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The Effect of Different Resin Composite Thicknesses and Irradiation Times on the Degree of Conversion of Two Bulk-Fill Resin Composites.

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ABSTRACT

To evaluate the effect of resin thickness, and irradiation time on the degree of conversion of two bulk-fill resin composites; Tetric EvoCeram, and X-tra Fil. 120 cylindrical specimens were prepared, and divided into 24 groups (n=5/group), representing the two bulk-fill resin composites, three different material thicknesses (2, 3 and 4mm) and the four irradiation times used in the study (10, 20, 40, and 60 seconds). The specimens were light-cured from the top surface only. Specimens were stored in light proof containers in complete darkness at 37°C for 24 hours. Degree of conversion test was conducted using FT-IR analysis. Data were statistically analyzed by Three-way ANOVA of Variance. Three-way ANOVA showed that different tested materials, thicknesses and irradiation times had a statistically significant effect on mean degree of conversion % at $p \leq 0.001$. The highest mean degree of conversion value was recorded for X-tra Fil 2mm thickness group light-irradiated for 60 seconds ($97 \pm 0.17\%$), while the lowest value was recorded for X-tra Fil 4mm thickness group light-irradiated for 10 seconds ($80.71 \pm 0.25\%$). A 10 seconds irradiation time was enough to reach an acceptable degree of conversion for both tested bulk-fill resin composites when placed in 4mm bulks as recommended by their manufacturers. The resin chemistry seems to positively influence the polymerization process in the two tested bulk fill resin composite materials.

Keywords: Bulk-fill, resin composite, degree of conversion, thickness, irradiation time

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INTRODUCTION

Dental resin composites are the most commonly used direct esthetic restorations worldwide. Bis-GMA represents the main monomer of most commercially available dental resin composites. However due to its high molecular weight, high viscosity, and low mobility other low-viscosity monomers, as TEGDMA and UDMA, were added in order to increase the cross-linking of the resultant polymer. Photo-polymerization of resin composites exhibits incomplete conversion of double bonds, leaving a significant proportion of residual methacrylate groups which are unreacted [1].

The degree of conversion (DC) of resin composites refers to the conversion of carbon-carbon double bonds monomer into carbon-carbon single bonds polymer which directly affects the mechanical properties of the resin composite and influence their performance and success clinically [2,3].

One of the main disadvantages of the light cured resin composite is their limited depth of cure. Accordingly, in deep cavities the attenuation of the light intensity reaching to the full depth of the resin composite material is an influencing factor during the restorative procedure. This will reduce the degree of conversion leading to a weak composite restoration with inferior physical and mechanical properties [4].

Consequently, the incremental build-up of multiple thin layers (2mm increment thickness) of dental composite is highly required; first because of the limited cure depth of the dental composite and second to potentially reduce the consequences of polymerization shrinkage stresses. However, layering techniques and multiple curing regimens of resin composites are time consuming and require increased effort for the dentist [4].

Recently, a new category of resin composite, so-called bulk-fill resin composites was introduced. The new materials are stated to be placed in 4mm or even 5mm thick bulks instead of the current incremental placement technique, without negatively affecting polymerization shrinkage or the DC of the resin composite [5].

As a result, the aim of the current study was to evaluate two methacrylate-based, packable bulk-fill dental composite resins which their manufacturers claim to have a depth of cure of 4mm when light-irradiated for only 10 seconds. The potential of the bulk-fill dental composite resins was determined in terms of the degree of conversion at different composite thicknesses and different irradiation times.

MATERIALS AND METHODS

Two bulk-fill resin composites were tested in this study. The materials manufacturer, composition and batch number are listed in Table 1.

Table 1: Materials manufacturers, composition and batch number			
Material	Composition	Manufacturer	Batch number
Tetric EvoCeram bulk-fill [TEBF] Nano-hybrid Bulk-fill resin composite	Bis-GMA, Bis-EMA, UDMA, Ba–Al–Si–glass, prepolymer filler (monomer, glass filler and ytterbium fluoride), spherical mixed oxide. Filler 79–81 wt.% (including 17% prepolymers) / 60–61 vol.%	Ivoclar Vivadent, Schaan, Liechtenstein	P72398
X-tra fil [XFBF] Hybrid bulk-fill resin composite	Bis-GMA, UDMA, TEGDMA, Inorganic fillers: SiO ₂ , glass, oxide (86 wt.% / 70.1 vol.%).	Voco GmbH, Cuxhaven, Germany	1740
Bis-GMA: Bis-Phenol-A glycidyl-methacrylate. Bis-EMA: Ethoxylated bisphenol A dimethacrylate. UDMA: Urethane dimethacrylate. TEGDMA: Triethylene glycol dimethacrylat.			

Study design and specimen grouping

One hundred and twenty cylindrical specimens were prepared and equally divided into 24 groups (n=5/group) according to the three levels of the study: Level 1: Bulk-fill resin composite (Tetric EvoCeram bulk-fill composite [TEBF] and X-tra fil bulk-fill composite [XFBF]). Level 2: Composite thickness (2, 3 and 4mm) and Level 3: Irradiation time (10, 20, 40 and 60 seconds (s)).

Specimen preparation

Sectional Teflon moulds of 6mm diameter and different thicknesses (2, 3 and 4mm) were used to prepare the specimens. The molds were first mounted on the top of a microscope slide and a Mylar strip, and then the mold was filled in bulk with one of the two bulk-fill resin composites. The top side of the mold was covered with a second Mylar strip to prevent the formation of the oxygen inhibited layer at the resin composite top surface. A glass slide with a load of 1 kg was applied for 30s to ensure consistent packing of the specimens, after which the load and the glass slide were removed [5].

The specimens were light-cured from the top surface only using LED Elipar S10 light curing unit (Elipar S10, 3M ESPE; USA) with an output of ≥ 1000 mW/cm² for 10, 20, 40 or 60s respectively. The light curing tip was kept centered and in direct contact with the second Mylar strip. The power density of light curing unit was assessed using a radiometer [Curing Radiometer, Demetron, Danbury, CT, USA]. After light-curing, the cylindrical specimens were pushed out of the mold and the excess resin composite material was removed using a plastic spatula [6].

The specimens were stored in light-proof containers before the FT-IR analysis was conducted, in complete darkness at 37°C for 24 hours to prevent ambient light from causing additional post light-curing polymerization [7].

Degree of Conversion (DC) testing

FT-IR spectra of the uncured and cured bulk-fill resin composite materials were obtained using 24 scans at 4cm⁻¹ in the absorbance mode (Jasco FT-IR 6400, Japan). The FT-IR spectroscopy was completed using a potassium bromide pellet technique.

An additional 10 uncured specimens (n=5/group) were prepared for FT-IR testing to be used as control specimens for the two bulk-fill resin composites used in the study.

For the control specimens; 2mg of each of the uncured bulk-fill resin composites were blended with 7mg of Ispectropic grade (IR) potassium bromide powder in a specimen holder; then it was pressed into a transparent disc (1mm thickness) under heavy pressure for one minute, using a pellet maker kit (KBr Product-A-Press, International Crystal Labs, Garfield, NJ, USA), before it was subjected to FT-IR analysis [7,8].

Each prepared light cured bulk-fill resin composite specimen was completely crushed and ground into a fine powder using a mortar and a pestle. Subsequently, 2mg of the each of the resin composite powder were blended with 7mg of Ispectropic grade (IR) potassium bromide in a specimen holder, and then it was pressed into a transparent disc using a pellet maker kit [7].

The specimen holder was transferred to the spectrometer and a spectrum was obtained using the same parameters as for the uncured resin composites specimens. FTIR spectra of the light cured resin composites specimens were obtained after 24 hours in dark storage at 37° C [7].

For calculating the DC %, the percentage of unreacted carbon-carbon double bonds (% C=C) was determined from the ratio of absorbance peak areas of aliphatic carbon-carbon double bonds C=C (peak at 1638 cm⁻¹) against aromatic component (peak at 1608 cm⁻¹) which was used as an internal standard before and after curing. The underlying peak area was calculated for each peak, using a standard baseline technique with the aid of a computer software program provided with the spectrometer (Spectra Manager Version 2).

The degree of monomer conversion was determined using the following equation:

$$DC \% = 1 - \left[\frac{(1638 \text{ cm}^{-1} / 1608 \text{ cm}^{-1}) \text{ peak area after curing}}{(1638 \text{ cm}^{-1} / 1608 \text{ cm}^{-1}) \text{ peak area before curing}} \right] \times 100$$

Statistical analysis

Data were presented as mean and standard deviation (SD) values. Three-way ANOVA was used to study the effect of different tested restorative materials, thickness and irradiation time on mean DC% values.

Tukey’s post-hoc test was used for pair-wise comparison between the means when ANOVA test is significant. Independent t-test has been used to compare between different tested resin composite materials.

One way-ANOVA was used to study the effect of thickness and irradiation time on mean DC followed by Tukey’s post-hoc test was used for pair-wise comparison between the mean values when ANOVA test is significant.

Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 22 for Windows.

RESULTS

Three-way ANOVA showed that the resin composite material, resin composite thickness and irradiation time, each independent variable had a statistical significant effect on DC%. The interaction between the three independent variables (resin composite material, thickness and irradiation time) had also a statistical significant effect on DC% at $p \leq 0.001$.

For the effect of the resin composite thickness on DC%, regardless the composite material and irradiation time, One-way ANOVA (Table 2) showed that there was a statistical significant difference between the three composite thicknesses groups (2, 3 and 4mm). The 2mm composite thickness group showed the highest mean DC% value ($91.96 \pm 3.83\%$) while the lowest mean DC% value was recorded for 4mm composite thickness group ($87.45 \pm 5.77\%$) at $P \leq 0.001$.

Table 2: Mean and standard deviation (SD) for the DC% for different composite thicknesses regardless of other variables.				
	Resin composite thickness			P-value
	2mm	3mm	4mm	
DC%	91.96 (3.83) ^a	89.84 (4.18) ^b	87.45 (5.77) ^c	$\leq 0.001^*$
Means with the same letter within each row are not significantly different at $p=0.05$. *= Significant, NS=Non-Significant				

For the effect of different irradiation times on DC%, regardless the bulk-fill composite material and composite thickness, One-way ANOVA followed by Post-hoc test (Table 3) showed that there was a significant difference between 60s irradiation time group which recorded the highest mean DC% value ($96.24 \pm 0.93\%$) and the other three irradiation time groups (10s, 20s and 40s) with the lowest mean DC% value was recorded for the 10s group ($86.75 \pm 3.05\%$) at $P \leq 0.001$.

Table 3: Mean and standard deviation (SD) for the DC% for different irradiation times regardless of other variables.					
	Irradiation time				P-value
	10s	20s	40s	60s	
DC%	86.75 (3.05) ^a	87.20 (2.68) ^a	88.82 (4.87) ^a	96.24 (0.93) ^b	$\leq 0.001^*$
Means with the same letter within each row are not significantly different at $p=0.05$. *= Significant, NS=Non-Significant					

The effect of the two different bulk-fill resin composite materials, the three resin composite thicknesses and the four irradiation times on DC% is shown in Table 4. Three-way ANOVA followed by Post-hoc test revealed that there was a statistical significant difference between the different groups of the three tested

individual variables. The highest mean DC% value was recorded for XFBF at 2mm thickness group light-irradiated for 60s (97±0.17%), while the lowest mean DC% value was recorded for XFBF at 4mm thickness group light-irradiated for 10s (80.71±0.25%). For both bulk-fill resin composite materials, there was a decrease in the DC% with the increase in the resin composite thickness at different irradiation times. Furthermore, there was an increase in the DC% with the increase in the irradiation time at the different resin composite thicknesses at $P \leq 0.001$.

Table 4: Means and standard deviations (SD) for the effect of different materials, different thicknesses and different irradiation times on DC%.						
		10s	20s	40s	60s	P-value
TEBF	2mm	89.40 (0.23) ^{Aa}	89.52 (0.29) ^{Aa}	92.85 (1.39) ^{Ab}	95.74 (0.17) ^{Ac}	0.001*
	3mm	89.26 (0.46) ^{Aa}	88.24 (0.20) ^{Ba}	88.52 (1.03) ^{Ba}	95.65 (0.57) ^{Ab}	≤0.001*
	4mm	87.90 (0.23) ^{Aa}	87.90 (0.25) ^{Aa}	87.99 (0.18) ^{Aa}	95.54 (0.75) ^{Bb}	≤0.001*
XFBF	2mm	86.56 (0.47) ^{Aa}	89.00 (0.45) ^{Ab}	95.54 (1.18) ^{Ac}	97.26 (0.17) ^{Ac}	≤0.001*
	3mm	86.68 (0.41) ^{Ab}	86.76 (0.37) ^{Ba}	86.38 (1.59) ^{Ba}	97.09 (0.08) ^{Aa}	≤0.001*
	4mm	80.71 (0.25) ^{Ab}	81.80 (0.15) ^{Bba}	81.64 (0.47) ^{Ab}	96.15 (0.25) ^{Ac}	≤0.001*

Means with the same lowercase letter within each row are not significantly different at $p=0.05$.
 Means with the same uppercase letter within each column are not significantly different at $p=0.05$.
 *= Significant, NS=Non-Significant

DISCUSSION

The introduction of bulk-fill resin composites has opened new avenues in the field of restorative dentistry. The producers' guarantee of placing the bulk-fill resin composites in 4mm or even 5mm bulks and light cured for 10s or 20s, according to the manufacturer's recommendations, with minimal loss in DC and mechanical properties seems to be of great value [9]

The DC is known to devolve up to about 24 hours after light curing of the resin composite [10-11]. Furthermore, the increase of temperature during resin composite polymerization can significantly affect the polymerization efficiency, and the rise of the temperature from room temperature (22 °C) to mouth temperature (35 °C) has been shown to increase the overall DC due to decreased monomer viscosity and increased mobility of the monomer [12]. Regarding such parameters that greatly affect the final DC, in the present study, all measurements were conducted after 24 hours in dark storage at 37 °C.

There are many factors that greatly affect the DC. Some are intrinsic such as; co-monomer composition and ratio, filler content and size and others are extrinsic factors such as; light spectrum, irradiance, and irradiation time [11].

The results of the present study revealed a wide variety of different influences on the final overall DC of the tested bulk-fill resin composites. The composite material was the major parameter which had the strongest influence on DC, followed by composite thickness and the irradiation time. Significant interaction effects between the irradiation time and the tested material as well as between the composite thickness and the tested material were also found in the present study. This indicates that the DC for different irradiation times and at different composite thicknesses was different for both tested materials. These findings were in agreement with Tarle et al [11].

A higher DC% was recorded for XFBF compared to TEBF at all specimens' thicknesses (2, 3 and 4mm) cured at the four tested irradiation times (10, 20, 40 and 60s). Since the polymerization conditions were kept standardized in this study, differences in the DC% of the materials can be attributed to variations in the chemistry of their resin matrix. Regarding the chemical composition of both resin bulk-fill composites [13,14], it can be stated that XFBF contains low viscosity monomer TEGDMA and UDMA, which might have increased the flow and decreased the viscosity of the resin matrix and thus increasing DC%, this is because monomers such as UDMA, TEGDMA, are more flexible and flowable monomers than Bis-GMA and Bis-EMA [11,15-17].

Some studies [15,17] reported that; when Bis-GMA is diluted with the low viscosity TEGDMA monomer, a synergistic effect on the rate of polymerization, network plasticization, and DC% has been observed.

Moreover; Czasch & Ilie [9] revealed that; the presence of low viscosity monomers such as TEGDMA might increase the final overall DC. It was reported that; DC% increases in the order: Bis-GMA<Bis-EMA<UDMA<TEGDMA [9,15,17].

Regardless the composite material and the irradiation time; the composite thickness had a statistical significant effect on DC%. The 4mm resin composite thickness group had the lowest DC% value. This could be due to the increase in the increment thickness would have increased the organic resin matrix portion which will increase the viscosity of the resin composite paste, lowering the monomer mobility and consequently lowering the DC% in the bulk increments thicker than 2mm [20-23].

These results come in accordance with some studies [24-26] that showed significantly less depth of cure of some bulk-fill materials than that claimed by manufacturers.

Regardless the composite material and the composite thickness; the irradiation time had a statistical significant effect on DC%. There was an increase in the DC with the increase in the irradiation time. This might be due to the increase in the radiant exposure is calculated as the product of irradiance and time of irradiation provided by the light curing unit. Consequently such increase in the radiant exposure could be due to the high irradiance generation from the LED curing unit used in the present study ($\geq 1000 \text{ mW/cm}^2$) associated with the maximum absorption wavelength of the camphoroquinone photo-initiator present in both resin composites used in the study [27-29].

Moreover; the increase in the DC with the increase in the irradiation time could be owed to the translucency of both resin bulk-fill composites used in the study, due to high light scattering throughout the translucent resin, as a higher transmittance of the light results in a higher DC, that is greatly affected by the resin translucency and filler contents [30,31].

These results were in agreement with Tarle et al [11], Albino et al [27] and Yap [32], they revealed that an increase in the DC% was found with the increase in the irradiation time in deeper composite layers.

The current study showed that; the interaction of investigated independent variables (composite material, composite thickness and irradiation time) had a statistical significant effect DC%. This is might be attributed to the explanation that the final DC% depends mainly on intrinsic factors such as the chemical structure of the dimethacrylate monomer, inorganic filler loading and photoinitiator concentration and extrinsic factors such as polymerization conditions [18,33].

Therefore, it can be reasonable to expect that bulk-fill composites with a degree of conversion above 80% may indicate satisfactory clinical performance. The current data indicate that the two investigated bulk-fill resin composites achieved at least 80% of their maximum DC% value at the manufacturers' claimed maximum incremental thickness of 4mm and an irradiation time of 10s.

CONCLUSIONS

Within the limitation of this study the following can be concluded:

- An irradiation time of 10s was enough to reach an acceptable degree of conversion for both tested bulk-fill resin composites when placed in 4mm bulks as recommended by their manufacturers.
- The resin chemistry seems to positively influence the polymerization process in the two tested bulk-fill resin composite materials.

REFERENCES

- [1] Miranda D, Pini P, Bovi A, Baggio A, Leite L. JSM Dent 2013; 1: 1013-20.
- [2] Prasanna n, Pallavi R, Kavitha s, Lakshmi N. Ind J Dent Res 2007; 18: 173-6.
- [3] Krämer N, Lohbauer U, García-Godoy F, Frankenberger R. Am J Dent 2008; 21: 135-42.
- [4] Leprince J, Palin W, Vanacker L, Sabbagh J, Devaux J, Leloup G. Dent Mater 2014; 42: 993-00.
- [5] Fleming G, Awan M, Cooper P, Sloan A. Dent Mater 2008; 24: 522-9.

- [6] Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Dent Mater 2012; 28: 521-8.
- [7] Taher N. Pak Oral Dent J 2011; 31: 457-63.
- [8] Yoshida K, Greener E. Dent Mater. 1993; 9: 246-51.
- [9] Czasch P, Ilie N. Clin Oral Invest 2013; 17: 227-35.
- [10] Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Biebuyck J, Mestdagh M, Larbanois P, Leloup G. Dent Mater 2006; 22: 405-12
- [11] Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, Tauböck T. Clin Oral Invest 2014; 18: 9-16.
- [12] Price R, Whalen J, Price T, Felix C, Fahey J. Dent Mater 2011; 27: 983-9
- [13] X-tra Fill bulk fill resin composite. Material Safety Data Sheet. Voco GmbH, Cuxhaven, Germany.
- [14] Tetric EvoCeram bulk fill resin composite. Scientific Documentation. Ivoclar Vivadent, Research & Development Scientific Service. Schaan, Liechtenstein.
- [15] Sideridou I, Tserki V, Papanastasiou G. Biomat 2002; 23: 1819-29.
- [16] Buonocore M. J Dent Res 1955; 34: 849-53.
- [17] Dickens S, Stansbury J, Choi K, Floyd C. Macromol 2003; 36: 643-53.
- [18] Alshali R, Silikas N, Satterthwaite J. Dent Mater 2013; 29: e213-7.
- [19] Ilie N, Stark K. J Dent 2014;42: 977- 85.
- [20] Marovic D, Panduric V, Tarle Z, Ristic M, Sariri K, Demoli N, Klaric E, Jankovic B, Prskalo K. J Mol Struct 2013; 1044: 299-302.
- [21] Ferracane J, Berge H, Condon J. J Biomed Mater Res 1998; 42: 465-72.
- [22] Nunes T, Pereira S, Kalachandra S. J Mater Sci Mater Med 2008; 19: 1881-9.
- [23] Hadis M, Leprince J, Shortall A, Devaux J, Leloup G, Palin W. J Dent 2011; 39: 549-57.
- [24] Garcia D, Yaman P, Dennison J, Neiva G. Oper Dent 2014; 39: 441-8.
- [25] Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Dent Mater 2012; 28: 521-8.
- [26] Alshali R, Salim N, Satterthwaite J, Silikas N. J Dent 2015; 43: 209-18.
- [27] Albino L, Rodrigues J, Kawano Y, Cassoni A. Braz Oral Res 2011; 25: 267-73.
- [28] Silva E, Poskus L, Guimarães J. Oper Dent 2008; 33: 287-93.
- [29] Krämer N, Lohbauer U, García-Godoy F, Frankenberger R. Am J Dent 2008; 21: 135-42.
- [30] Rueggeberg F, Ergle J, Mettenburg D. J Esthet Dent 2000; 12: 340-9.
- [31] Leloup G, Holvoet P, Bebelman S, Devaux J. J Oral Rehabil 2002; 29: 510-5.
- [32] Yap A. Oper Dent 2000; 25:113-20.
- [33] Leprince J, Palin W, Hadis M, Devaux J, Leloup G. Dent Mater 2013; 29: 139-56.