



Evaluation of the Polymerization Depth of Bulk Fill Resin Composites Polymerized by Different Procedures: An In-Vitro Study

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ABSTRACT

Objective: The aim of this study is to investigate the depth of cure (DOC) of the bulk fill composite samples prepared in different thicknesses with two different modes of light curing device with Vickers hardness tester.

Methods: Five different bulk-fill composite materials were used in the present study. 20 sample of 2 mm and 20 sample of 4 mm thick samples were prepared, 10 samples from each group were polymerized for 20 seconds with the standard mode (Mode 1) of the light curing device and 10 samples polymerized with soft-start mode (Mode 2) for 25 seconds (n=10). Hardness measurements were made from the upper and lower surfaces of each sample and the hardness ratio was calculated. The statistical analysis was performed using three-way analysis of variance (ANOVA) and Tukey multiple comparisons ($p = 0.05$).

Results: The hardness ratios of all composites were determined to be over 80%, which is clinically acceptable. There was a statistically significant difference between the DOC levels according to the composites ($p < 0.05$). There was a statistically significant difference according to the thickness ($p < 0.05$). There was no statistically significant difference according to polymerization procedures ($p > 0.05$).

Conclusions: The surface hardness ratios of all groups were determined to be over 80%, which is clinically acceptable. The polymerization with soft start technique may not different from the standard-mode for polymerization depth.

Keywords: Composite resins, hardness, polymerization, LED

1. INTRODUCTION

One of the significant problems associated with light-polymerized resin composites is the inability to maintain cure depth and the possibility of insufficient monomer conversion (1). Resin composites polymerize only at a certain depth depending on the light penetration into the material (2). It has been shown that insufficient polymerization can lead to a decrease in physical, mechanical properties of resin composites, fragmentation of the resin composite and negative biological reactions due to residual monomer toxicity (3-5).

In order to have sufficient polymerization, sufficient light from the light curing device, the appropriate wavelength range of light, and the exposure time of the material to the light are essential (6). Other factors such as type, color, and translucency of the resin composite, the thickness of the layer, the distance to the end of the light curing unit, the post-irradiation period, the size and distribution of the filler particles affect the depth of the polymerization as well (7-9).

The composite is applied in 2 mm layers to achieve polymerization in large cavities and to reduce polymerization shrinkage (5). The layering technique has some disadvantages

including the possibility of space or contamination between the composite layers, errors in inter-layer bonding, the difficulty of placement due to limited access in small cavities, and prolongation of treatment time (10). "Bulk fill" resin composites have been developed in order to overcome these disadvantages. Bulk fill resin composites can be applied in a single layer up to 4-6 mm.

The optical properties of resin composites can affect the light transmittance, therefore the mechanical properties and monomer conversion (11). Increasing the translucency of the material is an approach to improve the polymerization depth in bulk fill composites (12). To optimize the properties of the composite, bulk fill composites include new filler technologies, shrinkage stress relievers, polymer isolator modulators and light reactive photo initiator systems (11-13). Bulk fill composites have a higher polymerization depth and less polymerization shrinkage than conventional resin composites (14).

Various techniques are used to determine the depth of polymerization. Moreover, polymerization could be examined by direct (Fourier Transform Infrared Spectroscopy-FTIR-, Raman Spectroscopy) and indirect (scraping, micro hardness) methods (15, 16). Different devices can be used to measure

hardness. Due to the ease of application and measurement accuracy in dentistry, Vickers hardness tester is frequently used to measure the hardness of materials (17). Bouschlicher et al (18) have reported that the polymerization depth of the composite can be calculated by hardness method. In this method, hardness is measured from bottom and top surfaces of composite blocks of different thicknesses. The value obtained by dividing the base hardness value of the composites by the top hardness value is called the hardness ratio. It has been reported in the literature that the composite resin has an acceptable minimum hardness ratio of 0.80 to be sufficiently polymerized (19, 20).

The aim of this study is to examine the polymerization depth of the bulk-fill composite samples prepared in different thicknesses with Vickers hardness measurement following the polymerization with two different modes of the light emitting diode (LED) light curing device. Null hypotheses of the study are as follows; 1) There is no difference between the polymerization depths of bulk fill composites of different thicknesses. 2) There is no difference in polymerization depths of bulk fill composites polymerized with standard and soft start mode.

2. METHODS

Five different nanohybrid bulk fill composite material – Ecosite (DMG, Germany), Filtek Posterior Bulk-Fill (3M, UK), Tetric N-Ceram Bulk Fill (Ivoclar Vivadent), Admira X-tra (Voco), Grandioso X-tra (Voco) – were used (Table 1). Universal color was used for each composite A2 or for the

composites which were not produced in A2 in order to standardize the effect of color difference on polymerization depth. The samples were prepared by placing the composite material in cylindrical Teflon molds of 5 mm diameter in 2 mm and 4 mm thickness. 20 pieces of 2 mm and 20 pieces of 4 mm thick samples were prepared for each composite by means of Teflon molds. Composite materials placed on the molds were compressed between two microscope plates with a transparent matrix band on the top surface to obtain a smooth surface. The power of the light curing device (Radii Plus, SDI) was controlled by a radiometer and all samples were polymerized with a light power of 1500mW/cm². SDI Radii Plus light curing device that we used in our study is the second-generation LED with a wavelength range of 440-480 nm. 10 samples from each group were polymerized for 20 sec. with the standard mode (Mode 1) of the LED light curing whereas 10 samples were polymerized for 25 sec. with the soft-start mode (Mode 2) of the LED light curing device (n=10). Soft start mode revealed a reduced intensity for the first 5 seconds before irradiation at 100% intensity. The samples were light cured via a transparent matrix band to prevent oxygen inhibition zone formation. The samples were kept in distilled water at 37 °C for 24 hours in a dark environment to complete the polymerization. Subsequently, 300 gr load from the upper and lower surface of each sample were subjected to 3 Vickers hardness measurements (Tronic, Digital Microhardness Tester DHV-1000) for 15 seconds. The upper and lower surface hardness ratios of each sample were calculated and recorded. The statistical analysis of the findings of the study was performed using the one-way analysis of variance (ANOVA) and Tukey multiple comparisons (p= 0.05).

Table 1. The materials and product details used in the study.

RBC, color	Manufacturer	Matrix	Filler	Filler content	Photo initiator	Filler size (µm)
Filtek Bulk Fill Posterior, A2	3M Espe, USA	AUDMA, AFM,DDMA, UMA	Ytterbium trifluoride (YbF ₃), zirconia filler, silica filler	76/58	Camphorquinone	0,004 – 0,01
Grandioso, A2	Voco, Germany	Bis-GMA, Bis-EMA, TEGDMA	Glass ceramic fillers, functionalised SiO ₂	89/73	Camphorquinone	-
Tetric N-Ceram Bulk Fill, A	Ivoclar Vivadent, Liechtenstein	Bis-GMA – UDMA	Barium alumino silicate glass, prepolymer filler	75/53	Lucirin, Ivocerin, camphorquinone	0,4-0,7
Admira Fusion X-tra, Universal	Voco, Germany	Organically modified silicic acid	Ba–Al–Si-glass/Silica nanoparticles	84/70	Camphorquinone	0.05–10
Ecosite, Universal	DMG, Germany	Bis-GMA	Barium glass	82/65	Camphorquinone	0,02-0.07

3. RESULTS

The following results were obtained when the effect of composite, thickness and polymerization procedure on DOC (Table 2) were considered; No statistically significant difference was found between DOC levels according to composites (p<0.05). There was a statistically significant difference between the DOC levels according to the thickness (p<0.05) whereas was no statistically significant difference

between DOC levels according to polymerization procedures (p>0.05).

When the 2 mm thickness Mode 1 was used, the mean DOC of the Tetric composite was found significantly lower than all other composites (p₁:0.019; p₂:0.004; p₃:0.001; p₄:0.049; p<0.05). There were no statistically significant differences between Ecosite, Filtek, Grandioso and Admira X-tra composites (p> 0.05) (Table 3).

Table 2. Evaluation of the effect of composite, thickness and polymerization procedure on DOC. Three way ANOVA Test, * $p < 0.05$; df: degree of freedom; F: the F-statistic

Source	Type III Sum of Squares	df	Mean Square	F	p
Composite	0,168	4	0,042	13,031	0,000*
Thickness	0,039	1	0,039	12,077	0,001*
Polymerization procedure	0,002	1	0,002	0,542	0,463

When the 2 mm thickness Mode 2 was used, the average DOC of the Tetric composite was found to be significantly lower than the Ecosite, Filtek and Grandioso composites ($p_1:0.012$; $p_2:0.001$; $p_3:0.031$). The DOC average of Admira X-tra composite was significantly lower than the Filtek composite ($p_1:0.016$) (Table 3).

When the 4 mm thickness Mode 1 was used, the DOC average of Grandioso composite was significantly higher than Ecosite and Tetric composites ($p_1:0.020$; $p_2:0.028$) (Table 3).

When the 4 mm thickness Mode 2 was used, the DOC average of the Filtek composite was significantly higher than the Tetric composite ($p_1:0.026$) (Table 3).

Table 3. Intergroup comparisons of DOC according to thickness and polymerization procedures. One way ANOVA Test; * $p < 0.05$

Thickness	Polymerization procedures	Material	DOC	p
2 mm	Mode 1	Ecosite	0,91±0,05	0,001*
		Tetric	0,83±0,05	
		Filtek	0,93±0,09	
		Grandioso	0,94±0,02	
		Admira X-tra	0,9±0,05	
	Mode 2	Ecosite	0,92±0,04	0,000*
		Tetric	0,82±0,07	
		Filtek	0,95±0,03	
		Grandioso	0,9±0,03	
		Admira X-tra	0,87±0,07	
4 mm	Mode 1	Ecosite	0,83±0,08	0,018*
		Tetric	0,83±0,06	
		Filtek	0,87±0,07	
		Grandioso	0,91±0,04	
		Admira X-tra	0,85±0,04	
	Mode 2	Ecosite	0,9±0,04	0,010*
		Tetric	0,83±0,09	
		Filtek	0,92±0,04	
		Grandioso	0,85±0,07	
		Admira X-tra	0,90±0,05	

Intragroup comparisons of DOC according to thickness and polymerization procedures were shown in Table 4.

Table 4. Intragroup comparisons of DOC according to thickness and polymerization procedures. Student t test; * $p < 0.05$.

Material	Thickness	DOC		p
		Mode 1 Mean±SD	Mode 2 Mean±SD	
Ecosite	2 mm	0,91±0,05	0,92±0,04	0,732
	4 mm	0,83±0,08	0,90±0,04	
	p	0,014*	0,268	
Tetric	2 mm	0,83±0,05	0,82±0,07	0,685
	4 mm	0,83±0,06	0,83±0,09	
	p	0,929	0,769	
Filtek	2 mm	0,93±0,09	0,95±0,03	0,416
	4 mm	0,87±0,07	0,92±0,04	
	p	0,113	0,045*	
Grandioso	2 mm	0,94±0,02	0,90±0,03	0,011*
	4 mm	0,91±0,04	0,85±0,07	
	p	0,123	0,036*	
Admira X-tra	2 mm	0,9±0,05	0,87±0,07	0,348
	4 mm	0,85±0,04	0,90±0,05	
	p	0,024*	0,318	

4. DISCUSSION

Optical microscopy and scraping methods could reveal the results of curing depth higher than the actual value when compared to hardness and conversion degree determination methods (21). However, FTIR has been reported to be less sensitive than hardness assessment to identify small changes in the degree of conversion (22). The degree of conversion of resin composites can be examined indirectly by Vickers or Knoop surface hardness measurements (23). In our study, Vickers hardness measurement was used to determine the depth of polymerization.

Studies in the literature have reported that the shape, rate, and type of filler particles significantly affect the light transmittance and the polymerization of the material (24). The filler sizes and ratios of the composites used in the study were different from each other. This could have caused the differences in polymerization depth. In our opinion, the filler ratio of Tetric N-Ceram Bulk fill composite being lower than the other composites together with the larger filler particle size may have a negative effect on the light transmittance. In the studies of Tarle (25), it has been noted that the light transmittance of Tetric Evo Ceram, being less than other bulk fill composites in the filler ratio studies was found to be lower by examining the polymerization of bulk fill composites.

Due to the differences in refractive indexes between the inorganic filler and resin matrix, they have stated that light scattering increases in the materials with large fill-matrix interface area and reduces light transmittance (25).

Bis-EMA and low-viscosity urethane-derived monomers have been reported to show generally a higher degree of conversion than typical Bis-GMA/TEGDMA resins (25-27). In our study, the samples of Bis-EMA-containing Grandioso bulk fill composite polymerized in standard mode in 4 mm thickness showed a higher cure depth than the Bis-GMA containing Ecosite and Tetric groups. Bis-EMA could have been effective for the difference.

Although camphorquinone is currently used in all resin-based composites, some bulk fill composites also contain alternative photoinitiators activated at lower wavelengths of light. These composite resins benefit from the use of a broad-spectrum light source giving blue as well as purple light (28). As the lower wavelengths of light (in the purple range) did not penetrate deep into the composite resin, it has been reported that the polymerization may be reduced due to insufficient activation of alternative photoinitiators in deeper regions (29, 30). Therefore, single use of purple light-activated photoinitiators such as Lucirin TPO is not recommended for bulk fill composites (31). In contrast to camphorquinone, having an absorption peak close to 470 nm, the absorption peak of Lucirin TPO is close to 390 nm. As a result, ultraviolet light with a wavelength ranging from 340 nm to 430 nm is required to activate photoinitiator (32). The first and second generations of the light-emitting diode (LED) light curing units used in dentistry emit blue light at a narrow wavelength between 410 and 470 nm. It has been noted that CQ cannot suitably polymerize resin materials partly replacing alternative photoinitiators (33-35). The results of our study support these findings as well. The 2 mm thickness of polymerization depth of photoinitiator TPO and Ivocerin containing Tetric N-Ceram bulk fill groups along with camphorquinone were found significantly lower than other composites. Tetric groups with 4 mm thickness presented low polymerization depth results compared to other groups. However, the difference was not found statistically significant. Some studies have highlighted that Tetric N-Ceram and Tetro Evo-Ceram containing Ivocerin, and TPO have lower surface hardness (21, 36).

All groups have either reached or exceeded 80% lower/upper surface hardness threshold which is the clinically acceptable ratio at the end of polymerization at 20 seconds standard and at 25 seconds soft-start mode. This result was also in consistency with similar studies in the literature (37, 38).

The DOC average in 2 mm thickness of Admira X-tra and Ecosite bulk fill composites in the Mode 1 procedure were found to be significantly higher than in 4 mm thickness ($p: 0,024$, $p:0,014$). DOC average in 2 mm thickness of Filtek and Grandioso bulk fill composites in Mode 2 procedure was statistically and significantly higher than in 4 mm thickness ($p:0,045$, $p:0,036$). There was no significant difference regarding DOC in 2 mm and 4 mm thickness in other composites. Marais et al. have also reported that the polymerization effect was gradually decreased at the thicknesses exceeding 2 mm in light intensity (39). Similarly, conventional and bulk fill composites have been reported to

reduce light transmittance at increased thicknesses (37). The first null hypothesis of our study has been partially rejected.

When all groups were considered, there was no significant difference observed between the polymerization procedures. There are studies in the literature reporting that the continuous and gradual light-application procedures do not make a significant difference (39, 40). The results of our study were also in consistency with the studies in the literature. It could be stated that the performance of the soft-start technique is the same with the performance of the standard light application procedure for polymerization success. The second null hypothesis of our study has been partially rejected.

The soft start technique is an improved method used for reducing polymerization shrinkage. It will also be appropriate to evaluate the polymerization shrinkage of the materials examined in our study for polymerization success.

According to the results of our study, it could be highlighted that the application of bulk-fill resin composite in 4 mm layers can save us time. Moreover, the polymerization with soft start technique is not different from the standard-mode for polymerization depth. The selection of an appropriate light curing device plays an important role for reliable and durable restoration as well as the selection of bulk-fill resin composite.

5. CONCLUSION

Based on the current results and within the limitations of the present study, it can be concluded :

- the application of bulk-fill resin composite in 4 mm have reached and exceeded 80% lower/upper surface hardness ratio threshold being the clinically acceptable ratio and
- the polymerization with soft start technique may not different from the standard-mode for polymerization depth.

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