# Effect of Curing Time on Polymerization Rate of Bulk-Fill Composite Resins

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**Abstract**
***Objectives*:** The aim of this study was to compare polymerization depth of two bulk-fill and one conventional composite cured for different times.
***Methods*:** This in vitro experimental study was conducted on 54 composite samples (2×4×10mm) fabricated of Tetric N-Ceram bulk-fill, x-tra fil bulk-fill, and Grandio conventional composite cured for 20, 30, and 40 seconds. The microhardness of samples was measured at 0.1, 2, 2.5, 3, 3.5, 4, and 4.5mm depths using a Vickers hardness tester. The results were analyzed using ANOVA, t-test, and Tukey's test.
***Results*:** The x-tra fil, Tetric N-Ceram and Grandio had maximum microhardness at 0.1mm depth after curing for 40 seconds. The microhardness decreased as the depth of composite increased. Microhardness of x-tra fil was higher than that of Tetric N Ceram. By increasing the curing time, the microhardness value of x-tra fil significantly increased up to 2mm depth. In Tetric N-Ceram, by increasing the curing time from 20 to 30 seconds microhardness increased significantly ($P<0.05$) by up to 3.5mm depth. By increase from 20 to 30 seconds, no significant change occurred in microhardness of Grandio samples at 0.1 and 2mm depths, but further increase from 30 to 40 seconds significantly increased the microhardness at all depths ($P<0.05$).
***Conclusion*:** The maximum microhardness was obtained for x-tra fil at 0.1mm depth following 40 seconds of curing. Microhardness in deep areas (>2mm depth) depends on the type of composite, curing time and depth. Overall, 20 seconds of curing for x-tra fil and 30 seconds for Tetric N-Ceram seem appropriate.
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## Introduction

Achieving a beautiful smile is among the main reasons behind many dental visits. Tooth-colored restorations can greatly help in this regard.

Composite resins were introduced to the dental market about 60 years ago (1, 2). Limited polymerization depth is among the main disadvantages of these materials compromising the quality of restorations and increasing the clinical working time (3). Inadequate polymerization can result in discoloration, pulp injury, post-operative tooth-hypersensitivity and eventual failure of restorations (4). Also, it has been demonstrated that rate of polymerization significantly affects the mechanical properties of resin restorative materials (5, 6). Recently, bulk-fill composites were introduced to overcome the problem of limited polymerization depth of conventional composites. Bulk-fill composites enable the application of thicker increments of composite and facilitate the restoration of extensive cavities (7, 8). Translucency, additional initiators and stress decreasing technology are the main advantages of these composites resulting in increased depth of polymerization from 1-1.5mm to 4-5mm (9, 10).

Type of filler (size and volume), type of light curing unit, effects of restorative material on light absorption and method of clinical application and equipment i.e. distance from the light curing tip to the sample surface, and light curing duration are among the main parameters affecting the light polymerization of composites (11, 12). On the other hand, the correlation between the polymerization and hardness has been discussed in many studies (13,14). Li *et al*. (13) assessed the correlation of Knoop microhardness and curing depth of RZE04 experimental composite and reported that the Knoop microhardness decreased along the curing depth. Rode *et al*. (14) evaluated the effect of the distance from the curing tip on cure depth of composite resins via measuring the Vickers microhardness and reported that increasing the thickness of composite resin decreased the microhardness value.

Considering the fact that adequate polymerization is a key factor in longevity of composite restorations, this study aimed to assess the effect of curing time on depth of polymerization of two bulk-fill composites and a conventional composite resin using Vickers hardness test.

## Methods

This study was approved in the Research Committee of Shahid Beheshti University, School of dentistry. This in-vitro, experimental study was conducted on 54 composite samples divided into nine groups (n=6) as follows:

Group A1: Bulk-fill composite (x-tra fil; Voco, Cuxhaven, Germany) cured for 20 seconds

Group A2: Bulk-fill composite (x-tra fil; Voco, Cuxhaven, Germany) cured for 30 seconds

Group A3: Bulk-fill composite (x-tra fil; Voco, Cuxhaven, Germany) cured for 40 seconds

Group B1: Bulk-fill composite (Tetric N-Ceram; Ivoclar Vivadent AG, Schaan, Liechtenstein) cured for 20 seconds

Group B2: bulk-fill composite (Tetric N-Ceram; Ivoclar Vivadent AG, Schaan, Liechtenstein) cured for 30 seconds

Group B3: bulk-fill composite (Tetric N-Ceram; Ivoclar Vivadent AG, Schaan, Liechtenstein) cured for 40 seconds

Group C1: conventional composite (Grandio; Voco, Cuxhaven, Germany) cured for 20 seconds

Group C2: conventional composite (Grandio; Voco, Cuxhaven, Germany) cured for 30 seconds

Group C3: conventional composite (Grandio; Voco, Cuxhaven, Germany) cured for 40 seconds

For sample preparation, the respective composite was applied to a brass mold measuring 2mm in width, 4mm in length and 10mm in height and a transparent Mylar strip was placed over it to achieve a smooth surface at the level of the mold height. The mold was closed by fixing the screws at the sides and the composite was light cured from the exposed surface using an LED light curing unit (L.E. Demetron II, Kerr, CA, USA) with an intensity of 800 $mW/cm^2$.

After completion of lighting, unpolymerized material at the deepest part of the mold was removed using a plastic instrument. Care was taken not to detach the polymerized material from the mold. By doing so, a semi-circular shape was obtained at the bottom of the sample indicating the cured area of composite. The polymerized material remaining in the mold was immediately transferred to a Vickers hardness tester (HSV-1000; Display, Luzhou, Taiwan) for microhardness measurement. Microhardness of specimens was measured at 0.1, 2, 2.5, 3, 3.5, 4 and 4.5mm depths. For this purpose, since the mold was cubic, after curing, we opened the mold and evaluated the samples from lateral surface. On the other hand, the Vickers microhardness tester had a micrometer, which we used to measure any distance. For measurement of microhardness, 300g load was applied to 5 points in each layer with 0.2mm distances for 15 seconds to cause a diamond shape indentation. This protocol was adopted based on a previous study (15). The minimum and maximum values were disregarded and the mean of the remaining three values was calculated and considered as the Vickers number for the respective depth. Measurements (x40 magnification) for all samples were started at 0.1mm distance from the cured surface of sample to bypass the air-inhibited layer and terminated at 4.5mm depth in bulk-fill composites and at 3mm depth in the conventional composite samples. Data were analyzed using SPSS (SPSS Inc., IL, USA). The effect of time and type of composite and the interaction effect of both on microhardness were evaluated using two-way ANOVA. To assess the effect of type of composite following each curing time and also the effect of curing time on each composite, one-way ANOVA and t-test were applied. Also, for pairwise comparisons among the three types of composites subjected to each curing time, Tukey’s test was used.

## Results

The mean and standard deviation (SD) values for microhardness at different depths were calculated for the three composites cured for 20, 30 and 40 seconds by an LED light curing unit and are listed in Table 1.

The x-tra fil composite was found to have the highest Vickers number (127.55±3.33) at 0.1mm depth cured for 40 seconds. Tetric N-Ceram had the highest Vickers number (118.31±9.00) at 0.1mm depth cured for 40 seconds and Grandio composite had the highest Vickers number (116.69±6.56) at 0.1mm depth cured for 40 seconds. The difference in microhardness of composites cured for 20, 30 and 40 seconds and also at different depths was calculated and the following results were obtained:

*After 20 seconds of curing:*

-At 0.1mm depth, no significant difference was found in microhardness of understudy composites.

- At 2mm depth, a significant difference was found in microhardness between x-tra fil and Tetric N-Ceram and also between x-tra fil and Grandio ($P<0.05$). The Vickers hardness number was higher for x-tra fil but the difference in this regard between Tetric N-Ceram and Grandio was not significant ($P=0.96$).

**Table 1- The mean and standard deviation of Vickers hardness number in all groups**

| Type of composite | Curing time | 0.1mm depth | 2mm depth | 2.5mm depth | 3mm depth | 3.5mm depth | 4mm depth | 4.5mm depth |
|---|---|---|---|---|---|---|---|---|
| X-tra fil | 20s | 103.18±5.37 | 61.77±2.05 | 52.18±5.13 | 46.16±1.70 | 38.67±2.60 | 33.56±4.57 | 18.00±2.42 |
| | 30s | 112.67±5.11 | 60.52±3.10 | 53.09±8.88 | 46.75±2.93 | 39.41±1.21 | 32.09±2.75 | 16.62±1.43 |
| | 40s | 127.55±3.33 | 71.00±7.05 | 55.90±5.43 | 50.93±4.73 | 43.42±5.18 | 37.51±2.95 | 26.22±2.62 |
| Tetric N-Ceram | 20s | 107.04±8.55 | 41.18±3.42 | 36.15±1.18 | 35.58±3.45 | 30.12±3.47 | 26.73±2.44 | 13.44±2.83 |
| | 30s | 105.25±3.95 | 55.30±3 | 46.64±4.15 | 45.67±3.69 | 39.47±4.11 | 30.24±3.17 | 15.63±2.56 |
| | 40s | 118.31±9.00 | 57.53±10.4 | 51.28±9.48 | 42.01±2.92 | 36.32±3.86 | 31.56±5.47 | 17.13±2.02 |
| Grandio | 20s | 99.52±5.54 | 40.76±3.10 | 28.50±2.88 | 12.66±3.28 | | | |
| | 30s | 105.46±4.77 | 47.86±2.39 | 36.84±1.49 | 17.64±3.76 | | | |
| | 40s | 116.69±6.56 | 56.86±8.23 | 43.45±4.17 | 23.41±5.23 | | | |

-At 2.5 mm depth, the Vickers hardness number of x-tra fil was significantly higher than that of Tetric N-Ceram and Grandio ($P<0.05$). Also, the Vickers hardness number in Tetric N-Ceram was significantly higher than that of Grandio ($P<0.05$).

-At 3mm depth, the Vickers hardness number of bulk-fill composites was significantly higher than that of conventional composite ($P<0.05$).

-At 3.5, 4 and 4.5mm depths, the Vickers hardness number of x-tra fil was significantly higher than that of Tetric-N Ceram ($P<0.05$).

*After 30 seconds of curing:*

-At 0.1mm depth, a significant difference in hardness was detected between x-tra fil and Tetric N-Ceram and also between x-tra fil and Grandio ($P<0.05$). The hardness was higher in x-tra fil but no significant difference was found between Tetric N-Ceram and Grandio ($P=0.99$).

-At 2 mm depth, a significant difference was found in microhardness among all understudy composites ($P<0.05$). The microhardness of x-tra fil was higher than that of Tetric N-Ceram and the microhardness of the latter was higher than that of Grandio.

-At 2.5 and 3mm depths, no significant difference was noted between bulk-fill composites in terms of Vickers hardness number ($P=0.15$ and $P=0.85$, respectively); but the Vickers hardness number in these composites was higher than that of Grandio conventional composite ($P<0.05$).

-At 3.5mm depth ($P=0.97$), at 4mm depth ($P=0.30$) and at 4.5mm depth ($P=0.42$), no significant difference was noted between x-tra fil and Tetric N-Ceram in microhardness.

*After 40 seconds of curing:*

-At 0.1mm depth, no significant difference was noted in microhardness between x-tra fil and Tetric N-Ceram ($P=0.07$) or between Tetric N-Ceram and Grandio ($P=0.90$). However, x-tra fil had significantly higher microhardness than Grandio ($P<0.05$).

-At 2mm depth, significant differences were detected in microhardness between x-tra fil and Tetric N-Ceram and also between x-tra fil and Grandio ($P<0.05$). Vickers hardness number of x-tra fil composite was significantly higher than that of other composites but the difference between Tetric N-Ceram and Grandio in this regard was not significant ($P=0.99$).

-At 2.5mm depth, microhardness of x-tra fil was significantly higher than that of Tetric N-Ceram and Grandio ($P<0.05$).

-At 3mm depth, the microhardness of bulk-fill composites was significantly higher than that of Grandio conventional composite ($P<0.05$).

-At 3.5, 4, and 4.5mm depths, the hardness of x-tra fil was significantly higher than that of Tetric N-Ceram ($P<0.05$).

Statistical analysis showed that increasing the polymerization time increased the microhardness of x-tra fil composite by 2mm depth ($P<0.05$) but, this increase at 2 to 4mm depths was not significant.

For Tetric N-Ceram, by increasing the polymerization time from 20 to 30 seconds, microhardness increased at 2, 2.5, 3 and 3.5mm depths ($P<0.05$) but increasing the polymerization time from 30 to 40 seconds did not cause a significant change in this regard.

For Grandio composite (control), increasing the polymerization time from 20 to 30 seconds had no significant effect on microhardness at 0.1 and 2mm depths. But, increasing the polymerization time from 30 to 40 seconds significantly increased the microhardness at all depths ($P<0.05$).

Type of composite used had a significant effect on microhardness. Our study showed that the microhardness of x-tra fil was higher than that of Tetric N-Ceram at all depths and curing times (except for 20 seconds of curing at 0.1mm depth, where the microhardness of Tetric-N-Ceram was higher than that of x-tra fil).

In general, the highest Vickers microhardness number belonged to x-tra fil cured for 40 seconds. Also, at 4.5mm depth, microhardness significantly dropped below the acceptable level.

## Discussion

Adequate polymerization plays a critical role in success and durability of composite restorations. Unpolymerized components are responsible for decreased chemical stability, increased susceptibility to degradation and release of formaldehyde and methacrylate resulting in pulpal reactions, lower strength of the restoration and less color stability (4-6).

As mentioned earlier, bulk-fill composites were introduced to facilitate the restoration of large cavities. Their relative translucency is a main advantage increasing their polymerization depth by 4-5mm (7-10). This study assessed the curing depth of bulk-fill composites cured for different periods.

Several light curing units with different light sources and variable intensities are available in the market including but not limited to quartz-tungsten-halogen (QTH), LED, plasma-arc and laser technology light curing units with an energy intensity of 300 to more than 1000 $mW/cm^2$ (16,17). Variable light curing units have been used in similar previous studies (18,19). Malhotra and Mala in their review study in 2010 stated that the LED and conventional QTH light curing units were not different with regard to the depth of cure or microleakage of restorations (20). In the current study, we used an LED light curing unit.

In previous studies conducted in 2013 and 2014, 4mm curing depth has been confirmed for bulk-fill composites (21-23). In the current study, curing by up to 4.5mm depth was evaluated. Evidence shows that several factors namely the filler type (size and volume), passage of light, thickness and color (shade) of restorative materials, light curing time, distance form the light source to the surface of sample, and light intensity affect the rate and depth of polymerization (24). The current study evaluated the effect of duration of curing on polymerization depth of bulk-fill composites.

Microhardness measurement is a simple method to evaluate the quality of polymerization of composites (25-27). The quantity of hardness refers to strength and resistance against a compressive force (28). Surface hardness of a composite resin relates to its resistance to deformation and capability to remain stable (29). In the current study, we used microhardness test to assess the quality of polymerization of composites using Vickers hardness tester by applying 300g load for 15 seconds. Microhardness in each indentation was measured. This method has been successfully used for measurement of microhardness by many previous studies (30-33).

Our results showed that increasing the polymerization time increased the microhardness of x-tra fil composite to 2mm depth but had no effect on polymerization at 2-4mm depths. In Tetric N-Ceram, by increasing the polymerization time from 20 to 30 seconds, microhardness increased at 2, 2.5, 3 and 3.5mm depths but further increasing the polymerization time from 30 to 40 seconds did not cause any change in microhardness of samples. In this composite, microhardness at 4mm depth was not affected by the polymerization time. In Grandio composite, no difference in microhardness was noted between curing for 20 and 30 seconds to 2mm depth but further increasing the polymerization time from 30 to 40 seconds increased the microhardness at all depths.

Increased microhardness due to increased polymerization time has been reported in some previous studies (18,19,32). For instance, Alpoz *et al*, (19) in 2008 compared the microhardness and compressive strength of Tetric-Ceram, Compomer, Compoglass and Fuji II LC glass ionomer using halogen and LED light curing units following 20 and 40 seconds of curing and concluded that increasing the curing time using LED light curing unit increased the microhardness of all materials and was suitable for composite resin polymerization in deep cavities. Such an increase in microhardness following increased curing time has also been reported by Mousavinasab and Meyers (34) in 2011. However, some studies did not find a correlation between microhardness number and longer polymerization time (35-37). For instance, Flury *et al*, (36) in 2012 measured the curing depth of bulk-fill composites using two methods of ISO4049 and microhardness testing after 10 and 20 seconds of curing and reported no significant difference in microhardness of Tetric N-Ceram cured for 10 and 20 seconds. In our study, no change in microhardness of Tetric N-Ceram was observed by increasing the curing time from 30 to 40 seconds at 2-3.5 mm depths. This finding (no increase in microhardness) may be attributed to the different types of initiators, using two initiators and adequate polymerization of Tetric N-Ceram (38). Microhardness measurement at deeper underlying layers may reveal differences in microhardness as the result of different curing times (21).

Our study also revealed that the microhardness of x-tra fil was higher than that of Tetric N-Ceram following different curing times and at all depths (except for 20 seconds at 0.1mm depth where the microhardness of Tetric N-Ceram was higher than that of x-tra fil). This finding is in accord with the results of previous studies (21,39). Illie *et al*, (21) in 2013 investigated the effect of polymerization time and distance from the tip of the light curing unit to sample surface on micromechanical properties of two types of bulk-fill composites and reported that Vickers hardness number of x-tra fil was higher than that of Tetric N-Ceram. A possible explanation for this finding is the greater translucency of x-tra fil compared to that of Tetric N-Ceram. Higher translucency allows for better penetration of light to the deep layers. Moreover, the difference in the translucency of composites is attributed to difference in the refractive index of filler particles and resin matrix (40,41). Size of filler particles in x-tra base composite may reach 20µm. As the result, reflection of light at the filler-matrix interface decreases allowing for greater penetration of light into the material and subsequently better polymerization of composite. Size of filler particles, silane applied to the filler particles, and wavelength of irradiated light are among other factors influencing the penetration of light deep into the composite resin (42,43). Therefore, considering the presence of Ivocerin as a main photo initiator in Tetric N-Ceram and its different absorbance spectrum in comparison with other initiators as well as the use of LED light curing unit, which has a narrow spectrum, unexpected results in different depths are justifiable.

## Conclusion

Within the limitations of this study, it was found that increasing the polymerization time in x-tra fil composite increased the microhardness by 2mm depth. In Tetric N-Ceram, increasing the polymerization time from 20 to 30 seconds increased the microhardness at 2, 2.5, 3 and 3.5mm depths but further increase in curing time from 30 to 40 seconds did not cause a significant change in microhardness. In Grandio composite, microhardness did not change at 0.1 and 2mm depths by increasing the polymerization time from 20 to 30 seconds. But further increase in curing time from 30 to 40 seconds increased the microhardness at all depths.

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