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Hardness and Depth of Cure of Conventional and Bulk-fill Composite Resins in Class II Restorations with Transparent and Metal Matrix Strips

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ABSTRACT

Objectives: Hardness is relevant to the degree of conversion (DC) and depth of cure (DoC). The aim of this study was to determine the micro-hardness and DoC of conventional and bulk-fill composite resins in class II restorations using metal and clear matrix bands.

Materials and Methods: Twelve specimens of each of the two composite resins, *i.e.*, Filtek Z350 XT bulk-fill and Gradia posterior conventional composite, were prepared in the form of a class II cavity in a tooth mold, using a clear or metal matrix band. All specimens were cured and stored at 37°C for 24 hours. Vickers hardness was measured as a function of DoC at 2mm intervals. Data were analyzed by two-way ANOVA (alpha=0.05).

Results: The bulk-fill composite exhibited significantly higher hardness levels than the conventional composite in all tested surfaces (P<0.001). However, while the metal matrix band had a significant impact on the bottom surface (P=0.059) and also on the furthest surface from the matrix and light source (P=0.04), it did not have a consistent effect across all tested surfaces. The simultaneous interaction of the composites and matrix band types in all surfaces, did not show significant differences in hardness values. The highest bottom-to-top surface hardness ratio (73%) was observed in the conventional composite near the metal matrix band.

Conclusion: In deep class II cavities, the bottom-to-top surface hardness ratio did not reach the maximum of 80%, neither for bulk-fill nor conventional posterior composites. Therefore, in such cavities extended light-curing and more incremental composite placement is needed.

Keywords: Composite resins; Dental Cavity Preparation; Hardness Tests

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INTRODUCTION

Dental composites are one of the most commonly used restorative materials for the anterior and posterior restorations. The incremental placement technique (maximum of 2mm thickness) has been regarded as the gold standard to apply and cure the composite resins in increments of limited thickness. In addition to increased clinical time and technical complexities, other disadvantages of the incremental filling technique include reduced bond strength as well as void formation, contamination, and bond failure between the adjacent composite layers [1]. With advances in polymer chemistry, photo-activation, and curing light technologies, a new "class" of resin based composites (RBCs) called bulk-fill composites emerged that enable the application of composite resin in 4-5mm thick layers, which are cured easily. Thus, they simplify the restoration of large posterior cavities [2].

There are many concerns about the use of light curing composites in class II restorations especially deep cavities; one of which is the limited depth of cure (DoC) and the probability of insufficient polymerization in depth mainly for the bulk-fill composites [3].

As the distance between the light curing tip and restoration increases, the light intensity decreases much more proportionally. This reduction is 7% at a distance of 2mm, reaching up to 25% at a distance of 4mm [4]. Also, increasing the composite thickness and the reduction of light intensity caused by reflection, scattering, and absorption by the first layer decrease the polymerization rate, and consequently compromise the physical, mechanical, and biological properties of composite resins [5]. This phenomenon can be correlated with the failure of the restorations [6].

On the other hand, class II cavity walls of restorations are confined to the dental tissue and the matrix band with a different index of refraction (IoR). As we know, there is no comprehensive study on the effect of band types on composite polymerization. The IoR for the steel and transparent bands is different from that of the tooth structure. Therefore, the interactions of light with these materials is different [7] and there is a question about the effect of matrix band type on composite polymerization.

The light cure composites are cured to a certain depth. This depends on the penetration depth of visible light into the composite mass. The conversion rate decreases by an increase in depth [8]. The DoC is the thickness of composite that is adequately polymerized [9]. The use of thicker increments in bulk-fill composite resins is due to both developments in photo-initiator dynamics and their increased translucency, which allows additional light penetration and a deeper cure. DoC is dependent on filler (type, size, and load), light irradiance, exposure time, radiation exposure, and also resin composition and shade [10].

The DoC can be determined with several methods. For dental composites, ISO-4049 advocates scraping of the unset material after irradiation and measuring the length of the set specimen [11].

Also, by measuring the microhardness or degree of conversion (DC) of the top and bottom surfaces of a specimen, the DoC can be determined [12]. The concept of DoC in the scrape back technique differs from its practical (clinical) meaning that is equal to the depth at which the DC or hardness is 80-90% of the maximum value at the top layer, which is adjacent to the light source [13].

In general, the results of micro-hardness tests show that the ISO-4049 standard over-estimates the DoC, particularly for the bulk-fill composites [14,15]. Therefore, it will not be a good indication for the clinical performance of composites. Also, in vitro measurements of the hardness and DoC are usually performed on standard composite disks. The preparation of the standard disks in a metal or Teflon molds will change the scattering, absorption, and transmission of light compared with the clinical setting where composite restoration is surrounded by the tooth structure [16].

Micro-hardness measurement is a common, simple, and precise technique for indirect measurement of dental composite resin polymerization [9,17]. In this technique, the acceptable values of the DC in the specified depth are calculated as 80-90% of the upper layer hardness [18].

Based on all the above, the purpose of this study was to evaluate the DoC of two commonly used bulk-fill and conventional composite resins with metal and transparent matrix bands. The following parameters were evaluated: (I) the maximum Vickers hardness number (VHN), (II) 80% of the maximum VHN, and (III) the depth corresponding to 80% of the maximum VHN.

MATERIALS AND METHODS

Tooth preparation:

Two mandibular third molars extracted for orthodontic purposes were used in this study. The teeth were free from defects and had proper dimensions. One tooth was used to prepare a mold, and the other was used as an adjacent tooth for a class II restoration. The teeth were immersed in 0.05% Thymol solution for one week for disinfection; then they were subjected to scaling and root planing and kept in distilled water for one week until the experiment.

The teeth were mounted in dental stone and to achieve a flat occlusal surface, they were slightly shortened by a rough diamond disc (OptiDisc 4200; Kerr, Switzerland). To optimize accessibility, the Class II cavity was prepared on the occlusal surface rather than the proximal surface (buccolingual dimension 6mm, mesio-distal dimension 8mm, and occluso-gingival height 4mm). This resulted in most of the cavity height being situated on the occlusal surface, allowing for effortless placement of the matrix strip on the flattened occlusal surface. Furthermore, instead of utilizing occlusal exposure, exposure was achieved from the proximal side of the cavity. In essence, this cavity served as a tooth mold whereby the samples were removed after curing.

Preparation of composite specimens:

A2 shade Filtek Z350 XT composite and Gradia posterior conventional composite were used in this study (Table 1). This shade decreases the effect of pigments on composite polymerization [15]. Filtek composite contains zirconia filler particles (IoR=2.2) with different optical properties from silica (IoR=1.53), and was selected to investigate the effect of this filler on light transmission and, consequently, the DoC of the composite. To prevent composite-tooth adhesion, petroleum jelly was applied on the teeth and two groups of specimens were prepared. In one group, a metal matrix band and in the other group, a transparent matrix strip was placed on the occlusal surface and held firmly with fingers.

A wisdom tooth was fixed next to a transparent strip using high viscosity silicone putty. This was to simulate clinical situations regarding the possibility of light reflection from the adjacent tooth enamel into the class II cavity. The mold was filled with two increments (4mm-thickness) for the bulk-fill composite, and four increments (2mm-thickness) for the conventional posterior composite. The increments were cured using a LED curing unit (Litex 695; Dentamerica, CA, USA) with an intensity of 1200 mW/cm2 in the free proximal tooth surface, according to the manufacturer's instructions. Each layer of composite was cured for 10 seconds from the free side, then the metal strip was removed, and the tooth was cured for another10 seconds from buccal and another 10 seconds from the lingual side. The surfaces were polished by a consecutive series of polishing discs (OptiDisc 4200; Kerr, Switzerland) for the following purposes:

- Removing the air inhibited layer.
- Easy placement of the samples in the micro-hardness-test machine

Twelve samples were prepared from each composite and matrix (i.e. totally 48 samples). The top (A) and bottom (B) surfaces, and the surfaces adjacent (C) and opposite (D) to the strip were marked. The samples were placed in an incubator at 37°C for 24 hours to complete the polymerization process.

Micro-hardness tester.

The VHN of the composite surfaces was measured by a micro-hardness tester (MicroMet 5114; Buehler, USA), under standard conditions. To measure the hardness, the specimens were

Table 1. Specifications of the composite resins used in the present study

Composite resin	Matrix	Filler	Filler Load	Manufacturer
Filtek Z350 XT (Nano composite)	Bis-GMA, Bis-EMA, UDMA, TEGDMA	Combination of aggregated zirconia/silica cluster filler with primary particle size of 5-20nm, nonagglomerated 20nm Silica Filler and cluster 0.6-1.4μm	78.5% wt (63.3% vol)	3M ESPE/St. Paul, MN, USA
Gradia Direct Posterior (Microhybrid)	UDMA	Silica, pre-polymerized fillers, fluoro-alumino-silicate glass (average particle size 0.85 micron)	77% wt, (65% vol)	GC Co, Tokyo, Japan

Bis-GMA: bisphenol A-glycidyl methacrylate; Bis-EMA: bisphenol A ethoxylated dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate urethane dimethacrylate

Fig 1. Microscopic evaluation of composite surfaces (A) and Application of force on the composite samples using a pyramid diamond probe (B)

first placed in the device, and the desired surface was examined at ×40 magnification to ensure that the surface was free from any voids. Then 150g force was applied to the specimen for 15 seconds by a pyramid diamond probe with at 136° angle (Fig. 1).
The indentation of the pyramid probe was recorded on the sample surface similar to a positive sign (+) (Fig. 2). The VHN was determined based on the mean value of the vertical and horizontal dimensions of the indentation. The VHN was measured at the center of the top (A) and bottom (B) surfaces of each sample.
The surfaces adjacent (C) and opposite (D) to the matrix strip (i.e., mesiodistal length of the sample) were divided into three parts (C1, C2, C3, D1, D2, and D3) with the same length of 2mm intervals. The first (C1, D1) and third (C3, D3) parts were considered as the nearest and farthest points from the light source, respectively.
The raw data from the micro-hardness tests were analyzed using two-way analysis of variance with a significant level of $P \leq 0.05$.

RESULTS

The following results were obtained, based on our statistical analysis:
The maximum mean micro-hardness was observed at the (A) surface in bulk-fill composite with metal matrix band (VHNmax=185.36). The minimum micro-hardness was found at the (B) surface in the conventional composite near the transparent strip (VHNmin=53.58).
In general, the measured hardness related to the bulk-fill composite was significantly more than the conventional composite ($P<0.001$) at all tested surfaces. The effect of using metal matrix bands on the hardness value was significant on (B) and (D1) surfaces ($P=0.05$ and $P=0.04$, respectively).

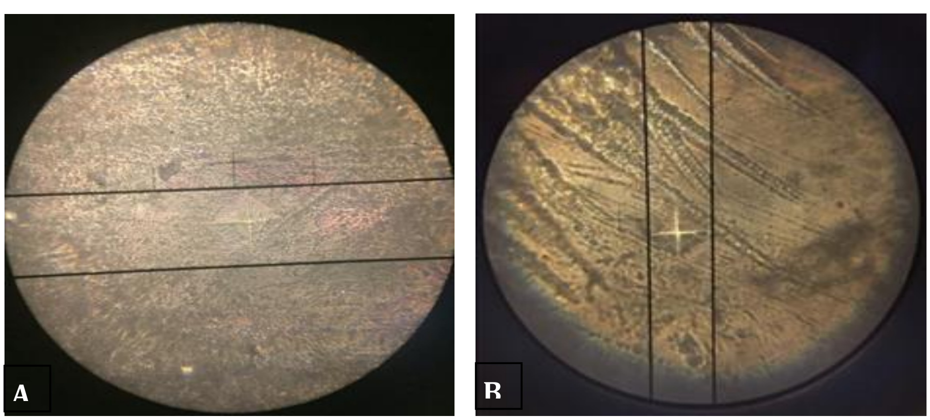

Fig 2. Microscopic images of the probe trace on (A) conventional, and (B) bulk fill composite surfaces

Table 2. Mean (± standard deviation) value of Vickers hardness values and the bottom-to-top surface hardness ratio (B/A)

Composite Resin	Matrix Band	Surfaces		
		A	B	B/A (%)
Filtek Z350 XT	Metal	185.4±31.2	110.7±29.8	59.7
	Mylar	177±42.4	102±18.4	57.6
Gradia Direct Posterior	Metal	88.5±11.4	64.6±3.3	73
	Mylar	94.4±10.4	53.6±4.2	56.7

The tests of between-subjects effects for composites and matrix bands did not show significant differences in hardness value in all surfaces (P>0.05).

The depth corresponding to 80% of the maximum value of hardness (DoC) was not the same for the surfaces adjacent and opposite to the metal and transparent strips. DoC near the metal band was about 1mm greater than the opposite surface. DoC near the metal strip in bulk-fill and conventional composites was 3 and 4mm, respectively. DoC near the transparent strip was 1mm less than the metal strip. DoC in the bulk-fill and conventional composites was 2 and 3mm, respectively.

According to Table 2, the bottom to top surface hardness ratio (B/A) did not reach 80% in any of the samples. The maximum ratio (73%) in the conventional composite was observed near the metal strip. This ratio for bulk-fill composite with metal and transparent bands and conventional composite with the transparent strip was 59.7%, 57.6%, and 56.7%, respectively. The B/C2 and B/D2 ratios exceeded 80% and the C2/A, D2/A ratios varied between 60-70%.

The ratios of B/C3 and B/D3 in the conventional composite were approximately 100%. The ratios of C3/C2 and D3/D2 near the metal and transparent bands were 80% and 75%, respectively. The ratios of C2/C1, D2/D1 for the samples were measured to be >80%. The ratios of C1/A and D1/A were measured to be >90% and 100% for the metal and transparent bands, respectively. Therefore, it can be concluded that the positive effect of metal matrix band on this ratio in deep layers was more significant than the surface layers, as can be seen in Figures 3.

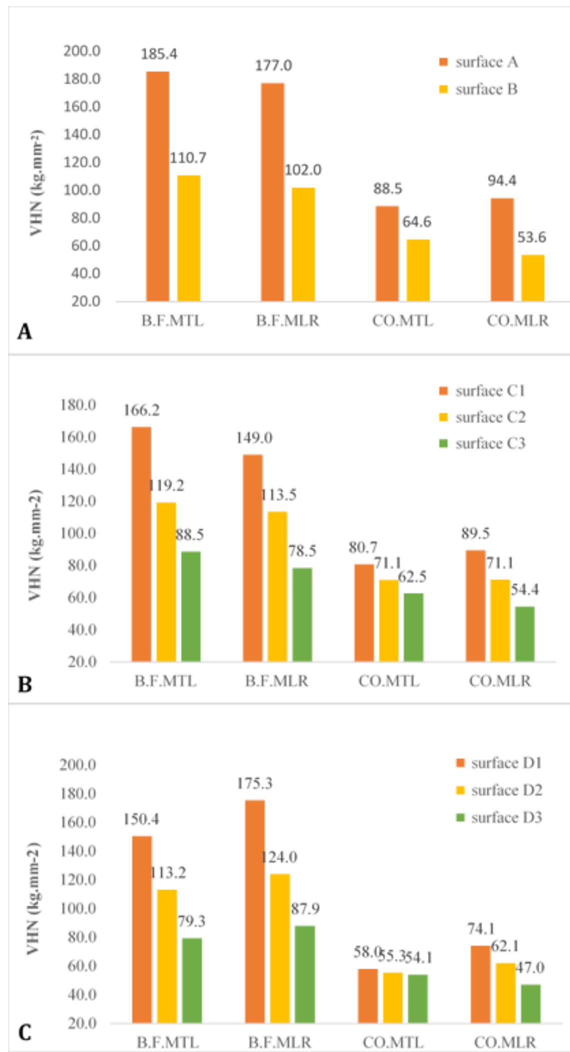

Fig 3. Microhardness in bulk fill (BF) and conventional (CO) composites with metal (MTL) and transparent (MLR) bands measured near A and B surfaces (chart A), adjacent to the matrix strip for C1, C2, and C3 surfaces (chart B), and D1, D2, and D3 surfaces (chart C)

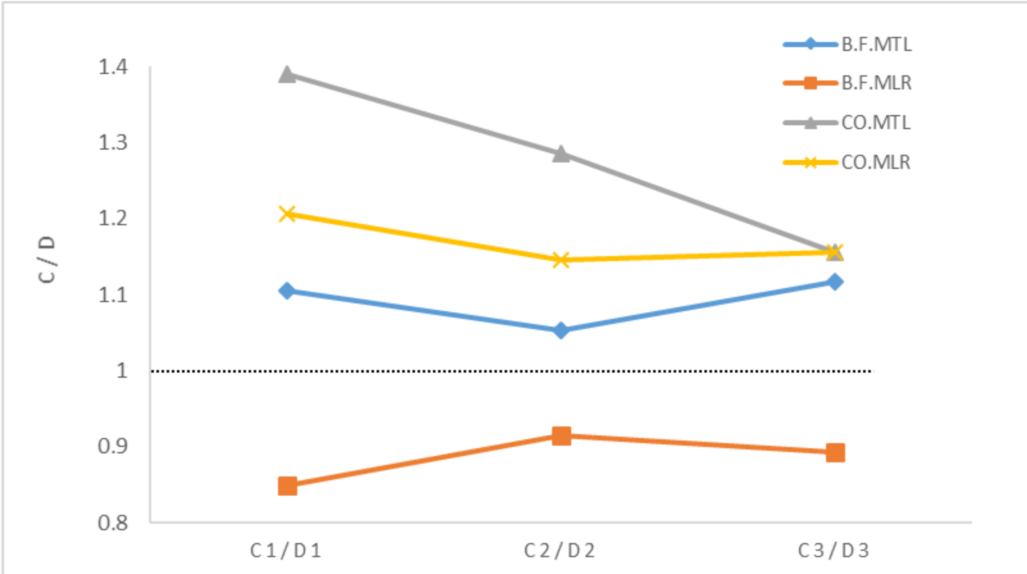

Fig 4. The C/D ratio of hardness at different depths for bulk fill (BF) and conventional (CO) composites with metal (MTL) and transparent (MLR) bands.

According to Figure 4, the C/D ratio of hardness was only less than 1 for the bulk-fill composite with the transparent strip and was more than 1 in all other groups

DISCUSSION

The results of this study showed that there was a significant difference in micro-hardness values between the studied groups for the composite types (P=0.0001). The maximum mean value of VHN was measured near the curing unit (surface A) for the bulk-fill composite with the metal band (185.36). This is due to the intense reaction of bulk-fill composite to the light and the effect of light reflected by the metal band [19]. The minimum mean value of VHN was found for the conventional composite on the B surface and near the transparent band (53,58).

The possible reasons for higher hardness of Filtek Z350 XT are the nano-sized particles and more filler load [20]. Besides, Gradia and Filtek Z350 XT composites contain fluoro-alumino-silicate and silica-zirconia, respectively. Fillers containing silica have a lower hardness than zirconia and their bonding to resin matrix is more difficult [21].

The B/A ratio of hardness did not reach 80% in any of the samples. The great light reduction by transmission through the composite mass and increasing the depth of restoration, decreased the micro-hardness [8].

The degree of conversion of bisphenol A-glycidyl methacrylate (Bis-GMA) monomer in Filtek Z350 XT is lower than that of UDMA in the Gradia composite [22]. Sideridou et al [23] showed that the presence of amine group in the urethane structure of urethane dimethacrylate (UDMA) monomer caused a chain transfer reaction, continuation of polymerization reaction, an increase in mobility of free radicals, and consequently, an increase in polymerization.

A previous study showed that the smaller the filler particles, the greater the light scattering during light curing would be, resulting in a reduction in DC. On the other hand, by increasing the filler content of Filtek Z350 XT, the amount of light transmission through the composite mass and the hardness decrease [24]. Therefore, in bulk-fill composite, light scattering is higher because of the smaller size of nano-fillers and higher load of fillers [25]. The larger particles in the conventional micro-hybrid Gradia composite also have a higher DoC because of lower light scattering [26]. Comparison of the DoC of nanofilled, nano-hybrid, and microfilled composites indicated that the bottom-to-top surface hardness ratio reached 80% only in the hybrid composite [27]. A nanofilled and a micro-hybrid composite were used in a class II restoration in the present study, and this ratio did not reach 80%. The next factors that affect DC are the optical properties of the material, i.e., IoR of zirconia filler (IoR=2.35) is not the same as that of resin matrix. The closer the filler refraction index to that of the resin matrix, the better the light transmission through the composite mass and the higher the DC and DoC would be [15].

When evaluating composites of the same shade, Gradia stands out as having superior translucency and light transmittance in comparison to other opaque composites and bulk-filled alternatives used in the current study. These characteristics lead to greater polymerization at deeper levels [28]. Furthermore, the conventional composites exhibit a higher bottom-to-top surface hardness ratio as a result of the additional layers and extended exposure time of deeper levels. This is in line with the findings of a prior investigation by Poskus et al [22].

In Gradia conventional composite, the bottom-to-top surface hardness ratio for nearly all layers and the surface adjacent to the transparent and metal matrixes was above 80% and for the deepest layer, it reached almost 100%. However, for the bulk-fill composite, this ratio only for the deepest layer reached to more than 80% and for the top surface, this ratio varied from 60-70%, which was consistent with previous studies [22]. Aggarwal et al. [29] reported that the high-viscosity bulk-fill composites were not sufficiently cured in 4-mm increments. The above-mentioned findings were not consistent with the results of Cetin et al, [30] and Alrahlah et al [31], who showed that most of the bulk-fill composites have a bottom-to-top surface ratio of 80% at the depth of 4mm. On the other hand, the results are contradictory with those of Garcia et al [32], who did not record any difference in the bottom-to-top surface hardness ratio of four types of bulk-fill and conventional composites. This may be due to the cumulative effect of light on the conventional composite layers, and the first layer of the bulk-fill composite. The deep layers of conventional composites receive extra light and experience greater polymerization [32]. Cornelio et al. [33] showed the positive effect of exposure time on DoC. Rueggeberg et al. [34] demonstrated that the LED intensity and the exposure time were the main effective factors to determine DoC for depths of more than 2mm. De Jong et al. [35] reported that the 10-second exposure time for each 2mm of the conventional composite provided sufficient hardness in small to medium class II restorations. In this study, the dimensions of the restoration were 6×5×4mm and the illuminating distance from the restoration surface was 1mm. Therefore, achieving a sufficient DC in deeper and wider posterior restorations will be more difficult [36]. We prepared a 4×6×8mm cavity, thus, the distance between the LED and gingival margin was 9mm. The results indicated that in the deep areas, the bottom-to-top surface hardness ratio did not reach 0.8 in any of the composites. Therefore, sufficient DC was not achieved at the depth of restorations. It is worth to mention that these results are related to two types of composites and cannot be generalized to other composites or darker shades of this composite. The DoC corresponding to 80% of the maximum hardness near the metal strip was 3mm and 4mm in the bulk-fill and conventional composites, respectively. This was in conflict with the recommended ISO standard values for these composites. It can be deduced from the results that the clinical and standard values of DoC are different depending on the composite and restoration type, the number of layers, and the exposure time. The reason for this may be attributed to the time of exposure of this layer (4mm) of the conventional composite. This layer is exposed to the cumulative exposure time of 20 seconds. The manufacturer claims that the exposure time of 10 seconds for the LED device with an output of more than 1200 mW/cm2 is sufficient for polymerization of composites. This exposure time leads to a reduction in the hardness, DoC and clinical DC, which is also expressed in previous studies [16,37]. This is more significant in the gingival floor of the restoration because of the increase of the distance from the LED and light loss due to transmission through the composite mass.

In all study groups, the values of hardness near the metal matrix strip were more than other points, which were statistically significant in (B) and (D1) surfaces (P=0.05 and P=0.04, respectively). However; it can affect the clinical DoC. These results coincide with the results of a study by Menees et al, [37] and contradict the study by Cornelio et al, [33] since they used a maximum exposure time (40 seconds), which may eliminate the effect of mold type on the DoC. Price et al. [38] demonstrated that DoC increased with increased values of the mold diameter, and the cure near the metal strip was less than the center of the mold, which was different from our findings. Comparison of the results shows that the positive effect of metal strips on hardness and DoC is more significant in critical conditions. Therefore, by increasing the distance from the LED, -either vertically (increasing the depth of a restoration) or horizontally (increasing the diameter of a restoration)- or using an opaque composite, the use of metal strips will have more advantages.

The hardness near the transparent strip was less than the opposite surface, which was an intact tooth (P>0.05); however, this was not statistically significant. It may depend on the IoR. The IoR of steel, cement, dentin, and enamel is 2.5, 1.58, 1.54, and 1.63, respectively [39,40]. The IoR of zirconia filler in bulk-fill composites, glass in conventional composites and air is 2.35, 1.5, and 1, respectively. Therefore, the light interactions are different in different materials, and the hardness may be reduced by decreasing IoR. Consequently, we recommend longer exposure time (two times the rate instructed by the manufacturer) to ensure complete curing at the depth of restorations, especially for bulk-fill composites.

CONCLUSION

The hardness of Filtek Z350 was greater than Gradia Posterior composite. The bottom-to-top surface hardness ratio did not reach more than 80% in any of the above-mentioned composites. The maximum bottom-to-top surface hardness ratio (73%) was found in the conventional composite in use of metal strip. The clinical DoC of the composites in class II restorations was different from the ISO standard.

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CONFLICT OF INTEREST STATEMENT

None declared.

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