# CURING DEPTH OF BULK- FILL COMPOSITES- AN IN- VITRO STUDY

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### ABSTRACT

The aim of this in-Vitro study to evaluate the depth of cure of Tetric EvoCeram® Bulk Fill Composite and Tetric N-Ceram® Bulk Fill Composite nano-hybrid low-shrinkage resin based composite materials using the Vickers hardness test. Specimens were fabricated in 4 mm bulk depth and 10 mm width and each sample was light cured using blue phase- G2 light cure device, then finished and polished using Sof-Lex<sup>TM</sup> disks. Specimens were immersed in ultrasonic bath with distilled water for two minutes and kept inside an oven at 37°C for 24 hours. Thereafter, the top and bottom surfaces of each specimen were subjected to the Vickers hardness testing machine. The hardness was measured in three different indentations in the top and bottom surfaces for each sample. Data were collected and analyzed using independent T-test. The bottom-to-top ratio of the surface-hardness for both materials was over 80% which indicates that the bottom surfaces were adequately cured. This study showed that adequate curing of the samples was possible up to a depth of 4mm with no significant differences between both types of nano-hybrid low-shrinkage bulk fill resin based composite materials.

Key Words: Bulk Fill, Nano-hybrid, Low-shrinkage, Depth of cure, Hardness, Vickers hardness test.

#### **INTRODUCTION**

Composite resins were first introduced in the mid-1960s for posterior dental restorations.<sup>1</sup> Since then, the use of composite resins continues to flourish as the materials meet the increasing demand for esthetics.<sup>2</sup> The massive increase in the use of these materials has lead the manufacturers seeking further improvement and development of new sophisticated systems. As a result, the compositions and techniques have improved resulting in enhanced longevity and strength of the composite restorations. However, with all these improvements, dentists still face problems while placing posterior restorations that are time and technique sensitive, necessitating the development of a procedure that must be quickly and accurately performed. It has been continuously reported that placing and curing resin composites in increments was required to achieve successful posterior restorations.3 However, if not performed properly, placing multiple layers may result in polymerization shrinkage and marginal leakage.<sup>4</sup>

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Moreover, inadequate curing depth can affect both chemical and physical properties of resin composite.<sup>5,6</sup> In order to minimize the undesired effects, composite resin should be cured to a high degree and appropriate depth.<sup>7</sup> Such challenges have led to numerous studies, some of which suggest reducing the number of composite layers or utilizing a bulk fill materials.

The bulk fill type of composite materials are placed in up to 4-mm thick increments and can be adequately cured up to that depth. The composition of these materials has been altered in various ways to allow for increased depth of curing while having less shrinkage and shrinkage stress than previous generations of composite materials. In 2008, Polydorou et al published an in-vitro study to evaluate the depth of cure in two translucent composites; the study showed that adequate curing of the samples was possible up to a depth of 3.5 to 5.5 mm.<sup>5</sup>

Recent studies support that the bulk fill materials result in significant reduction of polymerization shrinkage and, satisfactory bond strength in comparison to the conventional types of composite resins.<sup>8</sup> Thus, problems related to polymerization shrinkage such as gap formation, secondary caries, pulp irritation, post-operative sensitivity during chewing, or cusp deflection could be minimized.<sup>9</sup> Placing a self-adapting material as bulk saves time as well as improves material handling. The progress of bulk-fill composites and new improvements in resin and photo-polymerization technology enables dentists to be productive without sacrificing quality.

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There are a number of in-vitro tests for establishing depth of cure. One of the mostly used is Vickers hardness test.<sup>10</sup> Hardness is often expressed in percentage; the surface hardness is always compared to 100% which represents the maximum surface hardness. According to research carried out by Watts in University of Manchester; an acceptable curing depth is achieved, if bottom hardness corresponds to at least 80% of the surface hardness.<sup>11</sup> Experience has shown that the simple hardness measures (top and bottom) correspond well to the more thorough hardness profile measurements.<sup>12</sup>

The aim of this study was to evaluate cure depth of two nano-hybrid low-shrinkage bulk-fill resin based composite materials using the Vickers hardness test 24 hours post-cure. The null hypothesis was: There is no significant difference between the two materials (Tetric EvoCeram® Bulk-Fill Composite and Tetric N-Ceram® Bulk-Fill Composite) in the curing depth at a standardized distance.

# METHODOLOGY

Approval for the present project was obtained from the Collage of Dentistry Research Center in King Saud University. Forty disk shaped specimens of Tetric EvoCeram® bulk-fill Composite (Ivoclar Vivadent, Schaan, Liechtenstein), and Tetric N-Ceram® bulk-fill Composite (Ivoclar Vivadent, Schaan, Liechtenstein) were fabricated (20 disks of each material) in universal A shade IVA (Table 1). IVA shade was selected to minimize the effects of colorants on light polymerization.<sup>13</sup>

Using a special custom Teflon mold (10 mm in diameter and 4 mm depth) the materials were condensed in 4 mm bulk in the mold over the glass slab (Fig 1). After the materials were inserted into the mold, a transparent plastic matrix strip was placed over the material and a glass plate with 1.00 mm thickness was secured over it to flatten the surface. Each sample was light cured for twenty seconds using blue phase-G2 light cure device (Ivoclar Vivadent, Schaan, Liechtenstein) following manufacturer instructions within the range of 1200 to 1400 Mw/cm2. The tip of the curing device was kept in direct contact to the glass plate to maintain standardized distance from the tip of the device to the top surface of the specimen (Fig 2).

All specimens were finished and polished using Sof-Lex<sup>™</sup> disks (3M ESPE, St. Paul, MN, USA) gradually moving from the coarse to superfine disk with constant speed for the same duration. The specimens' thickness was verified by a micrometer (Ultra-cal MarkIII, Fowler Tools and Instruments, Sylvac, Newton, MA, USA) (Fig 3) to ensure uniform 4 mm thickness of all samples.

After polishing, specimens were immersed in ultrasonic bath with distilled water for two minutes to remove any remaining debris. All specimens were kept

inside an oven at 37°C for 24 hours in a light-proof container. After that, the top and bottom surfaces of each specimen were subjected to the Vickers hardness testing machine (Micromet 2100, Buehler, Lake Bluff, IL, USA) (Fig 5). The specimens were placed on a platform and a square diamond pyramid indenter was utilized to apply load of 300g to the surface for a 15 seconds dwell time. Three different squared pyramid indentations were done on each surface (Fig 6). Both the vertical and horizontal diameters of the pyramid were obtained from the machine. The corresponding Vickers Hardness Number (VHN) values were taken from specially designed tables supplied with Vickers Hardness machine. The mean of the vertical and horizontal VHN readings was calculated to have one reading per indentation. The mean of the sum of indentations per surface was calculated to have one representative reading for both the bottom and top surface hardness. The values measured at the top were considered as 100% and the values measured at 4mm distance were expressed as percentage of the value and were obtained from the following equation:%VHN=bottom VHN/TOP VHN × 100. Data were entered using the SPSS Program version 16.0 and, analyzed using Independent T-Test.

# RESULTS

Table 2 shows the top and bottom surfaces means and standard deviations of the Vickers hardness numbers (VHN) for all specimens. Tetric EvoCeram Composite material gave slightly higher results. However Independent T- Test showed p-values >0.05 for the top to top, bottom to bottom and the overall bottom to top ratio for the surfaces hardness (Table 3); indicating an insignificant difference between the two materials (Fig 4).

VHN mean values were 103.42 for the top surfaces and 88.28 for the bottom of the Tetric EvoCeram Composite specimens. The bottom-to-top surfaces hardness ratio equals to 85.4%. This meant that the top surfaces were harder in average by about 14.6% than the bottom surface. For Tetric N-Ceram, VHN mean values were 99.69 and 82.94 for the top and bottom surfaces respectively. The bottom-to-top surfaces hardness ratio equals 83.61%; which meant the top surfaces were harder in average by about 16.39%.

# DISCUSSION

The present study investigated the surface hardness and curing depth of two recently introduced bulk-fill resin based composite materials (Table 1). There are several methods available for testing the depth of cure. Infrared spectroscopy and laser are considered direct methods while micro- hardness, scraping and visual inspection are some of the indirect methods.<sup>6</sup>



Fig 1: The equipment and materials



Fig 2: The specimen was ready to be light cured







Fig 4: Comparison between Tetric EvoCeram and Tetric N-Ceram bulk-fill Composites



Fig 5: The Vickers Hardness Machine



Fig 6: The appearance of the indentation under the microscope

Hardness is defined as the resistance of a material to indentation or penetration, and this property is highly related to a material's strength and is used to evaluate the wear resistance and determine to which degree a material will deform under load. Surface hardness is generally accepted as an important property and valuable parameter.<sup>14</sup> Ferracane<sup>15</sup> demonstrated good

<b>Restorative materials</b>	Tetric EvoCeram	Tetric N-Ceram
Manufacturer	Ivoclar Vivadent, Schaan, Liechtenstein	Ivoclar Vivadent , Schaan, Liechtenstein
Composition	Matrix:Bis-GMA, UDMA Filler: Barium glass, YbF3, oxides, and prepolymers	Matrix: dimethacrylates Filler: Bar- ium glass, YbF3, and mixed oxides
Filler content	80% by wt. and $60%$ by vol.	75-77% by wt., $53-55%$ by vol.
Туре	Nano-hybrid RBC	Nano-hybrid RBC
Shade	IVA	IVA

# TABLE 1: TETRIC EVO-CERAM AND TETRIC N-CERAM BULK-FILL COMPOSITE

\*Bis-GMA: bisphenol A-glycidyl methacrylate, \*UDMA: urethane dimethacrylate, \*YbF3: ytterbium trifluoride, \*wt: weight, \*vol: volume, \*RBC: resin based composite

TABLE 2: COMPARISON BETWEEN MEANS AND STANDARD DEVIATIONS OF TETRIC EVOCERAM AND TETRIC N-CERAM BULK-FILL COMPOSITES

TypeofComposite	Surface	Mean	Std. Deviation	Std. Error Mean
Tetric EvoCeram	Тор	103.4245	7.32332	1.63754
	Bottom	88.2770	12.22084	2.73266
	Bottom-to-top ratio	85.3960	10.77669	2.40974
Tetric N-Ceram	Тор	99.6915	9.21192	2.05985
	Bottom	82.9390	12.94422	2.89442
	Bottom-to-top ratio	83.6090	13.24721	2.96217

### TABLE 3: COMPARISON BETWEEN TETRIC EVOCREAM AND TETRIC N-CREAM COMPOSITES USING INDEPENDENT T-TEST AND LEVENE'S TEST

Test	Surface	<b>P-Value</b>
Independent	Тор	0.1645
T-Test for Equality	Bottom	0.188
of Means	Bottom :Top Ratio	0.6425
Levene's Test for	Тор	0.603
Equality of Vari-	Bottom	0.781
ances	Bottom :Top Ratio	0.413

correlation between increasing hardness and increasing degree of conversion. Bouschlicher et al<sup>16</sup> concluded that the bottom-to-top surface micro-hardness ratios of a composite resin proved to be an accurate reflection of bottom-to-top degree of conversion. The depth of cure and degree of conversion throughout the bulk of the resin composite materials is very important for the dentists to achieve the success and best clinical results.<sup>17,18</sup> Multiple parameters can play role in the effectiveness of curing depth; some are related directly to the material itself such as its thickness or the composition as the filler particle type, size, and quantity; while others are related to the light source such as the intensity and time of the light cure device.

In this study, the results demonstrated that 4mm increment can obtain (under in vitro conditions) a high

degree of conversion. These findings were in contrary to the reports that the composite resin materials were not well polymerized if placed in more than 2mm increments, the reason for using incremental technique to ensure full polymerization. However, new materials and technologies are being developed creating a great interest among researchers for further improvements.

Enamel and Dentine Vickers hardness values have been stated as 348 VHN and 80 VHN respectively.<sup>19</sup> To assure an optimal clinical performance of the restorations, it is important to use materials with hardness at least similar to that of dentine not only superficially but also in deep layers.<sup>20</sup> Tetric EvoCeram exhibited 103.42 and 88.82 mean VHN values for the top and bottom surfaces respectively. The top surface mean VHN value for Tetric N-Ceram was equal to 99.69 while its bottom VHN mean value was 82.94. Therefore, both materials exhibited VHN values more than that of dentine even at the 4mm depth; so these materials are expected to perform well clinically. The total Hardness is usually expressed as a percentage of the surface hardness. To define the depth of cure based on top and bottom hardness measurements, it is common to calculate the ratio of bottom-to-top hardness. To consider the bottom surface as adequately cured, values of 80% and 85% have often been used.<sup>21</sup>

The amount of light to which the composite resin materials are exposed affects the degree of polymerization.<sup>22</sup> Ideally, the bottom-to-top surface hardness scores have to be very close or equal to each other indicating that the degree of polymerization of the material remained the same throughout its depth. But intensity of the light is gradually reduced because of light scattering as it passes through the depth of the composites, therefore the effectiveness of the light in curing the bottom surface is reduced.<sup>23</sup>

It is generally accepted that an adequate depth of cure has been achieved if the bottom hardness correspond to at least 80% of the surface hardness.<sup>24</sup> The bottom-to-top surface ratio of VHN at 4 mm distance was equal to 85.4% and 83.61% for Tetric EvoCeram and Tetric N-Ceram bulk fill composites respectively. Both materials exhibit hardness percentages that were higher than the generally accepted 80% level. The difference between two materials was less than 2% (p >.05); thereby the null hypothesis was accepted. Several previous studies have shown similar results for bottom-to-top surface hardness ratio of Tetric EvoCeram bulk fill composite.<sup>25</sup> However, no published data is available regarding the surface hardness of Tetric N-Ceram bulk fill composite at 4 mm depth.

The possible reasons behind the 4mm increments to be cured as a bulk in Tetric EvoCeram and Tetric N-Ceram bulk-fill Composites could be advanced composite-filler technology, a pre-polymer shrinkage stress reliever, a light initiator/polymerization booster (Ivocerin®) and a light sensitivity filter.

#### CONCLUSION

- Adequate curing was achieved for both the tested materials; Tetric EvoCeram bulk fill composite and Tetric N-Ceram bulk fill composite at 4 mm depth.
- There was no significant difference between the two materials (Tetric EvoCeram bulk fill composite and Tetric N-Ceram bulk fill composite) in the curing depth from a standardized distance.

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#### **Conflict of Interest Statement**

The authors have no commercial interest in the materials tested in this study.

#### REFERENCES

- 1 Bowen RL. US Patent 3,006,112 No. 1962.
- 2 Jordan RE, Suzuki M. Posterior composite restorations where and how they work best. J Can Dent Assoc 1991; 122(11): 30-37.
- 3 Sarrett DC. Clinical challenges and the relevance of materials testing for posterior composite restorations. Dent Mater 2005; 21(1): 9-20.
- 4 Cheung GS. Reducing marginal leakage of posterior composite resin restorations: a review of clinical techniques. J Prosthet Dent 1990; 63(3): 286-88.

- 5 Polydorou O, Manolakis A, Hellwing E, Hahn P. Evaluation of the curing depth of two translucent composite materials using halogen and two LED curing units. Clin Oral Investig 2008; 12(1): 45-51.
- 6 Aguiar FH, Bracerio A, Lima DA, Ambrosano GM, Lovadino JR. Effect of light curing modes and light curing time on the microhardness of a hybrid composite resin. J Contemp Dent Pract 2007; 8(6): 1-8.
- 7 Koupis NS, Vercruysse CW, Marks LA, Martens LC, Verbeeck RM. Curing depth of polyacid-modified composite resins determined by scraping and penetrometer. Dent Mater 2004; 20(10): 908-14.
- 8 Van Ende A, De Munck J, Van Landuyt KL, Poitevin A, Peumans M, Van Meerbeek B. Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity-bottom dentin. Dent Mater 2013; 29(3): 269-77.
- 9 Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Invest 2013; 17(1): 227-35.
- 10 De Wald JP, Ferracane JL. A Comparison of four models of evaluating depth of cure of light-activated composites. J Dent Res. 1987; 66(3): 727-30.
- 11 Watts D, Amer O, Combe E. Characteristics of visible light activated composite systems. Br Dent J 1984; 209-15.
- 12 Pilo R, Cardash HS. Post irradiation Polymerization of different anterior and posterior visible light-activated resin composites. Dent Mater 1992; 8: 299-304.
- 13 Yap AU, Soh MS, Siow KS. Effectiveness of composite cure with pulse activation and soft-start polymerization. Oper Dent 2002; 27: 44-49.
- 14 Yap AU. Effectiveness of polymerization in composite restoratives claiming bulk placement: Impact of cavity depth and exposure time. Oper Dent 2000; 25: 113-20.
- 15 Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1985; 1: 11-4.
- 16 Bouschlicher MR, Rueggeberg FA, Wilson BM. Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions. Oper Dent 2004; 29: 698-704.
- 17 Yap AU, Soh MS. Curing efficacy of a new generation high-power LED lamp. Oper Dent 2005; 30: 758-63.
- 18 Koupis NS, Martens LC, Verbeeck RM. Relative curing degree of polyacid-modified and conventional resin composites determined by surface Knoop hardness. Dent Mater 2006; 22(11): 1045-50.
- 19 Chinelatti MA, Chimello DT, Ramos RP, Palma-Dibb RG. Evaluation of the surface hardness of composite resins before and after polishing at different times. J Appl Oral Sci 2006; 14: 188-92.
- 20 Bouschlicher MR, Rueggeberg F, Wilson BM. Correlation of bottom-to-top surface micro-hardness and conversion ratios for a variety of resin composite compositions. Oper Dent 2004; 29: 698-704.
- 21 Moore BK, Platt JA, Borges G, Chu Tm, Katsilieri I. Depth of cure of dental resin composites : Iso 4049 depth and microhardness of types of materials and shades . Oper Dent 2008; 33: 408-12.
- 22 Rueggeberg FA. Determination of resin cure using infrared analysis without an internal standard. Dent Mater 1994; 10: 282-86.
- 23 Ruyter IE, Oysaed H. Conversion in different depths of ultraviolet and visible light activated composite materials. Acta Odontol Scand 1982; 40(3): 179-92.
- 24 Watts D, Amer O, Combe E. Characteristics of visible light-activated composite systems. Br Dent J 1984; 156: 209-15.
- 25 Zawawi S, Brulat N, Nathanson D. Curing duration vs. depth of cure and modulus of bulk fill composites. IADR Abstract 2012; 121 Brazil.