

Original article

Physical and mechanical characteristics of short fiber-reinforced resin composite in comparison with bulk-fill compositesShiva Jafarnia¹⁾, Alireza Valanezhad¹⁾, Sima Shahabi²⁾, Shigeaki Abe¹⁾, and Ikuya Watanabe¹⁾¹⁾Department of Dental and Biomedical Materials Science, Graduate School of Biomedical Sciences, Nagasaki University, Nagasaki, Japan²⁾Department of Dental Biomaterials, Research Center for Science and Technology in Medicine, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran

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Abstract

Purpose: The objective of this study was to evaluate the physical and mechanical properties of a short fiber-reinforced resin composite: everX-Posterior and compare it with two bulk-fill composites, namely, Filtek Bulk-fill and Beautifil-Bulk, which are intended for large posterior restorations.

Methods: Investigated properties were flexural strength, flexural modulus, surface roughness, volumetric shrinkage and depth of cure. Scanning electron microscopy images of each specimen after the flexural test were used for cross-sectional comparison. Results were analyzed using ANOVA following Tukey *post-hoc* test.

Results: Flexural strength of everX-Posterior was comparable with two other resin composites, showing higher flexural modulus. EverX-Posterior showed the highest surface roughness after polishing and the lowest volumetric shrinkage (2.29%) among all composites used in this study. Data also showed that the everX-Posterior depth of cure was 4.24 mm, which was the highest among the three groups.

Conclusion: Based on the results of this study, it was concluded that everX-Posterior as a short fiber-reinforced composite showed improvements and satisfactory performance in mechanical and physical properties, which make it a reliable base material candidate for large posterior restorations.

Keywords; flexural properties, physical properties, posterior restoration, short fiber-reinforced resin composite

Introduction

Resin composite as a direct restorative material in stress-bearing areas has been widely used in clinical practice [1]. Resin composite has several advantages over amalgam, such as esthetics and biosafety; it also has a reasonable price compared to gold alloy, which makes this material popular among dentists [2]. However, there are some issues with mechanical properties and high polymerization shrinkage that limit the success of the restoration [3,4]. Conventional resin composites have inadequate mechanical properties that will lead to restoration deformation and body fracture under occlusal load [5]. High polymerization shrinkage followed by oral fluid and bacterial penetration causes marginal degradation and, subsequently, secondary caries on the margin of the restoration [4]. Various factors such as the organic and inorganic content of a resin composite affect its polymerization shrinkage; as the amount of organic matrix increases, the material's shrinkage will become greater [6].

In recent years, research has been carried out on reinforcement techniques to strengthen resin composites and overcome shortcomings. The nature of resin composite components and the network between them are major factors affecting physical and mechanical properties of restorative materials [7]. Despite advancements in the fabrication of resin compos-

ites, there remain numerous issues concerning the formulation of optimal posterior resin composites. Bulk-fill composites were established for large posterior cavities to mitigate the limitations of conventional resin composites in depth of cure and mechanical properties [8]. Bulk-fill resin composites showed a favorable depth of cure at 4 mm and can be placed in bulk instead of the incremental technique [9]. It has been reported that the majority of bulk-fill restorative materials have less polymerization shrinkage even though they can be placed in the cavity in bulk and their physical and mechanical properties have been improved [6,10]. Short fiber-reinforced resin composite is another category, consisting of e-glass fibers, resin matrix and inorganic fillers [11]. The resin matrix in this composite is bisphenol-A-glycidyl methacrylate (Bis-GMA), and triethylene glycol dimethacrylate (TEGDMA) with linear polymethylmethacrylate (PMMA); this matrix formed a semi-interpenetrating polymer network (semi-IPN) during polymerization, which results in good bonding properties [12,13]. The short glass fibers incorporated into the resin matrix are millimetre-scale, discontinuous and in high aspect ratio, which provide enhanced mechanical properties that aim to be close to natural tooth tissues [11,14,15].

Since there are various available options for posterior resin composite restorations, there is uncertainty among clinicians in choosing the best material for optimum results regarding tooth condition. Properties such as flexural strength, flexural modulus, surface roughness, polymerization shrinkage and depth of cure affect the restoration's microstructural characteristics and mechanical performance. Therefore, there is a need for a detailed comparison of available composites that will allow clinicians to make an informed decision regarding appropriate restorative materials.

Accordingly, the purpose of this study was to make a comparative evaluation of physical and mechanical properties of the short fiber-reinforced composite everX-Posterior and two bulk-fill resin composites, Filtek Bulk-fill and Beautifil-Bulk, which are intended for large posterior restorations.

Materials and Methods**Study materials**

The resin composites used in this study were short fiber-reinforced composite, everX-Posterior (GC, Tokyo, Japan), and bulk-fill resin composites intended for large posterior restorations: Beautifil-Bulk (Shofu, Kyoto, Japan) and Filtek Bulk-fill (3M ESPE, St. Paul, MN, USA). The investigated materials were kept at 22°C in a cool, dry place. A list of resin composite components and manufacturers is shown in Table 1.

Flexural strength and flexural modulus measurement

A three-point bending test was performed according to ISO 4049 [16]. Specimens were prepared in a stainless steel mould (2 × 2 × 25 mm), covered with a glass slab. Resin composites were placed on each mould and photopolymerized with light emitting diode (LED) curing unit (Delight series, Dentall, Seoul, Republic of Korea) with a light intensity of 1,200 mW/cm² in close contact with the glass slab and at 90° angle to the specimen for 20 s in three overlapping parts. All the samples were carefully removed from the mould and kept in distilled water at 37°C for 24 h. Specimens were then subjected to a universal testing machine (5566S, Instron, Canton, MA, USA) for flexural properties analysis. The distance between supports set to 20 mm (l) and crosshead speed was 0.5 mm/min, the maximum fracture load (F) of each sample was recorded for flexural

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Table 1 Resin composites investigated in this study

Material	Classification	Resin matrix	Filler type	Manufacturer
everX-Posterior	short fiber-reinforced resin composite	Bis-GMA, PMMA, TEGDMA	short e-glass fiber, barium borosilicate glass (74.2 wt%, 53.6 vol%)	GC, Tokyo, Japan
Beautiful-Bulk	bulk-fill resin composite (high viscosity)	Bis-GMA, TEGDMA	S-PRG: surface reaction type pre-reacted glass-ionomer with fluoro-aluminosilicate glass (87.0 wt%, 74.5 vol%)	Shofu, Kyoto, Japan
Filtek Bulk-fill	bulk-fill resin composite (high viscosity)	AUDMA, UDMA, DDMA	silica, zirconia, zirconia/silica cluster and ytterbium trifluoride (76.5 wt%, 58.4 vol%)	3M ESPE, St. Paul, MN, USA

Bis-GMA, bisphenol-A-glycidyl methacrylate; PMMA, polymethylmethacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate; DDMA, 1, 12-dodecanediol dimethacrylate; wt%, weight percentage; vol%, volume percentage

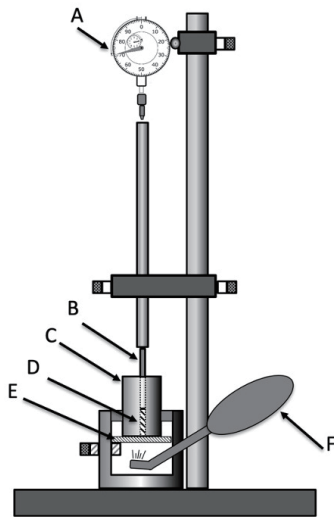


Fig. 1 Schematic illustration of the apparatus designed for polymerization shrinkage measurement. A: dial gauge, B: metallic bar, C: mould, D: resin composite, E: glass slab, F: LED curing unit

strength measurement. The flexural strength was calculated according to the following equation:

$$\sigma_f = 3Fl/2bh^2$$

In this equation, b is the specimen width and h is specimen height. The flexural modulus was calculated from the slope of the load-deflection curve in the linear region.

Scanning electron microscopy (SEM) observation

The representative fractured surface of each specimen following a three-point bending test was sectioned using a low speed diamond saw (Isomet, Buehler, Lake Bluff, NY, USA) and sputter coated by gold (IC-50, Shimadzu, Kyoto, Japan) for 5 min with 10 mA current. Samples were observed by scanning electron microscopy (SEM, JCM-6000 Plus, JEOL, Tokyo, Japan) with an accelerating voltage of 15 kV.

Surface roughness test

To measure surface roughness, thirty specimens of each restorative material were made, and samples were prepared in 8 mm diameter and 2 mm thickness. Specimens were divided into three groups ($n = 10$) based on the polishing condition: non-polished, polish #400 and polish #80 with the silicon carbide sandpaper. The surface roughness was measured using a profilometer (Surfcom 480A-12, Tokyo Seimitsu, Tokyo, Japan) after calibration. The surface roughness (R_a , μm) of each specimen was recorded using five intervals with a 0.8 mm cut off and 0.1 mm/s speed. Five readings were taken and the mean of the five readings was recorded to represent the roughness of each specimen.

Polymerization shrinkage

An apparatus was designed to obtain the polymerization shrinkage of each resin composite. A schematic illustration of the device is presented in Fig. 1. Fifteen specimens were made for polymerization shrinkage measurement of each material. A rectangular stainless steel mould ($4 \times 2 \times 15$ mm) was filled with the resin composite and pressed by a metallic bar to

eliminate voids. Then it was fixed in a dial gauge with 100 grams pressure. The bottom side of the mould was covered with a glass slab and the resin composite was cured using an LED curing unit with a light intensity of $1,200 \text{ mW/cm}^2$ in close contact with the glass slab at 90° angle to the specimen for 20 s. The displacement of the dial gauge was recorded until the dial gauge stopped. Finally, the displacement of dial gauge (ΔL) was calculated, and the thickness of specimens, which had been cured (L), was measured. In a study by Gee et al. [17], to measure polymerization shrinkage, the linear shrinkage ($\text{Lin}\%$) was calculated and then the result was converted mathematically to volumetric shrinkage ($\text{Vol}\%$) by the following equations:

$$\text{Lin}\% = \Delta L / (L + \Delta L) * 100$$

Volumetric shrinkage equation:

$$\text{Vol}\% = 3\text{Lin}\% - 0.03(\text{Lin}\%)^2 + 0.0001(\text{Lin}\%)^3$$

Depth of cure measurement

The depth of cure measurement of resin composite materials was performed according to ISO 4049 protocol [16]. A cylindrical stainless steel mould, which was black-painted for light reflection prevention with 4 mm diameter and 10 mm height, was used for this test. The mould was filled with resin composite paste, and a glass slab placed on the top of the mould, with a mylar strip covering the top surface. The material was cured for 20 s by an LED curing unit with a light intensity of $1,200 \text{ mW/cm}^2$ in close contact with the glass slab with a 90° placement angle from the top of the mould, then the specimen was removed. The bottom part, which was soft and not polymerized, was removed with a spatula. The length of the cured part of the resin composite was measured with a digital calliper (Mitutoyo CD-15C, Mitutoyo, Kawasaki, Japan) three times for each specimen and then the average value was divided by two. The value for each resin composite was recorded as the final depth of cure ($n = 10$).

Statistical analysis

The mean and standard deviation of results obtained from the tests were calculated for each group. The Kolmogorov-Smirnov test primarily assessed the normality of the data, followed by Levene's test for equality of variance evaluation ($P < 0.05$). The data were analyzed using analysis of variance (ANOVA) to determine the significant difference among groups for each variable ($P < 0.05$). Tukey *post-hoc* was used to determine the difference and analysis for each data set.

Results

The Kolmogorov-Smirnov normality test indicated that the results of all the experiments showed a normal distribution. The Levene test results showed homoscedasticity for all experiments; for example, the P -values of flexural strength, flexural modulus, surface roughness, polymerization shrinkage and depth of cure were 0.82, 0.19, 1.13, 0.24, and 0.19, respectively.

Flexural strength and flexural modulus

The mean values of flexural strength for Beautiful-Bulk, Filtek Bulk-fill and everX-Posterior were 114.4 ± 14.1 MPa, 167.5 ± 15.7 MPa, and 145 ± 12.0 MPa, respectively. One-way ANOVA indicated that Beautiful-Bulk flexural strength was significantly lower than Filtek Bulk-fill and everX-Posterior. Also, statistical analysis showed that Filtek Bulk-fill showed significantly higher flexural strength compared to Beautiful-Bulk and everX-Posterior ($P < 0.05$) (Fig. 2a).

The mean values of flexural modulus for Beautiful-Bulk, Filtek Bulk-fill

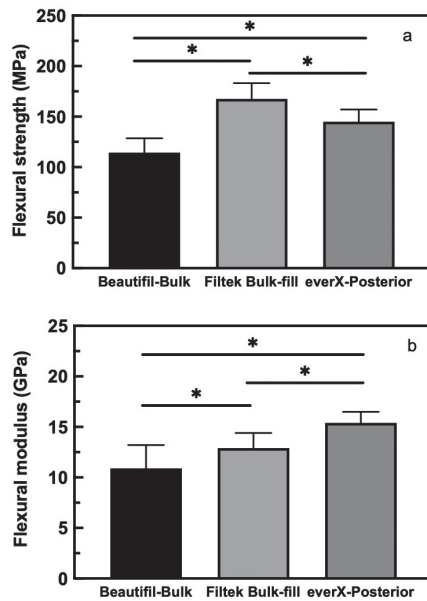


Fig. 2 a. flexural strength and b. flexural modulus bar graph. Significant difference was indicated between all groups in flexural strength and flexural modulus ($*P < 0.05$, statistically significant difference).

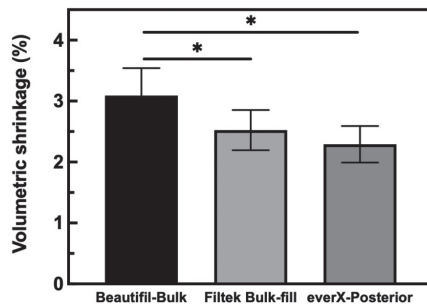


Fig. 4 Bar graph of volumetric shrinkage for each resin composite ($*P < 0.05$, statistically significant difference)

and everX-Posterior were 10.9 ± 2.3 GPa, 12.9 ± 1.5 GPa, and 15.4 ± 1.1 GPa, respectively. One-way ANOVA indicated that everX-Posterior had the highest value in flexural modulus results, and Beautifil-Bulk showed the lowest value. The flexural modulus among all three groups was significantly different (Fig. 2b).

SEM observation

Representative SEM images of the fractured surface of each resin composite after the three-point bending test are shown in Fig. 3. The fractured surface of each resin composite showed typical topography. The everX-Posterior specimens showed random orientation of short glass fibers and pulled out fibers after fracture. The two other composites without glass fibers had a relatively homogenous flat fractured surface.

Surface roughness

Surface roughness test results before and after abrasion with different sandpapers are shown in Table 2. Two-way ANOVA showed significant influence of sandpaper smoothness on the surface roughness of resin composites in all three experimental groups. There was a significant difference between surface roughness in the group without abrasion and the group abraded with sandpaper #400 grit in the Filtek Bulk-fill and everX-Posterior ($P < 0.05$). However, in the Beautifil-Bulk there was no significant difference between groups without polish and #400 grit. Grinding with sandpaper #80 grit showed a statistically significant difference between the group without abrasion and the group abraded by sandpaper #80 grit. On the other hand, Beautifil-Bulk had a higher surface roughness before polishing compared to other composites. Polishing with sandpaper #80 grit showed that everX-Posterior had the highest roughness among all three groups.

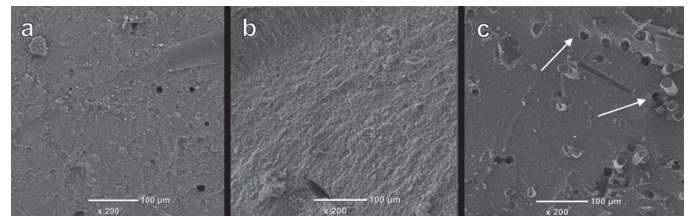


Fig. 3 SEM micrographs of the fractured surfaces a. Beautifil-Bulk, b. Filtek Bulk-fill and c. everX-Posterior. Random orientation of glass fibers which had been pulled out (white arrows) from the everX-Posterior and smoother surface of bulk-fill composites is clear in these pictures

Table 2 Comparison of mean \pm (standard deviation) of the surface roughness (Ra, μm) of each resin composite group

	Non-polished	Polish #400	Polish #80
Beautifil-Bulk	$0.53 \pm (0.06)^{A,a}$	$0.63 \pm (0.01)^{A,b}$	$1.53 \pm (0.28)^{B,d}$
Filtek Bulk-fill	$0.30 \pm (0.04)^{B,a}$	$0.81 \pm (0.04)^{E,bc}$	$2.54 \pm (0.39)^{F,e}$
everX-Posterior	$0.47 \pm (0.06)^{C,a}$	$1.01 \pm (0.10)^{H,c}$	$3.06 \pm (0.51)^{I,f}$

Different uppercase letters indicate significant difference among grinding conditions for each resin composite (within the row), and different lowercase letters indicate significant difference among composite resin in each condition (within the column) ($P < 0.05$).

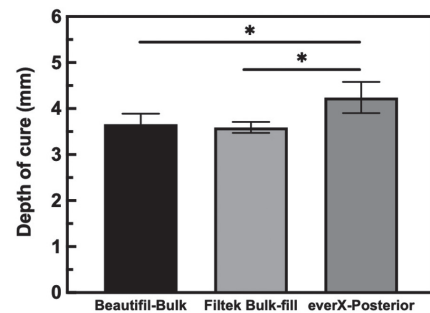


Fig. 5 Bar graph of depth of cure for each resin composite ($*P < 0.05$, statistically significant difference)

Polymerization shrinkage

The result of polymerization shrinkage of each resin composite is illustrated in Fig. 4. The volumetric shrinkage mean values for Beautifil-Bulk, Filtek Bulk-fill and everX-Posterior were $3.09 \pm 0.45\%$, $2.52 \pm 0.33\%$, and $2.29 \pm 0.30\%$, respectively. One-way ANOVA indicated that Beautifil-Bulk had significantly higher volumetric shrinkage than the two other composites ($P < 0.05$) and everX-Posterior had the lowest shrinkage. However, the volumetric shrinkage of Filtek Bulk-fill and everX-Posterior were not significantly different ($P > 0.05$).

Depth of cure

The depth of cure results for resin composites are shown in Fig. 5. As indicated in the statistical analysis, the depth of cure of Filtek Bulk-fill and Beautifil-Bulk was 3.59 ± 0.12 mm and 3.66 ± 0.23 mm, respectively. There was no significant difference between these groups ($P > 0.05$). However, the depth of cure for everX-Posterior was 4.24 ± 0.34 mm, which was significantly higher than other composites ($P < 0.05$).

Discussion

The aim of this research was to analyze the physical and mechanical properties of the short fiber-reinforced composite everX-Posterior and two bulk-fill resin composites, Filtek Bulk-fill and Beautifil-Bulk, which are intended for large posterior restorations. In the present study, Filtek Bulk-fill showed significantly higher flexural strength compared to other composites, however in flexural modulus analysis everX-Posterior result was significantly higher than the two other composites. The higher result of flexural strength in Filtek Bulk-fill may be due to the resin matrix mixture, which contains AUDMA, UDMA and the monomers that make it plasticize

more [18,19]. The lower flexural modulus of Filtek Bulk-fill compared to that of everX-Posterior is probably influenced by differences in filler size and shape between these two resin composites. The e-glass fibers in the short fiber-reinforced composites enhance the modulus by the mechanism of transforming stress from the resin matrix to the fibers which act as a stress barrier [20]. The fibers in this resin composite have a random orientation, which improves elasticity and prevents toughness. Also, they can stop propagation of cracks in the restoration and increase modulus [21]. The differences in flexural properties presented in this study for bulk-fill composites and short fiber-reinforced composites are in accordance with previous research [11,22]. Differences in flexural properties between resin composites can be attributed not only to filler content but also to the type and size of the filler in the composition of the restorative material.

The SEM images of fractured surfaces of resin composites (Fig. 3) show differences in resin matrix and inorganic filler content in each specimen after fracture. Differences in mechanical properties of the composites were influenced by the microstructure shown in the surface topology. The typical topography showed a smoother fractured surface in Beautiful-Bulk and Filtek Bulk-fill, and glass fibers in everX-Posterior. An SEM image of everX-Posterior indicated pulled out fibers that carry the load and reinforce the elastic modulus of this short fiber-reinforced resin composite.

Surface roughness is one of the factors that can affect biofilm formation and bacterial adherence on the surface of a restoration [23]. It is recommended by GC corporation that everX-Posterior requires a layer of a universal composite as a capping layer. The everX-Posterior showed lower surface roughness before polishing; however, its roughness drastically increases after polishing. The higher surface roughness of this resin composite after polishing may result from abrasion of the resin matrix and its glass fibers, which reduce its polishability. The increased surface roughness also compromises the restoration's esthetic, which might be one of the reasons for the capping layer recommended by GC corporation [24]. All three experimental composites had significantly higher roughness after polishing with silicon carbide sandpaper #80 grit. Although, Beautiful-Bulk showed less increase compared to the two other composites, it might be related to filler size and type, which leads to a smoother surface. Along with the smoothness of sandpaper, various factors such as filler size, distribution of filler and size influenced the surface roughness of the resin composites [25-27].

Polymerization shrinkage induces contraction and stress between the tooth and restoration and could cause inadequate marginal adaptation, gap formation and secondary caries [28,29]. Shrinkage results between three experimental groups documented that everX-Posterior had lower shrinkage than the two other bulk-fill composites. Previous studies indicated that volumetric shrinkage has a relationship with factors such as filler load, filler size and shape and organic matrix of a resin composite [30,31]. The everX-Posterior has a semi-IPN resin matrix that results in a plasticized matrix and short glass fibers in different directions, which does not allow the material to shrink easily in one dimension; these characteristics result in less shrinkage in comparison with bulk-fill composites.

In clinical practice, since it is hard to place the light cure unit in close contact with the tooth surface in posterior restorations, depth of cure is an important feature. It can affect the overall success of the restoration. Insufficient polymerization throughout the restoration might cause clinical problems such as marginal leakage, pulpal inflammation and restoration failure [32]. In the present research, the depth of cure values measured by the ISO method were significantly higher in everX-Posterior than the two other groups. Previous studies showed that everX-Posterior had a high translucency, which leads to a high depth of cure; the current study result is consistent with that result [12]. The high depth of cure in both bulk-fill composites and short fiber-reinforced composites influence and enhance their clinical performance. Further investigation is necessary to compare other characteristics, such as the biological properties of these resin composites.

Based on the results of this study, it was concluded that everX-Posterior as a short fiber-reinforced composite showed improvements in flexural modulus, polymerization shrinkage and depth of cure, which result in the better performance of this resin composite compared to other types of composites. The high surface roughness of everX-Posterior after polishing

supports the manufacturer's recommendation that capping with conventional resin composite is necessary. Improvements in everX-Posterior properties make this resin composite a promising candidate as a base material for application in high stress-bearing areas and large cavities in posterior teeth.

Conflict of interest

The authors declare that they have no conflict of interest.

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