49.73
5	1300	20	5.73	61.32	47.39
6	1500	20	6.79	63.89	47.27

Table 4.4.
Reccomended regimes for light curing of BC Filtek One Bulk Fill Restorative with layer thickness in the range of 2-4 mm.

№	Intensity mW/cm^2	Time s	Layer Thickness mm
1	I>1000	20	2
2	600-1500	40	2
3	I>1000	20	3
4	600-1500	40	3
5	I>1250	20	4
6	700-1250	40	4
7	I<700	60	4

3. Universal higly filled light cured composite

G-aenial Universal Flo

3.1. Reccomendations for efficient light curing:

- All light curing modes with intensity in the region 600-1500 mW/cm^2 and irradiation time 20-60 s satisfy the requirement for the optimal hardness on the top surface of the restoration.
- To ensure a maximum hardness of 46-48 HV on the top surface, the layer thickness should not exceed 5 mm.
- All curing regimes in the table can be used for successful polymerization of FC *G-aenial Universal Flo.*

3.2. Reccomended regimes for FC *G-aenial Universal Flo*

Reccomended regimes for *light curing of* FC *G-aenial Universal Flo.*

№	**Intensity mW/cm^2**	**Time s**	**Layer thickness mm**	**Hardness, HV 50**	
				Top	**Bottom**
1	600	20	2.42	43.89	35.11
2	600	40	2.66	45.24	36.19
3	600	60	3.23	45.81	36.65
4	700	20	2.44	44.46	35.57
5	700	40	2.71	45.64	36.51
6	700	60	3.37	46.00	36.80
7	800	20	2.46	45.02	36.02
8	800	40	2.77	46.02	36.82
9	800	60	3.54	46.15	36.92
10	1000	20	2.51	46.09	36.87
11	1000	40	2.91	46.68	37.35
12	1000	60	4.01	46.29	37.03
13	1200	20	2.57	47.06	37.65
14	1200	40	3.11	47.16	37.73
15	1200	60	4.77	46.01	36.81
16	1300	20	2.61	47.50	38.00
17	1300	40	3.24	47.29	37.84
18	1300	60	5.37	45.58	36.46
19	1500	20	2.72	48.22	38.58
20	1500	40	3.61	47.19	37.75
21	1500	60	7.63	43.22	34.58