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Research Article

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Physicochemical characterization of two bulk fill composites at different depths

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No potential conflict of interest relevant to this article was reported.

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ABSTRACT

Objectives: This study analyzed the physical-chemical behavior of 2 bulk fill resin composites (BFCs; Filtek Bulk Fill [FBF], and Tetric-N-Ceram Bulk Fill [TBF]) used in 2- and 4-mm increments and compared them with a conventional resin composite (Filtek Z250).
Materials and Methods: Flexural strength and elastic modulus were evaluated by using a 3-point bending test. Knoop hardness was measured at depth areas 0–1, 1–2, 2–3, and 3–4 mm. The translucency parameter was measured using an optical spectrophotometer. Real-time polymerization kinetics was analyzed using Fourier transform infrared spectroscopy.
Results: Flexural strength was similar among the materials, while TBF showed lower elastic modulus (Z250: 6.6 ± 1.3, FBF: 6.4 ± 0.9, TBF: 4.3 ± 1.3). The hardness of Z250 was similar only between 0–1 mm and 1–2 mm. Both BFCs had similar hardness until 2–3 mm, and showed significant decreases at 3–4 mm (FBF: 33.45 ± 1.95 at 0–1 mm to 23.19 ± 4.32 at 3–4 mm, TBF: 23.17 ± 2.51 at 0–1 mm to 15.11 ± 1.94 at 3–4 mm). The BFCs showed higher translucency than Z250. The polymerization kinetics of all the materials were similar at 2-mm increments. At 4-mm, only TBF had a similar degree of conversion compared with 2 mm.
Conclusions: The BFCs tested had similar performance compared to the conventional composite when used in up to 2-mm increments. When the increment was thicker, the BFCs were properly polymerized only up to 3 mm.

Keywords: Resin composite; Flexural strength; Hardness; Polymerization

INTRODUCTION

Resin composites are the most used direct restorative materials in the dental clinic daily [1]. Among the properties of resin composites, polymerization shrinkage is currently one of the most significant issues, which could lead to failures in the adhesive interface between resin composites and tooth structure [2,3]. In order to reduce the effect of the polymerization shrinkage stress, the incremental technique is used. However, this technique has disadvantages such as increased clinical time, the incorporation of air bubbles and

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the probabilities of making errors in the procedure [2,4,5]. Also, because a conventional resin composite does not polymerize correctly in thicknesses greater than 2 mm, and greater thickness would increase the contraction stress generated, it is established that the incremental technique is performed in a maximum of 2-mm increments in order to reduce the polymerization stress generated with the least amount of increments as possible [6,7].

Thus, new systems called bulk fill resin composites (BFCs) have currently been introduced to the market, and generated a paradigm shift by establishing their use in up to 4-mm increments [2,3,5]. These BFCs are presented with consistencies equal to conventional resin composites, and although they can be used in any cavity, they are mainly indicated in posterior restorations [4,6-8]. Despite this, it should be noted that in some cases a conventional resin composite surface layer is indicated, to achieve a better aesthetic result [9]. The BFCs composition is similar to conventional ones; however, each manufacturer adds some modifications to achieve adequate behavior up to 4 mm as modified monomers, flexible fillers or even photoinitiators to achieve correct polymerization and decrease the generated polymerization stress [4,10-12]. On the other hand, a lower amount of filler is reported compared to conventional resin composites, which would allow a better light passage at those depths [11].

BFCs would simplify the clinical steps of conventional resin composites such as the incremental technique, or air trapping between layers since they would be placed in a single increment [6,13]. Due to the paradigm shift, a debate arises as to whether these new generation materials actually manage to polymerize in 4 millimeters, maintaining the physical, chemical and biological characteristics of conventional resin composites [12,14]. Despite that there are several studies that characterize these kinds of resin composites, it is still unclear its behavior at several depths [15-17]. Therefore, the purpose of this work was to analyze the physical-chemical behavior of 2 commercial BFCs and compare them with a conventional resin composite. The null hypothesis to be tested was that the physicochemical properties of BFCs will be similar to conventional resin composite when evaluated in 2- or 4-mm thicknesses.

MATERIALS AND METHODS

Experimental design

This *in vitro* study investigated the physicochemical properties of 3 resin composites. The materials tested were: 2 BFCs (Filtek Bulk Fill [FBF], 3M ESPE, St. Paul, MN, USA; and Tetric-N-Ceram Bulk Fill [TBF], Ivoclar-Vivadent, Schaan, Liechtenstein) and one conventional resin composite (Filtek Z250, 3M ESPE). These materials were chosen since they are the first worldwide available and their use is widely spread among clinicians. The materials were manipulated according to the manufacturer's instructions and they were photoactivated using a LED curing unit (1,000 mW/cm^2; Radii Cal, SDI, Bayswater, Australia). Flexural properties were evaluated according to International Organization for Standardization (ISO) 4049. Knoop hardness measurements were performed at depth areas 0–1, 1–2, 2–3, and 3–4 mm from the top surface. The translucency parameter (TP) was measured using an optical spectrophotometer. The degree of conversion and polymerization rate were determined at 2- and 4-mm depths.

Flexural strength and elastic modulus

Bar-shaped specimens (2 × 2 × 25 mm; $n = 10$) were prepared by filling the uncured resin composite material into a stainless-steel mold placed on a glass slide covered by a Mylar strip

(DuPont, Wilmington, DE, USA) to avoid oxygen inhibition of polymerization. Samples were irradiated 3 times on both sides using the overlapping technique [18]. Each overlap was light-cured for 20 seconds. After polymerization, specimens were removed from the mold and flashes and irregularities were removed with 600-grit abrasive papers and their dimensions measured with a 0.01 mm accuracy using a digital caliper. The specimens were then stored in distilled water for 24 hours at 37°C prior to mechanical testing. The mechanical test was performed following ISO 4049 standard, using a universal testing machine (EMIC DL500, EMIC Co., Sao Paulo, Brazil) at a cross-head speed of 1 mm/s until fracture [18]. The flexural strength (σ_f) and elastic modulus (E_f) were determined using the equations provided by international standards and expressed in MPa and GPa respectively:

$$\sigma_f = 3Pl/2bh^2 \quad E_f = 3P1l^3/4bh^3d$$

where P is the load at the time of fracture (N), l is the distance between the supports (20 mm), b is the width (mm) and h is the height of the specimen (mm), $P1$ is the maximum load on the linear portion (proportional limit) of the stress-deformation trace, and d is the deflection of the specimen at load $P1$.

Knoop hardness number

Specimens for hardness determination ($n = 5$) were created using custom-made stainless-steel mold with a semicircular notch measuring 5 mm in length and 2 mm in diameter as described previously [19]. Briefly, the semicircular notch was then filled with each one of the resin composites. Then, the mold was covered with a Mylar strip and covered by a stainless-steel plate. The resin composite was then light-cured through the surface opening for 20 seconds. After light-polymerizing, the plate and Mylar strip were removed and the mold including the resin composite specimen was placed under a microhardness tester (FM 700, Future-Tech Corp., Kawasaki, Japan). Following, hardness measurements (kgf/mm^2) were made on the specimens at defined areas from the top surface (0–1 mm, 1–2 mm, 2–3 mm, and 3–4 mm) with a Knoop indenter using a 25-gram load applied for 10 seconds (**Figure 1**). On each area,

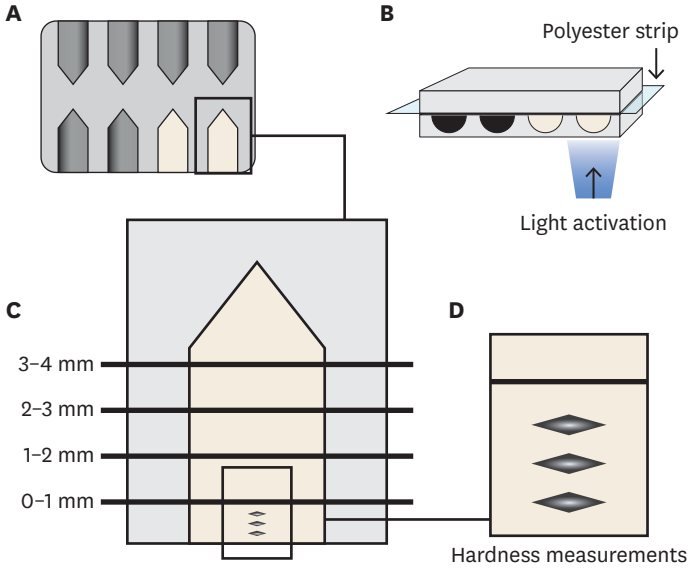

Figure 1. Illustration for hardness measurements at different depths of the specimen. (A) Upper view of the inferior part of the mold. (B) Lateral view of mold and photoactivation direction. (C) Division of the specimen in 4-mm layers for the hardness test. (D) Indentation performed at the 0–1 mm area.

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3 subsequent indentations were done, and the mean value was registered. Hardness loss (%HL) for each depth was calculated in percentage using the following formula: $\%HL_x = (KHN_x - KHN_{0-1}) \times 100/KHN_{0-1}$. Where KHN_x is the Knoop hardness number at 1–2, 2–3, or 3–4 mm and KHN_{0-1} is the Knoop hardness at 0–1 mm.

Translucency parameter

The TP of each resin composite was measured using an optical spectrophotometer (SP60, X-Rite, Grand Rapids, MI, USA). For this, the cylindrical specimens (diameter 7 mm, thickness 1 mm; $n = 5$) were obtained by filling the uncured material into silicon molds and polymerized for 20 seconds. The CIE L*a*b* color coordinates were measured after storing the specimens in distilled water at 37°C. Color readings were taken over white (L* = 93.07, a* = 1.28, b* = 5.25) and black (L* = 27.94, a* = 0.01, b* = 0.03) backgrounds. The TP for each specimen was calculated using the formula:

$$TP = [(L^*w - L^*b)^2 + (a^*w - a^*b)^2 + (b^*w - b^*b)^2]^{1/2}$$

where w and b refer to the color coordinates measured on the white and black backgrounds [20].

Polymerization kinetics

An infrared spectrophotometer equipped with an attenuated total reflection (ATR) cell unit (Prestige-21, Shimadzu, Kyoto, Japan) was used to measure the polymerization rate and the degree of carbon double bonds conversion. A small specimen (up to 100 mg; $n = 3$) of each material was placed into a cylindrical mold (5-mm diameter, and 2- or 4-mm height) on the ATR unit diamond cell, then a celluloid sheet and a slide glass were placed, standardizing the material thickness. The IR solution spectrophotometer software package (v. 1.60, Shimadzu) was used in the monitoring scan mode in the range of 1,500–1,800 cm^{-1}, a resolution of 4 cm^{-1} and a mirror speed of 2.8 mm/s. With this configuration, 1 scan was acquired every second during 40 seconds of photoactivation. The experiment was performed 3 times for each of the evaluated groups. In each of the spectra, the absorption band height of the aliphatic uC=C bond was measured at 1,638 cm^{-1} as well as the absorption band height of the aromatic uC=C bond located at 1,609 cm^{-1}. The degree of C=C conversion of the materials was calculated using the following equation [21]:

$$\text{Degree of C} = \text{C conversion (\%)} = 100\left[1 - \left(\frac{A_{1638}}{A_{1609}}\right)\text{polymer} \Big/ \left(\frac{A_{1638}}{A_{1609}}\right)\text{monomer}\right]$$

where A_{1638} is the maximum band height at 1,638 cm^{-1} and A_{1609} is the maximum band height at 1,609 cm^{-1}. The degree of double bond conversion vs. polymerization reaction time data was plotted and Hill's 1 3-parameter nonlinear regression was performed for curve fitting. Using these data, the polymerization rate (R_P) was calculated as the degree of conversion at time t subtracted from the degree of conversion at time t–1 [22].

Statistical analysis

Statistical analysis was performed using Sigma Plot 14.0 software (Systat Software, Inc., San Jose, CA, USA). Data from flexural strength, elastic modulus, Knoop hardness number, TP, and degree of conversion were analyzed to verify the normal distribution and homogeneity of variance. A 1-way analysis of variance (ANOVA) followed by Tukey's *post hoc* test was used to compare data from flexural strength, elastic modulus, hardness, and TP. The degree of conversion was analyzed using a 2-way ANOVA (material and depth factors) followed by Tukey's *post hoc* test. In all cases, the significance level was set at $\alpha = 0.05$.

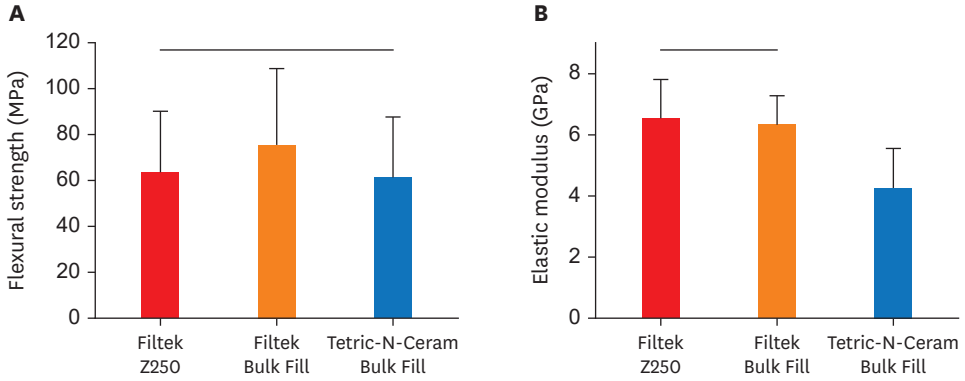

Figure 2. Means and standard deviations for flexural strength (A) and elastic modulus (B). Columns under the same horizontal line indicate no statistical difference ($p > 0.05$).

RESULTS

Figure 2 shows the results of flexural strength and elastic modulus. For flexural strength, there were no statistically significant differences among the evaluated groups ($p = 0.636$). When analyzing the elastic modulus, the Z250 and FBF resin composites did not show statistically significant differences between them ($p = 0.939$), while TBF presented a significantly lower elastic modulus ($p < 0.05$).

The results of the Knoop hardness test in depth can be seen in **Figure 3**. Each material was analyzed independently. For Z250, no statistically significant differences were detected only for the comparison between areas 0–1 and 1–2 mm ($p = 0.184$), being that the results of areas 2–3 and 3–4 mm decreased significantly ($p < 0.05$). For FBF, the results show that the Knoop hardness remained similar up to area 2–3 mm depth ($p > 0.05$), decreasing significantly at area 3–4 mm ($p < 0.05$). Regarding the results obtained for TBF, only statistically significant differences were detected for comparisons between areas 0–1 and 3–4 mm ($p < 0.01$), and between areas 1–2 and 3–4 mm ($p = 0.002$).

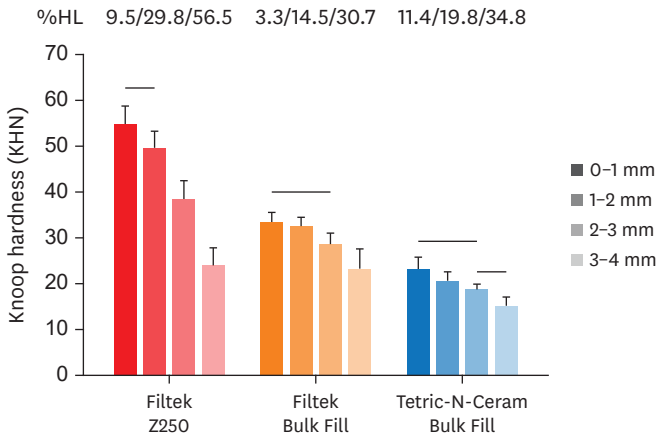

Figure 3. Means and standard deviations for Knoop hardness number (kgf/mm²). For each material, columns under the same horizontal line indicate no statistical difference ($p > 0.05$). Hardness loss (%HL) for each material is shown within the rectangles ($\%HL_2/\%HL_3/\%HL_4$).

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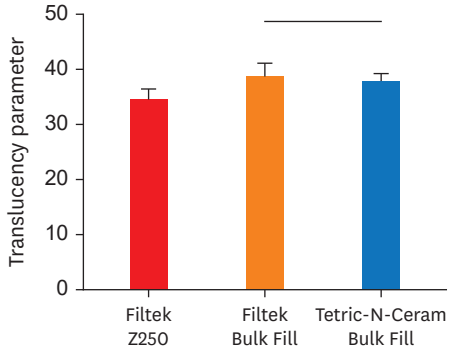

Figure 4. Means and standard deviations for translucency parameter. Columns under the same horizontal line indicate no statistical difference ($p > 0.05$).

The %HL analysis shows that for FBF, the hardness from areas 0–1 to 1–2 and from areas 0–1 to 3–4 mm was reduced by 3% and 31% respectively; for TBF, the reduction from area 0–1 to 1–2 mm was 11%, while from area 0–1 to 3–4 mm, the reduction was 35%. Finally, it was observed that the conventional resin composite Z250 reduced its hardness from area 0–1 to 1–2 and from area 0–1 to 3–4 mm by 9.5% and 56.5%, respectively.

For the TP, the Z250 material obtained significantly lower values than BFCs ($p < 0.05$), as shown in **Figure 4**. On the other hand, the comparison between both BFCs showed the absence of statistically significant differences ($p = 0.735$). The degree of conversion and the polymerization rate are shown in **Figure 5**. The degree of conversion was significantly influenced by depth ($p < 0.001$), material ($p < 0.001$) and the interaction of both ($p < 0.001$). Considering the depth factor, for Z250 and FBF materials, the degree of conversion decreased significantly between 2- and 4-mm depths ($p < 0.001$). On the other hand, for TBF, no statistically significant differences were detected between 2- and 4-mm depths ($p = 0.077$). Analyzing the material factor, none of the evaluated resin composites had statistically significant differences at 2-mm depth ($p > 0.05$), while at 4-mm depth, the Z250 resin composite obtained significantly lower values ($p < 0.001$). Regarding the polymerization rate, when evaluated at 2-mm depth, both BFC achieved higher polymerization rate max values (Z250: 0.12; FBF: 0.25; TBF: 0.26). While, when evaluated at 4-mm depth, a drastic decrease in R_p is observed for all the materials (Z250: 0.01; FBF: 0.07; TBF: 0.11).

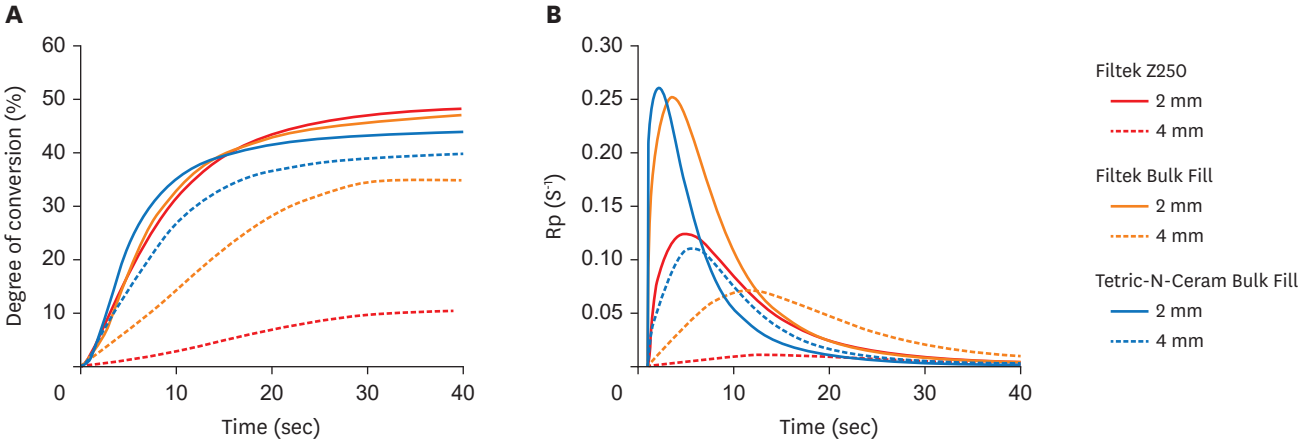

Figure 5. Data-fitted plots of polymerization kinetics of the resin composites evaluated. Degree of C=C conversion as a function of time shows the maximum conversion achieved at 2- and 4-mm depth (A). Polymerization rate (R_p) profiles (B) as a function of time for 2- and 4-mm increments.

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DISCUSSION

In this study, the physicochemical properties of 2 BFCs and one conventional resin composite were evaluated. The results showed that the properties evaluated mainly depended on the depth used. When analyzing the behavior of the material, it was observed that hardness and polymerization kinetics values drastically decreased at 4 mm when compared to those obtained at 2 mm. The main findings observed in this study help to demonstrate the influence of the BFCs composition and its relation with the physical and chemical properties. Higher BFCs translucency can be associated with the maintenance of the polymerization kinetic parameters. Also, the lowest DC observed at 4-mm depth, could be associated with a lower Knoop hardness at 4-mm depth, as observed here. Considering this, the null hypothesis tested in this study is rejected.

When analyzing flexural strength, all the resin composites seem to have a statistically similar value. At this point, it is important to highlight that the ISO 4049 standard establishes only the mechanical analysis with 2 mm thick specimens, so this analysis does not directly compare the material behavior when used in different thicknesses, but it was carried out to understand the general mechanical behavior of these materials compared to a conventional resin composite. In the literature, this controversy regarding the current standard to analyze resinous type materials has been discussed [23].

When the elastic modulus is analyzed, FBF seems to be the resin composite with the highest rigidity, with statistically similar values to Z250. This could be related to the fact that both materials had a similar amount of filler content [24]. On the other hand, TBF showed statistically lower values, which can be interpreted as greater material flexibility. This behavior could be explained by several reasons, first, the filler content of this material is lower than the other resin composites evaluated. Also, when the composition of this material was analyzed, it was observed that the manufacturer includes an elastic filler, whose function is to dissipate the stresses caused by polymerization [25]. The addition of this filler could be responsible for the decrease in the elastic modulus, even though it presents a greater amount of filler in its composition compared to Z250 or FBF. On the other hand, the addition of specific monomers, of high molecular weight or greater flexibility could be modifying the obtained values [26]. The results regarding flexural strength and elastic modulus obtained in this study are in agreement with previous findings [27,28].

Regarding the hardness analysis, it could be observed that Z250 conventional resin composite showed higher hardness at the 0–1 mm area than BFCs. These results are in accordance with previous findings, where it was found that conventional resin composites had significantly higher hardness values than several BFCs [29,30]. These results could be related to the presence of aluminum oxide in the Z250 material, also the differences in the organic matrix among the materials, such as the presence of elastic monomers within the BFCs composition, could also have an influence [31,32]. Despite the differences obtained, the hardness in BFCs does not present great variations up to the area of 2–3 mm depth, being that at the area of 3–4 mm a statistically significant decrease was observed in both BFC's ($p < 0.05$). When analyzing the variation in the decrease in hardness in each millimeter (%HLx) it is possible to see that, up to the area of 2–3 mm deep, the hardness decreases less than 20%, this corresponds to what was reported by Van Ende *et al.* [33] and establishes that the depth to which the material maintains 80% of the surface hardness is acceptable. Whereas the conventional resin composite meets this parameter of 80% of hardness, only up to 2-mm thickness as expected.

This corroborates the inability of conventional resin composites to be used at depths greater than 2 mm. When reaching the area of 3–4 mm depth, the hardness in all materials falls more than 30%, which could suggest a clinically relevant loss of mechanical properties, related to the polymerization quality obtained at that depth.

Flury *et al.* [23] related hardness and depth of polymerization in BFCs, confirming that the method proposed by ISO4049 for the curing depth analysis in this type of material generates an overestimation of the results. This is important since the properties within the resin composite decrease with increasing depth, resulting in a non-homogeneous material, which can render less predictable outcomes [33]. In this line of reasoning, it was possible to highlight that several modifications to the ISO 4049 standard may be necessary for the BFCs analysis, since, as previously discussed, it does not contemplate such behavior for flexural resistance either.

On the other hand, although hardness is not a parameter established by current ISO regulations, it is possible to see how conventional Z250 resin composite has a higher hardness in the first 2 mm compared to BFCs. This behavior was already described before and it was correlated with the differences in filler composition, size and loading of the BFCs [34]. This could explain why several manufacturers indicate the coating of this type of material with conventional resin composites, since lower hardness values are related to greater wear and loss of the material surface polishing [35]. Despite this, it should be highlighted that a recent systematic review with meta-analysis demonstrated that BFCs show a lower hardness when compared to conventional composites at 2-mm depth, but no significant differences were observed in the survival rate [34].

Regarding the TP, it was possible to determine that both BFCs presented significantly higher values than the conventional resin composite. These results are in agreement with those previously reported by Bucuta *et al.* [36]. The increase in BFCs translucency can be explained by several factors, among which it is possible to find the use of formulations with a lesser amount of inorganic filler, which is consistent with the found hardness results, as well as the use of fillers that, due to their composition, have a better refractive index matching with the organic matrix, which ends up favoring the light penetration through the material, and therefore, the maintenance of their properties after a certain depth [37-39]. This increase in light penetration through the material could be the reason why BFCs had the least decrease in Knoop hardness between the surface (area 0–1 mm) and the bottom (area 3–4 mm) of the specimens. In fact, it has been found that the polymerization depth of photoactivated materials is limited by the attenuation of the emitted energy by the light-emitting apparatus through the material, and that this is inversely proportional to the material translucency [33].

In this study, the degree of conversion and polymerization rate, at depths of 2 and 4 mm, were also evaluated. As expected, the degree of conversion values of the materials measured at a depth of 2 mm were similar. These findings could be confirmed by previous research by Garoushi *et al.* [40], where the degree of conversion was significantly influenced by thickness. The degree of conversion is a property that depends on intrinsic and extrinsic factors, among the intrinsic the photoinitiator system, the type of monomer system and the amount and type of inorganic filler are found. On the other hand, among the extrinsic factors, it is possible to find those variables related to the light emission source that triggers the polymerization process [41]. Considering this, it is probable that, at a depth of 2 mm, all the materials evaluated in this study have the same capacity to transmit the light emitted by the photopolymerizing apparatus, resulting in an adequate material polymerization [36].

When analyzing the polymerization rate at 2 mm, the BFCs had a considerable increase in R_p compared to conventional resin composite. This increase is likely to have occurred due to the presence of special, stress-relieving monomers in the BFCs formulation, as well as the use of alternative initiators such as Ivocerin (Ivoclar-Vivadent) [42,43]. The presence of stress-relief monomers within the BFCs composition has been proved to increase the propagation of the polymerization reaction during the auto acceleration stage [44]. On the other hand, previous studies have shown that Ivocerin acts as a polymerization booster in BFCs, allowing efficient polymerization at the depth of 4 mm [45,46].

On the other hand, when analyzing the polymerization kinetics parameters at a depth of 4 mm, it is possible to observe that the conventional resin composite had an unsatisfactory performance. This effect can be explained by the impossibility that the light emitted by the photopolymerizing device has to completely cross the 4 mm depth in which the material was evaluated. It has been previously determined that as the thickness of the resin composite material increases, there is a decrease in light transmission, resulting in a decrease in the degree of conversion [47,48]. In fact, this is the explanation why conventional resin composite manufacturers recommend that increments do not exceed 2 mm. In the case of BFCs, the values obtained at 4 mm are due, in part, to an increase in the material translucency [36]. On the other hand, for the TBF material, it is possible that the use of Ivocerin as a photoinitiator has also had some influence since it has proven to be more effective than camphorquinone for radical methacrylates polymerization [49].

CONCLUSIONS

The BFCs tested had similar performance compared to the conventional composite when used in up to 2-mm increments. When the increment was thicker, the bulk fill composites were properly polymerized only up to 3 mm. It seems that the use of increments thicker than 3 mm should be carried out with caution in the clinical setting.

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