Original papers

Comparative evaluation of fiber-reinforced, bulk-fill and conventional dental composites: Physical characteristics and polymerization properties

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Abstract

Background. Resin composites have various applications. At the same time, they have some drawbacks, such as polymerization shrinkage. Conventional composites are polymerized in 2-mm thick layers. However, in posterior restoration, the 2-mm depth of cure is not satisfactory. To find a solution, resin composites have been vastly improved in terms of fillers, matrix and initiators.

Objectives. To evaluate polymerization properties and physical characteristics of fiber-reinforced composites and compare them with bulk-fill composites that are designed for large posterior restorations.

Materials and methods. Samples were prepared from each resin composite. The 3-point bending test was performed to evaluate the flexural strength of all composites. The depth of cure of the composite from 1 mm to 4 mm of depth was analyzed using Vickers hardness test (VHN). To analyze the degree of conversion, Fourier-transform infrared spectroscopy (FTIR) of the top and bottom surfaces of the samples with 4-mm thickness was calculated. The data were analyzed using one-way analysis of variance (ANOVA) test followed by post hoc test (95% confidence interval (95% CI)).

Results. The Filtek showed the highest flexural strength followed by everX and X-tra fil. At 1-mm depth, X-tra fil had the highest and Gradia had the lowest microhardness. At the 4-mm depth, the microhardness trend was as follows: everX > Filtek > X-tra fil > Gradia > Beautifil. The everX composite had the lowest reduction of the degree of conversion at 4-mm thickness, which showed a significant difference in comparison with Filtek, Gradia and X-tra fil composites.

Conclusions. Based on the results of our study, it can be concluded that the fiber-reinforced composite everX showed more favorable results regarding polymerization properties, such as the degree of conversion and the depth of cure. However, the flexural strength results in Filtek were better than those in everX.

Key words: FTIR, resin composite, depth of cure, degree of conversion, physical properties

Background

Considering an increasing aesthetic demand among patients, tooth-colored restorative materials have received a lot of attention.[1] Resin composites have various applications in restorative dentistry because of their aesthetic and safety values.[2] At the same time, they have some drawbacks such as polymerization shrinkage, wear and technique sensitivity in posterior restorations, as well as discoloration, which lead to a debate among scientists regarding the effectiveness of composite restorations.[3] Brunthaler et al. reported that the main reasons for restoration failure and replacement are fracture and secondary caries.[4] Conventional composites are polymerized in layers with 2-mm thickness. However, in posterior restoration, the 2-mm depth of cure is not satisfactory.[5] To find a solution for this concern, resin composites have improved vastly in terms of fillers, matrix and initiators.[6,7] Fillers with higher translucency and loading in bulk-fill composites enable better light penetration and reduce polymerization shrinkage in a more efficient polymerization.[8] In conventional composites, the objective was to increase the amount of filler in order to improve their mechanical properties, and at the same time reduce the size of the fillers to improve their optical properties.[9] However, since the goal for posterior restoration is higher depth of cure, fillers in bulk-fill composites are fewer in number, greater in size and more translucent.[10] Factors such as color and thickness of composite layers alongside the chemical composition of the resin composite affect the polymerization of these materials.[11] The reduction of the light energy leads to a lower degree of conversion and reduced polymerization, which in turn cause poor mechanical properties of the composite. The difficulties in curing conventional composites increase the treatment time and chance of clinical errors.[12] Also, a complete polymerization of the composite is crucial for the restoration to achieve adequate physical and mechanical properties. An inadequate polymerization of the resin composite can lead to marginal microleakage,[13] discoloration,[14] reduced bond strength,[15] and recurrent caries. An incomplete polymerization also results in increased monomer release from composites, which compromises the biocompatibility.[16,17] Therefore, because of characteristics such as high polymerization depth which results in less chairside time and higher physicomechanical performance, bulk-fill composites have become more popular.[18] A newly introduced fiber-reinforced composite for large posterior restoration has several advantages such as the high depth of cure and mechanical properties similar to the dentin.[19] Although there are some studies on the physical and mechanical properties of bulk-fill composites, the scope of research regarding the comparison of polymerization kinetics and mechanical properties of fiber-reinforced composite with bulk-fill and conventional resin composites is limited. Different techniques have been used to evaluate the degree of conversion and depth of cure of resin composites, such as microhardness evaluation and Fourier-transform infrared spectroscopy (FTIR) spectra by the comparison of the unpolymerized residual monomer bands.[20,21] To guarantee the exact polymerization behavior of resin composites, both the depth of cure and the degree of conversion are essential.

Accordingly, the purpose of this study was to evaluate and compare fiber-reinforced and bulk-fill composites, designed for large posterior restoration, with each other and with a conventional resin composite, in terms of degree of conversion, depth of cure and flexural strength, at different thicknesses. The null hypothesis states that there are no significant differences between all resin composites regarding the abovementioned properties.

Materials and methods

Materials

The bulk-fill composites investigated in the current study included Beautifil-Bulk (SHOFU, Kyoto, Japan), Filtek Bulk Fill (3M Oral Care, St. Paul, USA) and X-tra fil (VOCO, Cuxhaven, Germany). The fiber-reinforced composite investigated in the study was everX Posterior (GC Dental, Tokyo, Japan), and the conventional composite was Gradia Direct (GC Dental). The detailed information on the materials is shown in Table 1.

Mini-flexural strength test

Cylindrical composite samples were made using rectangular shape molds with the size of 1.2×1.2×12.5 mm. The molds were cleaned using sterile gas and alcohol, and

Table 1. Detailed information on resin composites used in this evaluation

Manufacturer	Name	LOT No.	Composition	Filler (w%/v%)
GC Dental	Gradia Direct	1704241	UDMA, ethylen dymethacrylate	73/65
GC Dental	everX Posterior	1610061	Bis-GMA, TEGDMA	74.2/53.6
SHOFU	Beautifil-Bulk	101725	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	87/74.5
3M Oral Care	Filtek Bulk Fill	N782245	AUDMA, UDMA and 1,12-dodecane-DMA	76.5/58.4
VOCO	X-tra fil	1715341	Bis-GMA, UDMA and EBPDMA	86/70.1

UDMA – urethane dimethacrylate; Bis-GMA – 2,2-bis(4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl) propane; TEGDMA – triethylene glycol dimethacrylate; EBPDMA – ethoxylated bisphenol A dimethacrylate; Bis-MPEPP – 2,2-bis(4-(2 methacryloxyethoxyphenyl) propane; AUDMA – aromatic urethane dimethacrylate.

the releasing agent was applied for stress-free separation of samples (Al-Cote; Dentsply, Woodbridge, Canada). The resin composite paste was placed inside the molds and the excessive material was removed. The samples were covered with Mylar matrix, fixed to a 1-mm thick glass slab and photopolymerized using a light-curing unit (BlueLEX LD-105; Monitex Industrial Co., Taipei, Taiwan) 2 times for 20 s in overlapping fragments. The intensity of light emited from the light-curing unit was controlled using a radiometer. After light polymerization, the glass was separated and the samples were removed from the molds. The specimens were washed with water and kept in an incubator in a dark place for 24 h in order to complete the process of polymerization. To evaluate the flexural strength, the samples were placed in an Universal Testing Machine (Santam, Tehran, Iran) with 0.5 mm/min crosshead speed. The maximum fracture load was recorded, and the flexural strength (MPa) was calculated using the following equation (Equation 1):

$$FS = 3Fl/2bh^2 \quad (1)$$

Microhardness evaluation

In order to evaluate the depth of cure for each resin composite, the measurement of the composite microhardness in different thicknesses was performed. A mold with the height of 4 mm was prepared and the resin composite was inserted into the mold. Each sample was photopolymerized using light-emitting diode (LED) curing unit from the top of the mold. In every millimeter, 3 indentations were made, and the average hardness was measured and reported. For this purpose, the Vickers microhardness device (Bareiss, Oberdischingen, Germany) was used. The force applied by the device was set at 0.49 N (50 g for 15 s). The microhardness results for each depth were recorded. The adequate depth of cure for each specimen was considered at the minimum threshold of the 80% of the microhardness value of the sample surface.

ATR-FTIR analysis

The degree of conversion on the surface and at the 4-mm depth of cure of each resin composite was measured using the FTIR analysis. Samples were made from each resin composite in a disc with 4-mm thickness. Each specimen was analyzed using FTIR with an attenuated total reflectance sensor (ATR-FTIR; NICOLET™ iS™ 10; Thermo Fisher Scientific, Waltham, USA). The spectra were acquired at the range of 500–4000 cm^{-1}. The area under the absorption peaks graph with wavelengths of 1720 cm^{-1} as the internal standard and 1637 cm^{-1} as the aliphatic absorbance peak area was measured, and the degree of conversion (DC) was calculated based on the formula below (Equation 2):

$$DC = \left(1 - \frac{\frac{\text{aliphatic}}{\text{aromatic}}\ \text{area cured material}}{\frac{\text{aliphatic}}{\text{aromatic}}\ \text{area uncured material}}\right) \times 100 \quad (2)$$

It should be noted that aliphatic carbon varied during polymerization and aromatic carbon stayed constant, and the ratio of these 2 determines the variations in polymerization. In each group, the samples were compared to the uncured state of the same composite so that the polymerization rate could be calculated. These calculations were made for the surface of the composite and at 4-mm depth of cure.

Statistical analyses

All the data were analyzed using SPSS software.[22] The obtained data were examined using analysis of variance (ANOVA) performed for the significant differences between different composites, followed by post hoc test (95% confidence interval (95% CI)) for each variable on datasets ($p < 0.05$).

Results

Flexural strength

The results of the flexural strength measured from samples of each group showed that the highest flexural strength value belonged to the Filtek (289.86 ±31.92 MPa), followed by everX (274.30 ±37.95 MPa) and X-tra fil (258.95 ±40.88 MPa) (Fig. 1). The Beautifil and Gradia flexural strength results were 170.59 ±28.62 MPa and 160.40 ±14.95 MPa, respectively. The statistical analysis confirmed that these 2 groups had significantly lower flexural strength compared to other composites ($p < 0.05$).

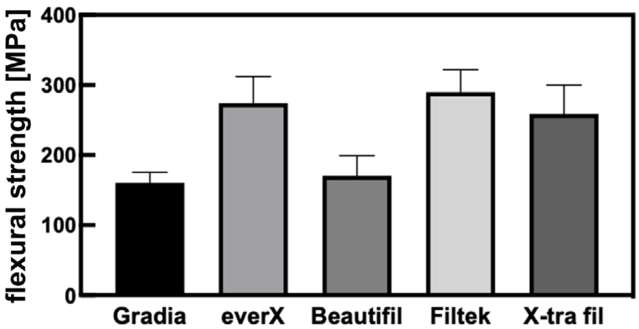

Fig. 1. Bar graph illustrating flexural strength results of all resin composite groups

Microhardness

The microhardness evaluation was carried out in 1-mm to 4-mm thick composites, and the results were reported as an average of the 10 samples (Fig. 2). At the 1st millimeter, the order of the microhardness values for all the groups was as follows: X-tra fil > Filtek > Beautifil > everX > Gradia. The highest microhardness value at the 1st millimeter was attributed to the X-tra fil which was 64.85. The Gradia microhardness results at the first 3 mm were 32.83, 30.83

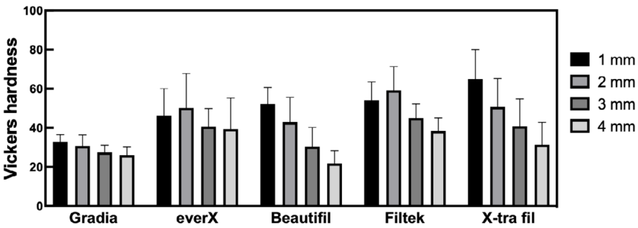

Fig. 2. Vickers microhardness results of each resin composite at 4 different depths of cure

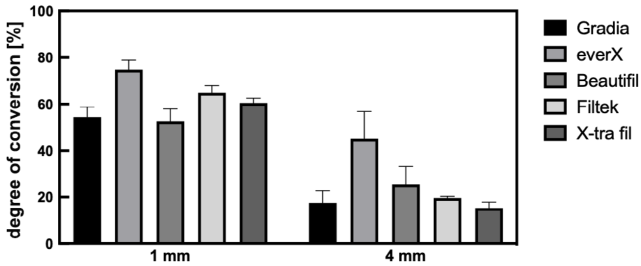

Fig. 3. Average degree of conversion for each resin composite at 1- and 4-mm depth

Table 2. Ratio of microhardness in 2-mm to 4-mm thickness to the surface microhardness of each composite

Composite	Group	Ratio
Gradia	2 mm/1 mm 3 mm/1 mm 4 mm/1 mm	94.4450 84.0332 79.5819*
everX	2 mm/1 mm 3 mm/1 mm 4 mm/1 mm	112.0892 92.5081 88.5457
Beautifil	2 mm/1 mm 3 mm/1 mm 4 mm/1 mm	82.3630 57.9888* 42.4291*
Filtek	2 mm/1 mm 3 mm/1 mm 4 mm/1 mm	112.1046 84.5168 72.3740*
X-tra fil	2 mm/1 mm 3 mm/1 mm 4 mm/1 mm	80.3615 65.1734* 48.1461*

* star-selected values indicate more than 20% decrease in microhardness among deep layers.

and 27.64, respectively, which was the lowest hardness among all examined composites. The microhardness trend at the 4-mm depth was as follows: everX > Filtek > X-tra fil > Gradia > Beautifil. At 4-mm thickness, the hardness values of Gradia and Beautifil were 26.10 and 21.84, respectively, that being the lowest values compared to other composites.

The hardness ratio for each millimeter in each composite was reported as a percentage of the 1st millimeter (Table 2). In a separate analysis of each group, the microhardness result showed an acceptable depth of cure; however, it significantly decreased at 3-mm and 4-mm thickness. In Filtek, Gradia, X-tra fil, and Beautifil composites, the decrease in hardness value at 4 mm constituted more than 20% of the surface hardness ($p < 0.05$).

FTIR

The average degree of conversion of the top and bottom surface of composite samples was measured using FTIR (Fig. 3). The results obtained during the FTIR analysis at the 1-mm and 4-mm depth and their comparison between composites showed a statistically significant difference between the groups.

The order of the degree of conversion values at the top surface of the specimens were as follows: everX > Filtek > X-tra fil > Gradia > Beautifil. The everX degree of conversion at the 1 mm was 74.85, which was significantly higher compared to the other groups. At the 4 mm, the order of the degree of conversion was changed and was as follows: everX > Beautifil > Filtek > Gradia > X-tra fil. The everX composite showed the reduction of degree of conversion at 4-mm thickness. Also, the value of this composite was significantly higher than Filtek, Gradia and X-tra fil composites ($p < 0.05$).

Discussion

The application of resin composites is getting more attention and is becoming more popular among both clinicians and patients. However, there is still an ongoing debate among scientists regarding a complete polymerization of resin composites and their mechanical properties in posterior restoration. The newly introduced fiber-reinforced composites and bulk-fill composites have been produced specifically for large posterior restorations. The manufacturers of these composites claim that their products can provide complete polymerization and adequate physical and mechanical properties in the thickness greater than 2 mm. The aim of this study was to evaluate and compare the depth of cure, the degree of conversion and the flexural strength of fiber-reinforced, bulk-fill and conventional resin composites.

In the mini-flexural test, which was performed in our study, the sample size dimensions were smaller than those in the ISO method. Smaller dimensions, compared to conventional tests, reduce the number of possible flaws such as porosities. Additionally, the stress distribution occurs with more precision. Also, the dimensions of the specimen are more similar to the real dimensions of the samples in dental practice.[23] The results of flexural strength tests showed that Filtek had the best flexural properties compared with all other groups, followed by everX and X-tra fil. A greater flexural strength of Filtek compared to the fiber-reinforced composite can be related to the fibers of the everX that make this composite more susceptible to fracture. A conventional Gradia composite had the worst results; however, the difference between Gradia and Beautifil was not significant. Studies showed that such difference in flexural performance of different composites can be attributed to the resin matrix mixture, filler properties and its percentage on each composite.[8,24]

The microhardness evaluation is an index which determines the adequacy of polymerization depth. In this test, the minimum threshold is 80% of the microhardness value of the top surface, according to the standards.[25,26] In the current study, the microhardness value of the bulk-fill composites at the 1st millimeter were higher than fiber-reinforced and conventional composites. However, at higher depths, the microhardness of Beautifil composite decreased significantly, and at the 4-mm thickness, the hardness of this composite became lower than in the conventional group. As it can be seen in Fig. 2, at the 4-mm depth, the fiber-reinforced composite everX showed the highest hardness value, which is also related to its higher depth of cure. The other composites did not show 80% of their top surface hardness at the 4-mm depth. Moreover, it is clear that the X-tra fil and Beautifil composites did not show acceptable depth of cure in terms of 80% hardness value of the top surface at the 3rd millimeter (Table 2). According to the results of our research, everX and Filtek had more stable hardness results at all depths and could be placed in bulk in posterior restorations. However, the results indicated that the Beautifil and X-tra fil composites are not suitable for single-step placement in bulk. The microhardness results of Filtek and everX showed higher hardness value at the 2nd millimeter compared to the 1st millimeter. This seems to be related to the initiation of the polymerization reaction and formation of free radicals in the composite.[27] At 2-mm thickness, a sufficient light penetration, the presence of free radicals from chain reactions in the 1st millimeter and heat from polymerization further stimulate free radicals and monomers, which enable this level of hardness to occur.

The FTIR analysis, which have been used in the current study, is one the most common tests for the degree of conversion analysis.[21,28,29] There is a carbon double bond (C=C) in the resin monomers of the composites, which breaks and turns into a C–C bond. This conversion links the monomers and creates the polymer. The aliphatic C=C differs before and after the polymerization, and the absorbed wavelength of this bond is 1637 cm^{-1}. As the values reported by the FTIR device have no quantitative value, they are compared to a constant absorptive peak in order to analyze the difference before and after the polymerization and estimate the polymerization rate. The C=C bond of the aromatic ring in monomers is constant and one of the indexes used in calculating the ratio of absorptive peaks is the wavelength absorbed by this bond (1608 cm^{-1}). Since some composites in this study did not have aromatic rings in their monomers, another constant bond in the monomer at the terminal end of the molecule (-OH) with the absorbed wavelength at the 1720 cm^{-1} was used. The everX showed the highest degree of conversion, followed by Filter and X-tra fil. The lowest degree of conversion was attributed to Gradia and Beautifil. The e-glass fibers in the everX composite had a positive effect on its polymerization, facilitating the light penetration and scattering that result in better polymerization and higher depth of cure.[30] One of the modifications applied to monomers is using more urethane dimethacrylate (UDMA) in bulk-fill resin-based composites. This monomer has a low molecular weight, higher concentration of double bonds and low viscosity. Studies showed that the combinations of 2,2-bis(4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl) propane (Bis-GMA) and UDMA or triethylene glycol dimethacrylate (TEGDMA) display a more rigid network and higher polymerization.[7,31,32] The high degree of conversion in Filtek and X-tra fil may be related to UDMA in their resin matrix. Even though X-tra fil composite showed the highest decrease in polymerization, it only had a statistically significant difference in this regard when compared with everX and Beautifil composites. Nevertheless, considering the decrease in hardness at 4-mm thickness, it showed poor results.

Based on the FTIR and microhardness results obtained in the current study, it should be mentioned that although Beautifil, Filtek and X-tra fil are introduced as bulk-fill resin composites, the polymerization at the depth of 4 mm is not complete. Since incomplete polymerization results in failure of the restoration over time, it is recommended to use layering technique in the clinics to attain more durable and satisfactory results. Regarding the application of everX in the deep cavities, based on the flexural strength results of our study and manufacturer's recommendation, it is advised to cover it with a conventional composite. A layer of conventional composite causes a synergic effect, stopping crack propagation on the restoration and leading to a strong biomimetic restoration (Table 3).

Conclusions

Based on the results of our study, it can be concluded that the fiber-reinforced composite everX showed more favorable results in terms of polymerization properties

Table 3. Comparison of all measured properties

Property	Gradia	everX	Beautifil	Filtek	X-tra fil
Flexural strength [MPa]	160.4	274.3	170.59	289.86	258.95
Microhardness (VHN) 4 mm/1 mm	79.58	88.54	42.42	72.37	48.14
Degree of conversion (%) 4 mm/1 mm	32.05	60.16	48.07	30.23	25.31

VHN – Vickers hardness test.

such as degree of conversion and depth of cure. However, the results of the flexural strength in Filtek were higher than those in everX. Among the experimental bulk-fill composites, Beautifil did not show reliable results to be used in bulk concerning its polymerization properties.

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