





## Effect of different curing modes on the Vickers hardness number and curing depth of bulk fill composites





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### Article Type

### ABSTRACT

#### Research Paper

**Introduction:** The purpose of this study was to compare the Vickers hardness number (VHN) and depth of cure of two types of bulk fill composites in high, low and soft light curing modes.

**Materials & Methods:** In this experimental study, 60 cylindrical samples were fabricated from two types of bulk fill composites (Tetric N-Ceram and X-tra fil) in a Teflon mold with one semi-circular notch. Then, the samples were randomly divided into the following three subgroups based on the curing modes (high, low and soft) and were light-cured. The samples were removed from the molds, and their VHN and depth of cure were measured. Data were analyzed using One-way ANOVA and Tukey's post hoc test at the significance level of  $P < 0.05$ .

**Results:** The mean VHN of the X-tra fil composite was significantly higher than that of Tetric N-Ceram composite ( $P < 0.001$ ). The depth of cure of X-tra fil composite was also significantly higher than that of Tetric N-Ceram composite in high and soft curing modes ( $P < 0.001$ ).

**Conclusion:** According to the current results, X-tra fil composite is a convenient material for the restoration of deep cavity in posterior teeth compared with Tetric N-Ceram.

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## Introduction

The improvements in physical and esthetic properties of novel composite resins have made them the most commonly used tooth-colored restorative materials. They are extensively used for the replacement of amalgam restorations.<sup>[1,2]</sup> In addition to the numerous advantages of light cured dental composite resins, limited depth of cure and high polymerization shrinkage are among the main problems of them.<sup>[3]</sup> The degree of polymerization of composite resins is an influential factor in the clinical success of composite restorations.<sup>[4]</sup> There are many ways to overcome this problem such as using the layering technique, adjusting the exposure mechanism, and use of the bulk fill composite resins. Basically, the curing of composites depends on composite properties such as type of photo-initiator, filler type. The degree of conversion depends on factors such as curing time, radiation, optical spectrum emission, and increment thickness.<sup>[5]</sup> One solution to achieve a restoration with a high degree of conversion and minimal polymerization shrinkage is the incremental application of composite resins with increment thickness < 2 mm.<sup>[6]</sup> However, the application of incremental technique and separate curing of each layer is time-consuming for both patients and dentists. There is also a probability of void formation or moisture contamination between the composite layers.<sup>[7]</sup>

Recently, bulk-fill composites were presented to enhance the process of composite application and decrease its technical sensitivity. Bulk fill composites have advantages such as bulk placement and uniform polymerization to a depth of 4 mm in one step without any crack or void formation.<sup>[8, 9]</sup> Evidence shows that bulk fill composites have significantly lower polymerization shrinkage than conventional composite resins. Therefore, subsequent problems associated with polymerization shrinkages such as gap formation, secondary caries, and post-operative sensitivity can be minimized. On the other hand, the use of these composites can save time compared with the incremental composite application.<sup>[10]</sup>

Increased depth of cure of bulk-fill composites is not only because of their translucency but also because of their modified monomers and photo-initiator system.<sup>[11]</sup> The higher radiation intensity that is used for composite resins results higher degree of conversion. In other words, there is a possibility of more shrinkage stress during polymerization reactions. A soft polymerization model has been introduced to minimize the polymerization shrinkage and subsequent formation of the marginal gap.<sup>[12]</sup>

One of the most commonly used technique for measuring of the hardness is the Vickers hardness test. Hardness is the measure of resistance to the permanent indentation or surface penetration. The minimum acceptable hardness level is above 80% for the hardness ratio of the lower part to the upper part of a restoration.

By measuring the composite hardness in the upper and lower parts, the depth of cure can be determined.<sup>[13]</sup>

Tetric N-Ceram bulk-fill is a light cure composite with radiopaque nanofillers According to the manufacturer claims, this restorative material contains barium glass, mixed oxide, ytterbium trifluoride, prepolymer (weight: 78%-81%), catalysts, stabilizers, and pigments, additives. The filler particle size is between 40-3000 nm with an average size of 550 nm. It shows high radioopacity that makes it easier to detect secondary caries. With Ivocerin as photo-initiator in this composition, it can be cured in 10 seconds even when applied in 4-mm increments. Ivocerin is a polymerization enhancer in a standard photo-initiator system and is more sensitive than camphorquinone.<sup>[4]</sup>

The X-tar fil composite is a light-cured and packable posterior composite for restoration of posterior teeth. In addition, core build-up is based on bulk-filling techniques with high filler level and lower shrinkage than conventional composites.<sup>[14]</sup>

Makhdoom et al. Compared depth of cure's Three composites Tetric EvoCeram Bulk Fill, Filtek Bulk Fill, and Tetric EvoCeram with two method scrape test method (ISO-4049) and FTIR, as well as showed that similar trends and with increased percentage values obtained upon increased mean irradiance (mW/cm<sup>2</sup>).<sup>[15]</sup> However, Sampaio et al. showed No differences were observed for the same bulk fill resin composite within curing modes.<sup>[16]</sup>

Although the manufacturers recommend that bulk-fill composites should be used in over 4 mm thickness, due to the lack of information regarding the depth of cure and mechanical properties (including hardness) of these composites, this study aimed to assess the Vickers hardness number (VHN) and depth of cure of bulk-fill composites subjected to different light-curing modes.

## Materials & Methods

This study was approved by the Ethics Committee of Babol University of Medical Sciences, Babol, Iran (with the code of IR.MUBABOL.REC.1396.20 )

In the current experimental study, 60 specimens samples were prepared from two types of bulk-fill composites (N=30 for each type) in a semi-cylindrical Teflon mold. The general composition of materials used in this study is provided in Ttable1.

**Table 1. Characteristics of composites, their type, composition and manufacturer**

| Composite/Code           | Manufacturer                            | Shade/ LoT<br>(expiration date) | Resin<br>composition   | Filler<br>amount wt%<br>/vol% |
|--------------------------|---|---------------------------------|------------------------|-------------------------------|
| Tetric N-Ceram Bulk Fill | Ivoclar Vivadent, Schaan, Liechtenstein | IVA/U55063 (2019-09)            | Bis-GMA, Bis-EMA, UDMA | 81.61                         |
| X-tra fil                | Voco, Cuxhaven, Germany                 | Universal/1637111(2019-03)      | Bis- GMA, UDMA, TEGDMA | 86.70                         |

To prepare the samples, the mold was filled with Tetric N-Cream bulk-fill and X-tra fil composite in bulk until it was slightly overfilled. A Mylar strip was then placed over the composite's surface and next the top surface of each specimen was covered with a glass slide to ensure the smooth surface of specimens of resin composite. A LED curing unit (Blue-Phase C8; Ivoclar, Vivadent, Schaan, Liechtenstein,) was used to cure the samples. Thirty samples of each material were randomly subdivided into the following three subgroups based on the mode of curing:

High: Curing with an output intensity of 800 mw/cm<sup>2</sup> for 15 seconds

Low: Curing with an output intensity of 650 mw/cm<sup>2</sup> for 20 seconds

Soft: Curing with an output intensity of 650 mw/cm<sup>2</sup> for 10 seconds and then 800 mw/cm<sup>2</sup> for 10 seconds

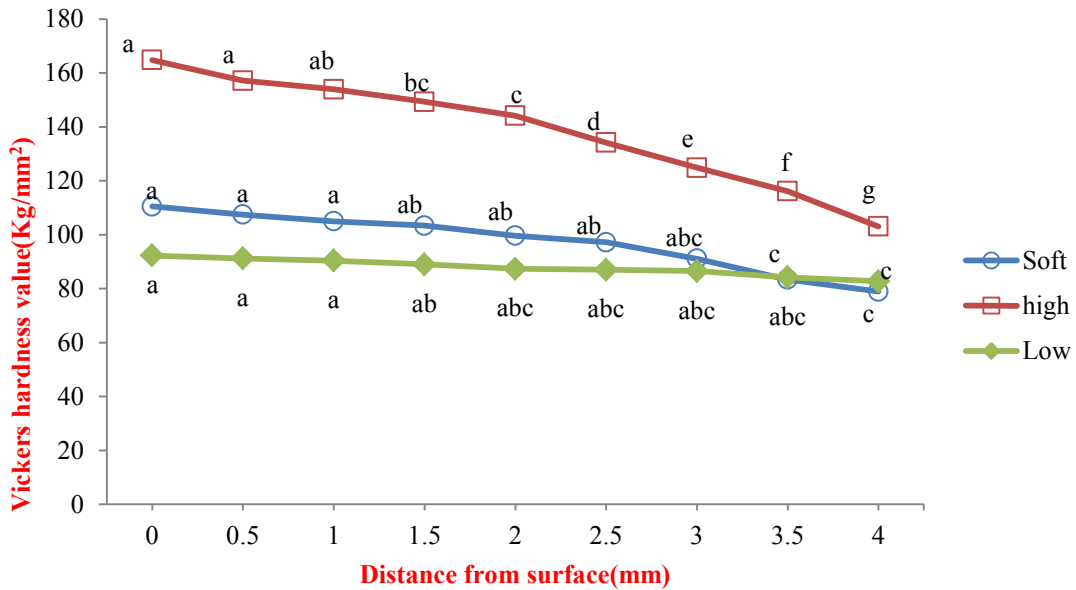
The tip of the light-curing unit was kept in contact with the glass slide during the curing process. The output power of the light-curing unit was checked after curing every 5 specimens using a radiometer (Blue-Phase C8; Ivoclar, Vivadent, Schaan, Liechtenstein, Germany) .

The Mylar strip and the glass slide were then removed and the samples were gently removed from the mold. A Vickers hardness tester (MH1, Coopamashhad, Iran) was used to measure the hardness of the samples. For each semi-cylindrical sample, the hardness of the upper surface, and then the lateral surface was measured at 0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4 mm distance from the surface. Indentations were made with 50 g load with a dwell time of 30 seconds using a diamond indenter. <sup>[1]</sup> For each sample, the depth of cure was determined as the point where the bottom/top hardness ratio of 0.80 was calculated.

The obtained data were statistically analyzed by SPSS 17 (SPSS Inc.; Chicago, 11, USA) through repeated-measurement ANOVA and independent T-test.

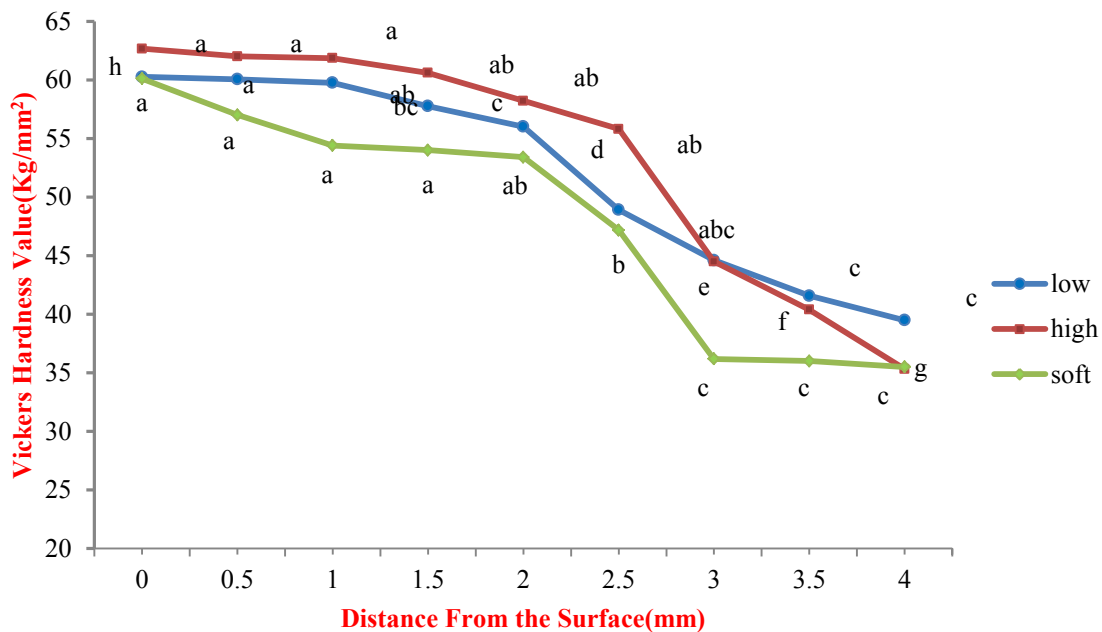
## Results

The microhardness depth profiles of X-tra fil composite in different curing modes are presented in Figure 1. As shown, the highest and the lowest Vickers hardness number(VHN) were recorded on the top surface and bottom surface of the X-tra fil composite in all curing modes. Comparison of different curing modes at each distance from the surface showed that the highest and the lowest hardness values were recorded in high and low curing modes, respectively. The exception was at 3.5 and 4 mm distance from the surface where the lowest values were recorded in soft curing mode. Values with similar lowercase letters in each curing mode are not significantly different.



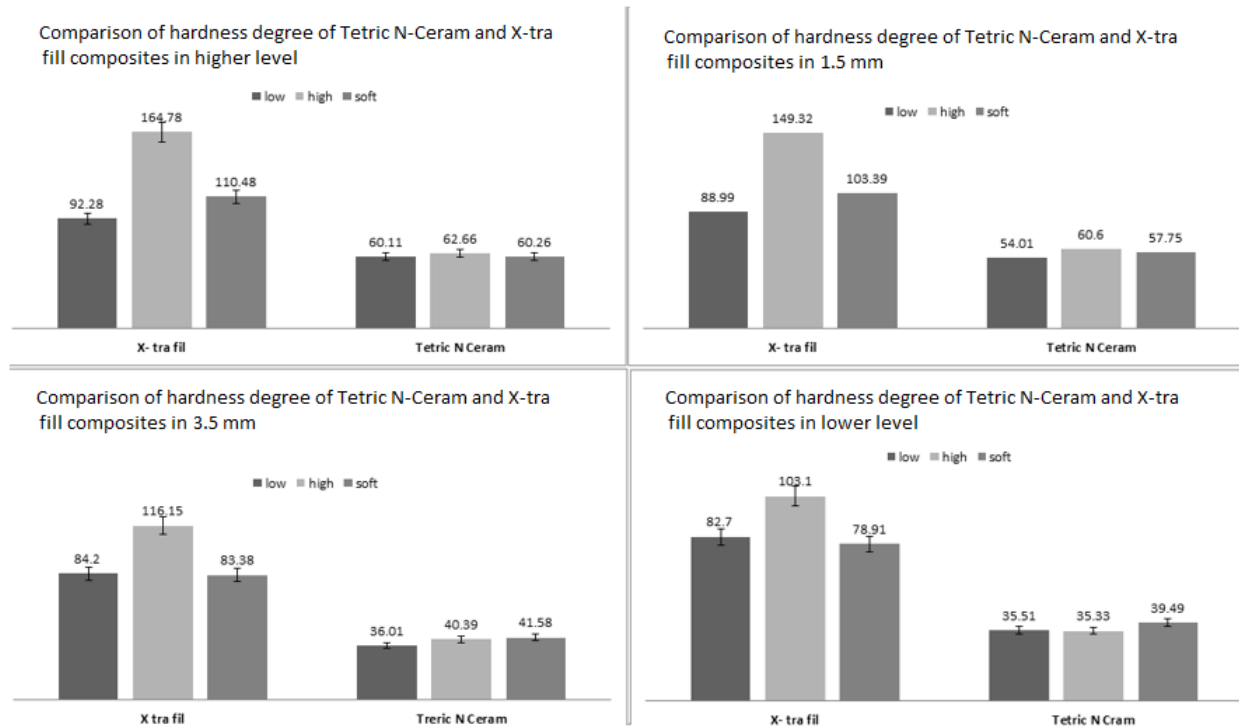
**Figure 1. Mean Vickers hardness profile of X-tra fil composite in different curing modes and different distances from the surface.**

Figure 2 shows the VHN of Tetric N-Ceram composite in different curing modes and different distances from the surface. Similar to the X-tra fil composite, the highest and the lowest hardness VHN were recorded on the top surface and the bottom surface of the Tetric N-Ceram composite for all curing modes. At each distance from the surface, the hardness values of different curing modes were not significantly different.



**Figure 2. Mean Vickers hardness profile of Tetric N-Ceram bulk fill composite in different curing modes and different distances from the surface.**

Figure 3 shows the mean microhardness values of X-tra fil and Tetric N-Ceram composites concerning the influence of curing modes and distance from the surface. The results showed that X-tra fil composite reached significantly higher VHN than Tetric N-Ceram composite ( $P < 0.001$ ). This was true for all curing modes and all distances from the surface.



**Figure 3. Comparison of the Vickers hardness number( $\text{kg}/\text{m}^2$ ) of X-tra fill and Tetric N-Ceram in higher and lower levels and in 3.5mm and 1.5mm in depth**

There was a significant difference in depth of cure between X-tra fil composite samples cured with the low mode and those cured with high and soft modes; whereas, in Tetric N-Ceram bulk fill composite, there was no significant difference between the three curing modes. The depth of cure reached its maximum value of 3.95 mm in X-tra fil composite samples cured by the soft mode, and its minimum of 2.15 mm in Tetric N-Ceram bulk fill composite samples cured by the low mode (Table 2). Moreover, in both high and soft modes, the depth of cure in X-tra fil composite is significantly higher than that in Tetric N-Ceram bulk fill composite.

**Table 2. Mean curing depth (mm) of Tetric N-Ceram Bulk fill and X-tra fil composites in different curing modes**

| Composite                | Different modes of curing |                        |                        | P value |
|--------------------------|---------------------------|------------------------|------------------------|---------|
|                          | Low                       | High                   | Soft                   |         |
| X-tra fil                | 2.75±1.11 <sup>b</sup>    | 3.85±0.34 <sup>a</sup> | 3.95±0.16 <sup>a</sup> | 0.001   |
| Tetric N-Ceram bulk-fill | 2.15±0.24 <sup>b</sup>    | 2.50±0.24 <sup>a</sup> | 2.50±0.47 <sup>a</sup> | 0.040   |
| P value*                 | 0.521                     | <0.001                 | <0.001                 |         |

(Different lowercase letters in one row show significant differences in depth of cure of each composite)  
P Value: One Way ANOVA, P Value\*: Independent samples T Test

## Discussion

In this experimental study, the depth of cure and the VHN of Tetric N-Ceram and X-tra bulk fill composites were assessed in three different curing modes (low, high, and soft). The VHN for both types of composites in the three modes of curing was the highest in the upper surface and lowest in the lower surface. The mean VHN of X-tra fil was significantly higher than that of Tetric N-Ceram. Also, in high and soft curing modes, the curing depth of X-tra fil was significantly more than that of Tetric- N-Ceram. The main characteristic of bulk fill composites is their increased depth of cure, which is due to their high translucency because of the changed filler content and organic matrix.<sup>[17]</sup> A higher polymerization rate is a necessary factor to reach higher physical and mechanical properties. However, inadequate polymerization leads to marginal leakage, discoloration, lower bond strength, and accelerated wear.

In this study, VHN testing was performed to measure the depth of cure. Generally, there are direct and indirect methods to measure the depth of cure. Direct methods include infrared spectroscopy and laser roman and indirect methods include scratching and micro-hardness testing. Direct methods are complex, expensive and time-consuming; however, hardness testing is the most popular indirect method to assess the depth of cure.<sup>[15, 11]</sup> The Vickers hardness testing method is easily used and its results are reliable. The application of Vickers diamond indenter does not change over time.<sup>[9]</sup>

Depth of cure is defined as the depth of cured composite with a hardness value of 80% of the surface hardness.<sup>[18]</sup> Accordingly, the depth of cure of X-tra fil composite in high and soft modes was significantly higher than that of Tetric N-ceram, which was consistent with the results of Kelic et al.<sup>[19]</sup> and Abed et al.<sup>[9]</sup> The reason can be the difference in the chemical composition of the resin matrix and the filler content. The resin matrix of X-tra fil contains Bis-GMA, UDMA and TEG-DMA while the resin matrix of Tetric N-ceram contains Bis-GMA, Bis-EMA and UDMA. The polymerization ratio of different polymers is as follows: Bis-GMA < Bis-EMA < UDMA < TEG-DMA. Bis-GMA is the most viscous monomer with the lowest flexibility. UDMA is also a viscous monomer, but it has a lower viscosity and higher flexibility. TEG-DMA has the lowest viscosity and the highest degree of conversion. Indicated that the polymerization rate of composites containing TEG-DMA and Bis-GMA is higher than that of composites containing other monomers.<sup>[9, 19, 20]</sup> Other effective factors on the polymerization rate of composites include filler volume and filler particle size.<sup>[12]</sup> X-tra fil is a micro-hybrid composite with 86wt% filler (70v %), but Tetric N- ceram is a nano-hybrid composite with 75-77wt% (53-56v %) filler. Higher filler volume and translucency can increase light penetration and depth of cure.

Abed et al. reported that larger filler size is responsible for the absence of a significant difference between the micro-hardness of the upper surface of Quix Fill compared with its lower surface, in comparison with other composites.<sup>[9]</sup> The hardness of the upper surface of bulk-fill X-tra fil and Tetric N-ceram was the highest in all three modes of curing. The lowest VHN was noted in the lower surface, which is consistent with other studies.<sup>[3, 21, 22]</sup> This is because of the fact that composite micro-hardness decreases by the increased height of cured resin samples. On the other hand, in this study, hardness of Tetric N-ceram composite in different depths and in three curing modes of low, soft and high had no significant difference with each other, which is in accordance with the results of Farahat et al.<sup>[4]</sup> Tetric N-ceram is a non-flowable bulk fill composite with a translucent filler content allowing light penetration. However, it contains Ivocerin (Ivoclar Vivadent) which is a light initiator with a germanium base. According to the manufacturer, Ivocerin has a higher potential for light activation compared with camphorquinone. The reason is because of higher absorbance in the light spectrum of 400-450 nm. Therefore, it can be used without adding amine as a co-initiator and produces at least two free radicals which are capable of initiating radical polymerization. Therefore, it is more effective than camphorquinone-amine systems with at least one free radical.<sup>[18]</sup> However, in our study, the hardness ratio of different depths in the three modes of curing had no significant difference. In a previous study, the degree of conversion and depth of cure of different composite resins including Tetric N-ceram were evaluated. The results showed that Tetric N- ceram samples had a lower degree of conversion, which justifies the hardness testing results.<sup>[23]</sup>

However, other studies explained that the characteristics of the light-curing units influenced the degree of polymerization of the bulk fill resin composites. The use of a wide tip with homogeneous light distribution and multiple peak light-curing units preferred the light curing of bulk fill resin composites.<sup>[24, 25]</sup>

Nevertheless, within the limitations of the study design (specimen size and shape, inaccessibility to new version of light-curing units, etc.) definitive conclusions cannot be made, and further investigations are needed to compare the result with the indirect method (infrared spectroscopy and laser roman).

## Conclusion

In general, the results demonstrated that the X-tra fil composite had a higher Vickers Hardness Number in comparison with the Tetric N-Ceram composite. Therefore, it can be a suitable candidate for the restoration of posterior teeth.

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## Conflicts of Interest

We declare no conflict of interest

## Authors' Contribution

The study was designed by Fariba Ezoji, Saeedeh Zahedian and Ghazaleh Ahmadi zenouz. The study data were collected by Saeedeh Zahedian. Analysis and interpretation of data were performed by Hemmat Gholinia. The article was written by Fariba Ezoji. Study supervision was conducted by Fariba Ezoji.

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