



Mechanical properties of bulk-fill versus nanohybrid composites: effect of layer thickness and application protocols

Propriedades mecânicas de compósitos de resina bulk versus nano-híbrida: efeito da espessura da camada e protocolos de aplicação

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ABSTRACT

Objective: The objective of this study was to evaluate the compressive strength, flexural strength and flexural modulus of high-viscosity, low-viscosity bulk-fill, and conventional nano-hybrid resin composite materials alone and when covered with nano-hybrid resin composite at different incremental thicknesses on the bulk-fill composites. **Material and Methods:** Specimens (N=60) were fabricated from the following materials or their combinations (n=10 per group): a) conventional nano-hybrid composite Z550 (FK), b) high-viscosity bulk-fill composite (Tetric N Ceram-TBF), c) low-viscosity bulk-fill composite SDR (SDR), d) Sonicfill (SF), e) SDR (2 mm)+FK (2 mm), f) SDR (4 mm)+FK (4 mm). After 24 h water storage, compressive strength was measured in a universal testing machine (1 mm/min). Additional specimens (N=40) (25x2x2 mm³) were made from FK, TBF, SDR and SF in order to determine the flexural strength and the flexural modulus, (n=10) and subjected to three-point bending test (0.5 mm/min). Data were analyzed using one-way ANOVA and Tamhane's T2 post-hoc tests (p<0.05). **Results:** The mean compressive strength (MPa) of the nano-hybrid composite (FK) was significantly higher (223.8±41.3) than those of the other groups (123±27 - 170±24) (p<0.001). SDR (4 mm)+FK (2 mm) showed significantly higher compressive strength than when covered with 4 mm (143±30) or when used alone (146±11) (p<0.05). The mean flexural

RESUMO

Objetivo: O objetivo deste estudo foi avaliar a resistência à compressão, resistência à flexão e módulo de flexão de materiais compósitos de alta viscosidade, baixa viscosidade e compósitos nano-híbridos convencionais e quando cobertos com resina composta nano-híbrida em diferentes espessuras incrementais sobre os compósitos de resina tipo bulk-fill. **Material e Métodos:** Os espécimes (N = 60) foram fabricados a partir dos seguintes materiais ou suas combinações (n = 10 por grupo): a) compósito nano-híbrido convencional Z550 (FK), b) compósito de bulk-fill de alta viscosidade (Tetric N Ceram-TBF), c) compósito SDR (SDR) de bulk-fill de baixa viscosidade, d) Sonicfill (SF), e) SDR (2 mm) + FK (2 mm), f) SDR (4 mm) + FK (4 mm). Após 24 h de armazenamento em água, a resistência à compressão foi medida em uma máquina universal de ensaios (1 mm / min). Espécimes adicionais (N = 40) (25x2x2 mm³) foram confeccionados com FK, TBF, SDR e SF para determinação da resistência à flexão e do módulo de flexão, (n = 10) e submetidos ao teste de flexão de três pontos (0,5 mm / min). Os dados foram analisados utilizando one-way ANOVA e testes post-hoc T2 de Tamhane (p <0,05). **Resultados:** A resistência média à compressão (MPa) do compósito nano-híbrido (FK) foi significativamente maior (223,8 ± 41,3) que os demais grupos (123 ± 27 - 170 ± 24) (p <0,001). SDR (4 mm) + FK (2 mm) apresentou resistência à compressão significativamente maior do que quando coberta com 4 mm (143 ± 30) ou quando usada sozinha (146 ± 11) (p <0,05). A resistência à flexão média (159 ±

strength (159 ± 31) and the flexural modulus of FK (34 ± 7) was significantly higher than that of the high- or low-viscosity bulk-fill composites ($p < 0.001$). The mean flexural strength of SF (132 ± 20) was significantly higher compared to TBF (95 ± 25) ($p < 0.05$). **Conclusion:** Bulk-fill resin composites demonstrated poorer mechanical properties compared to nano-hybrid composite but similar to that of SF. Increasing the thickness of low-viscosity bulk-fill composite (SDR) from 2 to 4 mm underneath the nano-hybrid composite (FK) can improve the mechanical properties of the bulk-fill composites.

KEYWORDS

Bulk-fill composites; Compressive strength; Flexural modulus; Flexural strength; Mechanical properties.

31) e o módulo de flexão de FK (34 ± 7) foram significativamente maiores do que os compósitos do tipo bulk-fill de alta ou baixa viscosidade ($p < 0,001$). A resistência à flexão média do FS (132 ± 20) foi significativamente maior em comparação ao TBF (95 ± 25) ($p < 0,05$). **Conclusão:** Os compósitos de resina do tipo bulk-fill demonstraram propriedades mecânicas mais insatisfatórias em comparação com o compósito nano-híbrido, mas semelhantes aos do SF. O aumento da espessura do composto de bulk-fill de baixa viscosidade (SDR) de 2 a 4 mm sob o compósito nano-híbrido (FK) pode melhorar as propriedades mecânicas dos compósitos de bulk-fill.

PALAVRAS-CHAVE

Compósitos de bulk-fill; Força compressiva; Módulo Flexural; Resistência à flexão; Propriedades mecânicas.

INTRODUCTION

Tooth-colored restorative materials have been developed to meet the aesthetic demands of patients. Although high-quality aesthetic results could be achieved with resin composite materials, several limitations such as polymerization shrinkage, microleakage, secondary caries, post-operative sensitivity, and debonding of the adhesive surfaces are still considered as challenges in restorative dentistry [1]. Layering techniques for resin-based composites is one way to tackle polymerization shrinkage [2-4]. However, restoring deep cavities using the incremental technique is time consuming, and has the risk of contamination and formation of air bubbles between the increments [5,6].

As a result of the recent advances in material science research, a new category of resin composites called “bulk-fill flowable composites” has been introduced in dentistry. Such composites are available in low-viscosity (flowable) or high-viscosity where the latter is applied in bulk of 4 or 5 mm thick, depending on the manufacturer’s instructions. Application of the material in bulk simplifies clinical procedures and decreases the

chairside time [5,7-10]. However, low-viscosity bulk-fill composites require the placement of a final composite layer over the 4-mm thick bulk layer owing to their low surface hardness and elasticity modulus [5]. In contrast, high-viscosity bulk-fill resin composites can be used without veneering in a single step.

The increased depth of polymerization of bulk-fill composites is a result of both higher translucency of composites and the developments in the filler contents along with the organic matrix [11,12]. Low-viscosity bulk-fill composites have in fact lower filler content [2-6,8-13]. And thereby lower elasticity modulus compared to hybrid composites [11,14]. Although a reduction in the filler content decreases the hardness, the recommended polymerization time remains the same, namely the same duration of polymerization is sufficient in order to double the polymerized thickness of the layer [2]. The presence of glass microfibers in the bulk-fill composite may account for the improvements in the elastic modulus, flexural strength, and fracture toughness [11]. In addition to the effect of filler amount, the translucency of the material is influenced by the difference in refractive indices between filler

particles and resin matrix [15]. Innovations in monomer chemistry, filler characteristics, and polymerization kinetics, have enabled the development of materials characterized by low levels of shrinkage while polymerization, allowing the composite materials to be placed in bulk into the cavities [16,17].

Restorative materials are subject to both compressive and flexural forces during chewing. Compressive strength determines the resistance of a restorative material to the longitudinal heavy load during mastication [18]. Although mechanical properties of bulk-fill composites have been evaluated for their mechanical properties, the effect of increment thickness of low-viscosity bulk-fill composite when used under conventional hybrid composites on compression strength is not known.

The objective of this study was to evaluate the compressive strength, flexural strength and flexural modulus of high-viscosity, low-viscosity bulk-fill, and conventional nano-hybrid resin composite materials alone and when covered with nano-hybrid resin composite at different incremental thicknesses on the bulk-fill composites. The null hypothesis tested was that increasing the low-viscosity bulk-fill composite thickness would not affect the mechanical properties of the bulk-fill- resin composite assembly.

MATERIAL AND METHODS

Specimen preparation

Types and chemical compositions of the materials used in this in-vitro study are listed in Table 1.

Specimens (N=60) were fabricated from the following materials or their combinations (n=10 per group): a) conventional nano-hybrid composite Z550 (FK, 3M ESPE, St. Paul, MN, USA), b) high-viscosity bulk-fill composite (Tetric N Ceram-TBF, Ivoclar Vivadent, Schaan Liechtenstein), c) low-viscosity bulk-fill composite SDR (SDR, Dentsply, Konstanz, Germany), d) Sonicfill (SF, Kerr, Orange, CA, USA), e) SDR (2 mm)+FK (2 mm), f) SDR (4 mm)+FK (4 mm).

Table 1 - Mechanical properties of the materials used in the numerical simulations

Brand	Type	Manufacturer	Filler	Filler content (wt%)	Resin matrix	Manufacturer's recommendation
Tetric N-Ceram Bulk Fill	Bulk fill Hybrid composite	Ivoclar Vivadent, Schaan, Liechtenstein	Ba-Al-Si glass, pre-polymerized filler (monomer, glass filler, and ytterbium fluoride), spherical mixed oxide	75-77	bis-GMA, UDMA	Up to 4 mm bulk-filling without capping
Filtek Z550	Nano-hybrid composite	3M ESPE, St Paul, USA	Surface-modified zirconia/silica	82	bis-GMA, UDMA, bis-EMA, PEGDMA TEGDMA	2 mm incremental filling
Sonicfill	Sonic-activated, bulk-fill composite Nano-hybrid	Kerr, Orange, CA, USA	Glass, oxide, chemicals, SiO ₂	83.5	BisGMA EBADMA, TEGDMA	Up to 5 mm bulk-filling without capping
Su-refill SDR	Posterior bulk-fill flowable base	Dentsply Caulk, Milford, DE, USA	Ba-Al-F-B-Si-glass and Str-Al-F-Si-glass as fillers	68	Modified UDMA, TEGDMA, EBADMA	Up to 4 mm bulk-filling with a capping layer

Compressive strength test

For the compressive strength tests, resin composite materials were placed in a cylindrical teflon mold (height: 6 mm; diameter: 3 mm) (n=10). The test groups were as follows:

TBF group: TBF was placed in the teflon mould 4 mm bulk and photo-polymerized. Then 2 mm TBF was placed and photo-polymerized.

FK group: FK was placed in the teflon mould in 2 mm increments up to 6 mm and each layer was photo-polymerized.

SF group: SF was inserted 4 mm, photo-polymerized and then another increment of 2 mm was applied and polymerized.

SDR group: SDR was inserted 4 mm increment, photo-polymerized and subsequently another increment of 2 mm was applied and polymerized.

SDR+FK1 group: SDR was inserted 4 mm increment and photo-polymerized. Then FK was placed 2 mm increment and polymerized.

SDR+FK2 group: SDR was inserted 2 mm increment and photo-polymerized. Then, 2 mm FK was applied and photo-polymerized.

In total, 60 specimens of resin composites were applied and packed inside the teflon mold as described above and each increment was photo-polymerized for 20 s (Hilux 200, Benlioğlu Dental, Ankara, Turkey). After the polymerization process, the specimens were stored in distilled water at 37°C for 24 h. Compressive tests were performed using the Universal Testing Machine (Shimadzu AG-5 KN; Shimadzu Corp, Tokyo, Japan) at a crosshead speed of 1 mm/min.

Flexural strength and the flexural modulus

The three-point bending test was performed in order to determine the flexural properties (flexural strength- FS, and flexural modulus-FM) of each resin composite, namely TBF, FK, SF, SDR (N=40, n=10 per group). The specimens were prepared in accordance with the ISO 4049 guidelines. A metal mold (25x2x2 mm³) was filled with the resin on a glass slab and photo-polymerized for 20 s (Hilux 200) at an output of 600 mw/cm². After polymerization, the resin was removed from the mould and stored in distilled water at 37°C for 24 h. FS and FM were measured using the Universal Testing Machine (Shimadzu AG-5 KN) at a crosshead speed of 0.5 mm /min.

Statistical analysis

Statistical analysis was performed using IBM SPSS Statistics 22 (SPSS Inc., Chicago, IL, USA). The data were analyzed using the Kolmogorov-Smirnov test for normal distribution. As the data obtained were normally distributed, statistical analysis was performed using one-way ANOVA. Tamhane's T2 test was used as post-hoc test at a significance level of $p < 0.05$.

RESULTS

The mean compressive strength was significantly different between the groups ($p=0.001$). Compressive strength of the FK group was significantly higher than that of the TBF ($p=0.001$), SDR ($p=0.002$), SDR + FK2 ($p=0.002$), SDR+FK1 ($p=0.042$), and the SF groups ($p=0.034$) (Tamhane's T2 post-hoc test) (Table 2). The compressive strength of the SDR + FK1 group was significantly higher than that of the TBF group ($p = 0.011$; $p < 0.05$). There were no significant differences among the other groups ($p > 0.05$) (Figure 1).

The mean flexural strength and flexural modulus were significant between the groups ($p=0.001$) (Table 3). The mean flexural strength and flexural modulus of the FK group were significantly higher than those of the TBF ($p=0.001$) and the SDR groups ($p=0.017$) (Tamhane's T2 post-hoc test) while SF group showed significantly higher mean values compared to TBF group ($p=0.012$). There were no significant differences between the other groups ($p > 0.05$) (Figure 2).

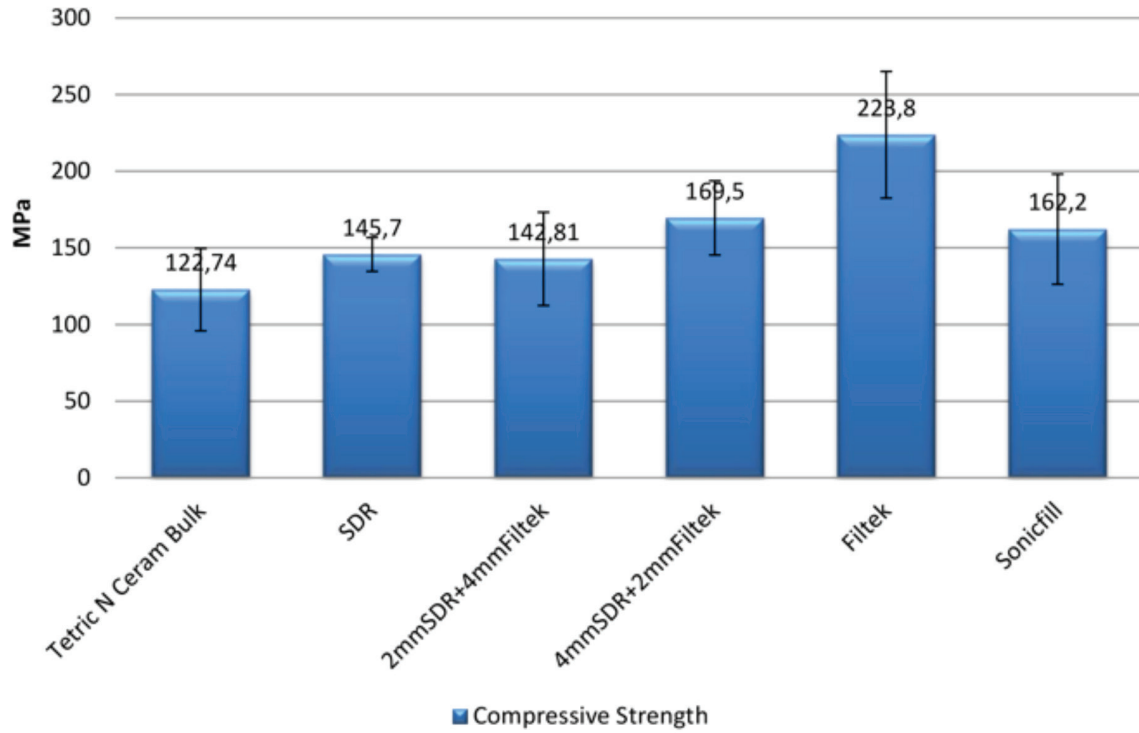


Figure 1 - Compressive strength of the resin based materials tested.

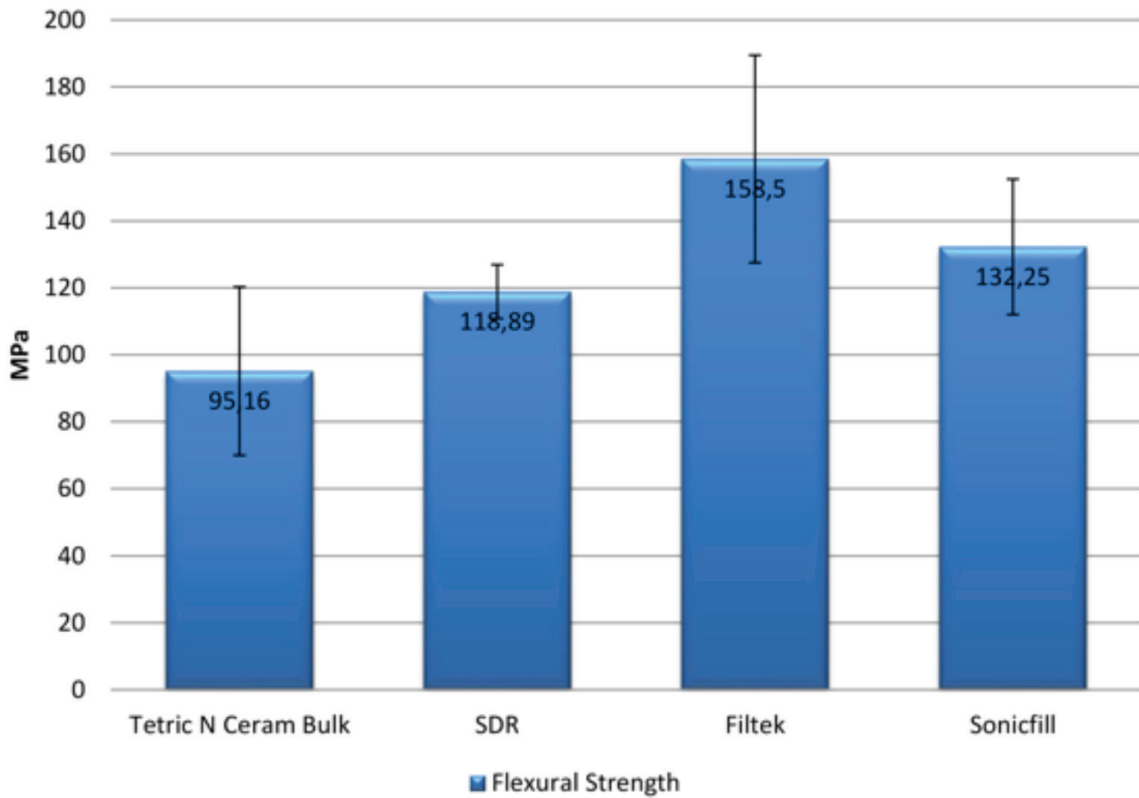


Figure 2 - Flexural strength of the resin-based materials tested.

Table 2 - Mean compressive strength and standard deviations of the materials and their combinations tested. Different superscript letters in the column indicate significant differences between groups (One-way ANOVA, ** $p < 0.01$)

	Compressive Strength (MPa)
	Mean±SD
Tetric N Ceram Bulk	122.7±26.9 ^{ac}
SDR	2 mm SDR+4 mm Filtek Z550
2 mm SDR04 mm Filtek Z550	142.8±30.4 ^a
4 mm SDR02 mm Filtek Z550	169.5±24.2 ^{ac}
Filtek Z550	223.8±41.3 ^b
Sonicfill	162.2±36 ^a
<i>P</i>	0.001**

Table 3 - Mean flexural strength, flexural modulus and standard deviations of the materials tested. Different superscript letters in the column indicate significant differences between groups (One-way ANOVA, ** $p < 0.01$) (One-way ANOVA, ** $p < 0.01$)

	Flexural Strength (MPa)	Flexural Modulus (GPa)
	Mean±SD	Mean±SD
Tetric N Ceram Bulk	95.7±25.2 ^{ac}	20.3±5.4 ^{ac}
SDR	118.9±8 ^a	25.4±1.7 ^a
Filtek Z550	158.5±30.9 ^b	33.8±6.6 ^b
Sonicfill	132.3±20.2 ^d	28.2±4.3 ^d
<i>P</i>	0.001**	0.001**

DISCUSSION

This study was undertaken in order to evaluate the mechanical properties of high-viscosity, low-viscosity bulk-fill, and conventional nano-hybrid resin composite materials alone and when covered with nano-hybrid resin composite at different incremental thicknesses on the bulk-fill composites. Based on the results of this study, since there were significant differences in compressive strength and the flexural strength between the groups, the null hypothesis could be rejected.

Resin composite materials have undergone huge improvements during the last decade [7,18]. When used 4 to 5 mm increments, the mechanical properties of bulk-fill composites such as flexural strength, flexural modulus, and fracture toughness, were significantly affected by the filler morphology and amount [13,19]. Although bulk-filling is ideal for the posterior

region, it has to be noted that this area is a high-stress bearing area of the mouth. Thus, bulk-fill composites should have the appropriate mechanical properties for their indication in the posterior. In general, filler volume is positively correlated with several properties of the resin composite materials, including the elastic modulus, strength and hardness whereas flowable bulk-fill composites generally have poor mechanical properties. The observed viscosity of the bulk-fill composites may vary for the same filler content as a result of the variations in the resin matrix viscosity and the relative concentrations of the different monomers that constitute the material [20].

Despite the increasing use of bulk-fill composites in restorative dentistry, studies are lacking regarding their compressive strength and increment thickness. While compressive tests are easy to perform, the results are complicated to interpret. For example, composite resins can suffer a barrel effect during a compressive test and expand until the plastic deformation, which can lead to misleadingly high values [19,21]. In the present study, compressive strength of low-viscosity bulk-fill composites varying between 2 to 4 mm in thickness, placed using either incremental or bulk-filling techniques, underneath conventional nano-hybrid composites, was evaluated. In addition, the flexural properties of all of these restorative materials were also tested.

Typically, compressive strength of low-shrinkage composites show values are dependent on the filler content to some extent [18]. The weak correlation may reflect a contribution of factors, such as the relative proportion of monomers and the degree of crosslinking [18]. The reduction in the size and the increase in the volume of fillers are directly proportional to the increase in compressive strength of a material [22]. Consequently, nano-composites have shown better compressive strength than micro-hybrid composites [23]. In contrast, results from another study have indicated that hybrid composites have a higher compressive strength compared to nano-composites, probably owing

to their different compositions [24]. The hybrid composite used in that study presented large size filler particles (zirconium fillers) improving the strength of the composite material [24]. In this study, the mean compressive strength of the FK group was higher than those of the other groups. Moreover, filler content of FK (82 wt%) was one of the highest among the groups tested, being slightly less than that of SF (83.5 wt%). These results indicate that the compressive properties improved upon applying bulk flow composites to a height of 4 mm under the conventional hybrid composites. The compressive strength of the SDR+FK1 group was the highest among the remaining groups; however, it was significantly different only from the TBF group. The higher compressive strength of this group may be due to the 4 mm placement of SDR.

The flexural strength of a material reflects its resistance to compressive and tension-related stresses. Hence, ISO norm was chosen as a screening parameter for the mechanical properties of resin-based materials [19,25,26]. The flexural strength of the bulk-fill and conventional composites have been investigated in several other studies [11,13,16,25,27,28]. Likewise, the filler content was closely related to the flexural strength and flexural modulus. On the contrary, Park et al. found a weak correlation between the filler content (vol% and wt%) and FS [18]. The authors attributed the weak correlation to the volume of the material and possible the internal defects (cracks or voids) generated during the manufacturing process [18]. Flexural strength may also be influenced by both the stress transfer between filler particles and the matrix, and the adhesion between them [16]. In this study, the SF group showed higher FS values than the TBF group. This finding can be explained by different techniques for the placement of the composite materials into the cavity.

Compared to the hybrid composites, nano-composites are characterized by an increased filler volume, increasing their mechanical properties [29]. Filler morphology and filler content influence the flexural strength and the

flexural modulus [19] where higher the filler content, greatly increases the flexural strength [11,25]. In the present study, the flexural strength of the FK group was higher than that of the other groups, except for the SF group. This is probably due to the fact that they are both nano-composites and have higher filler content than other materials tested. SF also showed significantly higher FS values compared to the other high-viscosity bulk-fill composite, TBF. These differences may be attributed to the fact that SF is a nano-hybrid, while TBF is a hybrid composite.

Evaluation of the mechanical properties of resin composites is based on not only the assessment of the inorganic filler components but also the organic matrix they contain [29]. Owing to its high mechanical strength, bisphenylglycidyl dimethacrylate (bis-GMA) is used as the primary component of resin composites. However, bis-GMA is highly viscous; less viscous dimethacrylates such triethylene glycol dimethacrylate (TEGDMA) are preferred for their better handling properties, despite their lower flexural strength [22,30]. Replacing bis-GMA and TEGDMA with urethane dimethacrylate (UDMA) may increase the flexural strength [22]. Components like UDMA, TEGDMA, and ethoxylated bis-GMA (EBPDMA) form more flexible polymers than bis-GMA [13]. On the other hand, monomers like bis-GMA and ethoxylated bisphenol-A dimethacrylate (BisEMA) are characterized by lesser cyclization, more cross-linking in the polymer, and better mechanical properties. The use of monomers such as TEGDMA and UDMA results in increased flexibility and intramolecular cyclization. Moreover, the stiffness of bis-GMA and bis-EMA is an important contributor to their improved compressive strength [24]. Among the materials used in this study, only the low-viscosity bulk-fill composite, SDR does not contain bis-GMA, which increases the cross-linking of its matrix and improves the mechanical properties. Nonetheless, there was no significant difference between the mechanical properties of low-viscosity and high-viscosity (containing bis-GMA) bulk-fill composites. Therefore, it could

be stated that the composition of the organic matrix may have a negligible effect on the mechanical properties. Yet, the organic matrix composition is known to affect the handling properties of the material. In order to decrease the number of restoration failures due to fracture, it is important to use materials with a flexural modulus similar to that of dentin [18]. The flexural modulus of the specimens used in our study (20-33 GPa) were similar to that of dentin (17-25 GPa) [18].

In summary, bulk-fill composites are important in simplifying clinical procedures and chairside time. Although they provide alternatives to conventional resin composites, clinicians have to be cogent to appropriate material selection for each case. The bulk-fill composites tested in this study demonstrated poor mechanical properties compared to the nano-hybrid composite, with the exception of Sonicfill. However, the compressive strength properties improved upon applying bulk flow composites to a depth of 4 mm under the conventional hybrid composites.

CONCLUSIONS

From this study, the following conclusions were drawn:

1. The composition of the organic matrix played a negligible role on the mechanical properties but compressive strength results were related to the filler content of the tested resin composite materials.

2. Increasing the layer thickness of the low-viscosity bulk-fill composite (SDR) from 2 to 4 mm under the conventional nano-hybrid composite (FK) improved the compressive strength.

3. The flexural strength and the flexural modulus increased with the filler content and size. Despite their similar filler contents, high-viscosity bulk-fill hybrid composite (TBF) showed lower flexural strength compared to the high-viscosity bulk-fill nano-hybrid composite (SF).

4. The mean flexural strength of the tested resin composites were higher than the 80 MPa established by ISO 4049/2009 for occlusal restorations.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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