An *in-vitro* Evaluation of Mechanical Properties and Surface Roughness of Bulk Fill vs Incremental Fill Resin Composites

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ABSTRACT

Aim: To investigate and compare the mechanical properties of two bulk fill resin composite materials (Tetric EvoCeram and Filtek Bulk Fill) and two incremental fill resin composites (Grandio and Filtek Z350 XT). The surface roughness and surface topographic analyses of these materials were also evaluated and compared.

Materials and methods: A total of 120 specimens (n = 20) were prepared from two bulk fill nanocomposite restorative materials and two incremental fill nanocomposite restorative materials to evaluate the mechanical properties [hardness (H), elastic modulus (E), compressive strength (S), flexural strength (O_f), and surface roughness]. The hardness and elastic modulus were measured using nanoindenter equipped with a Berkovich diamond indenter. Compressive strength and flexural strength of each material were determined using a universal testing machine. The surface roughness of the materials was determined using atomic force microscopy. One-way analysis of variance followed by Tukey's *post hoc* test was used to determine the statistical differences among groups at a significance of p < 0.05.

Results: The nanoindentation test showed that the Filtek Bulk Fill exhibited significantly high hardness values $(0.67 \pm 0.02$ GPa) compared with other groups. The elastic modulus values ranged from 12.2 to 18.2 GPa, Tetric EvoCeram presented with lowest modulus values, and Filtek Bulk Fill presented with highest values. For compressive strength test results, the values varied from 186.20 MPa for Grandio to 245.13 MPa for Filtek Z350 XT. For flexural strength test results, the values varied from 110.00 MPa for Grandio to 132.61 MPa for Filtek Z350

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Conclusion: Each material showed excellent values for some tests, but none of the material showed excellent values of all the measured properties. Filtek Bulk Fill could be a feasible choice among the bulk fills.

Keywords: Bulkfill Composites, Nanoindentation, Surface Roughness.

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INTRODUCTION

Resin-based composites were introduced to dentistry in the 1950s to overcome the esthetic problem rather than the functional problem of dental amalgam.¹ Composite resin is "a mixture or combination of two or more micro or macro constituents which differ in form and chemical composition and are essentially insoluble in each other."² The composites introduced in the early years had many disadvantages, such as polymerization shrinkage up to 20 to 25%, unstable color, low stiffness, and lack of adhesion to tooth structure.

During the last four decades, there has been tremendous improvements and innovations in developing more stable composite materials.³ These developments have focused mainly on reducing polymerization shrinkage and improving the mechanical properties, such as hardness, compressive strength, flexural strength, and fracture toughness.⁴ The progression in filler and polymer technology of dental composite resins has led to a wide variety of composite material selection based on clinical situation.^{5,6}

A low degree of monomer conversion is a major disadvantage of resin composites.⁷ Due to insufficient curing, incremental placement technique with a maximum of 2 mm thickness is being practiced for restoring large cavities, especially class II.⁸ However, the use of incremental placement technique of resin composite is time consuming for the patient and the operator.⁹ There may also be chances of moisture contamination or air bubble entrapment between each incremental layer of resin composite.¹⁰

The introduction of bulk fill composites has aimed at solving the obstacles faced by incremental placement techniques. The advantage of the so-called "bulk fill" composite material is that it can be placed in a 4-mm thickness bulk and cured in one step instead of the current incremental fill technique, without any effect on polymerization shrinkage, cavity adaptation, and decreased moisture contamination.^{11,12} Bulk filling composites have higher filler volume percentage, and occasionally a modified initiator system to ensure better curing in depth, as compared with traditional composites, thus ensuring superior physical and mechanical properties to combat higher masticatory forces.¹³ It is also reported that the bulk fill resin composites reduce cuspal deflection and promote light transmittance.^{12,14}

When defining composites, certain properties should be evaluated. These include, but are not limited to, hardness, flexural strength, and compressive strength.¹⁵ Therefore, the aim of this study was:

- To compare the mechanical properties of bulk fill resin composite and incremental fill resin composites. The following properties were evaluated: Nanohardness (H), elastic modulus (E), compressive strength (S), and flexural strength (O_f).
- To investigate the differences in surface roughness between the materials.

MATERIALS AND METHODS

The brand, composition, and manufacturer of the composite materials used in the current study are listed in Table 1.

Nanoindentation Measurement of Hardness and Elastic Modulus

Five disc-shaped specimens $(12 \times 8 \text{ mm})$ per each composite resin material were prepared using a silicone mold according to manufacturer's recommendation. The

specimens were polished with Swiss flex abrasive discs to obtain a smooth surface for nanoindentation measurement. The hardness and elastic modulus were measured using nanoindenter (Bruker, Tucson, Arizona, USA) equipped with a Berkovich diamond indenter in ambient temperature of 23°C and low noise conditions. The indenter loading rate was 0.01 mN/s and unloading rates was 0.02 mN/s, with a 5 s resting period for varying the load between 1.0 and 25 mN. Five indentations per composite specimen were made and mean values of nanohardness were calculated. Once the hardness values of the specimens were determined, the elastic modulus was obtained mathematically from the load displacement curve.

Compressive and Flexural Strength Measurement

Ten cylindrical specimens $(6 \times 3 \text{ mm})$ and ten bar-shaped specimens $(25 \times 2 \times 2 \text{ mm})$ were prepared per each composite resin material for compression and flexural strength measurements respectively. All the specimens were stored in distilled water for 24 hours before testing. The test was performed with a universal testing machine (Instron Corporation, Massachusetts, USA). The compressive load was applied along the long axis of the specimen at a cross-head speed of 1.0 mm/min until the compressive failure of the specimen occurred. For flexural strength measurements, the specimens were placed on the attachments on a universal test machine with 20 mm distance between the supports followed by a transverse load directed toward the center of the specimen at a crosshead speed of 1 mm/min. The data were collected by the computer connected to the universal testing machine.

Surface Roughness Measurements

Two disk-shaped specimens $(12 \times 2 \text{ mm})$ per each composite resin material were prepared using a silicone mold according to manufacturer's recommendation. The specimens were polished with Swiss flex abrasive disks with a decreasing size of the abrasive particles (coarse, medium, fine, ultrafine) to obtain a smooth surface for surface

Table 1: Composite materials used in the present study

Com	Composition			
Name Resin matrix	Inorganic filler (wt. %)	Manufacturer		
Tetric EvoCeram Nanohybrid Bulk Fill UDMA, EBADMA	Barium glass filler (80 wt. %)	Ivoclar Vivadent AG, Schaan, Liechtenstein		
Filtek Bulk Fill Nanofiller Bulk Fill Bis-GMA, Bis-EMA, UDMA	Zirconia (64 wt. %)	3M ESPE, St. Paul, Minnesota, USA		
Grandio Nanohybrid Incremental fill Bis-GMA, TEGDMA	Barium-boron-alumino- silicate glass (87 wt. %)	Voco, Cuxhaven, Germany		
Filtek Z350 XT Nanofiller IncrementalBis-GMA, UDMA,fillTEGDMA, Bis-EMA	Silica Zirconia (72.5 wt. %)	3M ESPE, St. Paul, Minnesota, USA		
fill TEGDMA, Bis-EMA	e dimethacoulate: ERADMA: Et	JIVI ESPE, SI		

*Bis-EMA: Ethoxylated bisphenol A dimethacrylate; UDMA: Urethane dimethacrylate; EBADMA: Ethoxylated bisphenol A dimethacrylate; Bis-GMA: Bisphenylglycidyldimethacrylate; TEGDMA: Triethylene glycol dimethacrylate



roughness measurement. The specimens were subjected to atomic force microscopic (AFM; MultiMode 8-HR, Bruker, USA) analysis operating in contact mode. The measurements were done in air. Totally, six areas (2 in right, 2 in left, and 2 in the center) were scanned for evaluating the surface roughness. The surface roughness was calculated by the Nanoscope software connected to the AFM system.

Statistical Analysis

The data collected from each test were subjected to statistical analysis using Statistical Package for Social Sciences version 18.0 (SPSS Inc., Chicago, Illinois). The means of each group were analyzed by one-way analysis of variance (ANOVA), and multiple comparisons of means were tested with Tukey's *post hoc* analysis at a significance limit of p < 0.05.

RESULTS

The analyzed data of nanohardness and elastic modulus measurements are presented in Table 2 and Graph 1. The nanohardness values ranged from 0.42 to 0.67 GPa. The one-way ANOVA test presented significant differences ($p \le 0.05$) among the tested composite materials. The elastic modulus values ranged from 12.2 to 18.2 GPa. Tetric

 Table 2: Mean and standard deviation (SD) of nanohardness

 and elastic modulus values of the analyzed data. The values are

 expressed in GPa

	Nanohardness			Elastic modulus		
			Post hoc			Post hoc
Groups ($n = 5$)	Mean	SD	analysis*	Mean	SD	analysis*
Tetric EvoCeram	0.42	0.05	А	12.2	1.92	A
Filtek Bulk Fill	0.67	0.02	В	18.2	0.83	В
Grandio	0.55	0.04	С	16.6	1.81	В
Filtek Z350 XT	0.48	0.04	A,C	13.6	0.89	А

*Means with same capital letters imply no statistically significant values (p<0.05)



Graph 1: The dual plot chart illustrating the mean nanohardness and elastic modulus of the tested composite specimens

Table 3: Mean and standard deviation (SD) of compressive andflexural strength values of the analyzed data. The values areexpressed in MPa

	Compressive Strength			Flexural strength		
Groups			Post hoc			Post hoc
(n = 5)	Mean	SD	analysis*	Mean	SD	analysis*
Tetric EvoCeram	238.32	8.22	А	115.16	8.41	А
Filtek Bulk Fill	213.40	7.89	В	130.84	5.53	В
Grandio	186.20	4.32	С	110.00	5.70	А
Filtek Z350 XT	245.13	9.67	А	132.61	9.09	В

*Means with same capital letters imply no statistically significant values (p<0.05)

EvoCeram presented with the lowest modulus values and Filtek Bulk Fill presented with the highest values.

The compressive strength values varied from 186.20 MPa for Grandio to 245.13 MPa for Filtek Z350 XT. There were significant differences ($p \le 0.05$) among the tested composite material group as shown by Tukey's honest significant difference test ($\alpha = 0.05$) (Table 3). The flexural strength values varied from 110.00 MPa for Grandio to 132.61 MPa for Filtek Z350 XT. There were significant differences among the bulk fill and incremental composite resin groups as shown by Tukey's *post hoc* analysis ($p \le 0.05$) (Table 3).

The analyzed data for surface roughness test measurements are presented in Table 4 and Figures 1A to D. The values varied from 51.66 MPa for Tetric EvoCeram to 93.40 MPa for Filtek Z350 XT. There were no significant differences ($p \le 0.05$) between the bulk fill composite resin groups and incremental composite resin groups as shown by Tukey's *post hoc* analysis.

DISCUSSION

The present study evaluated four nanocomposites, out of which two were bulk fill and two were incremental fill composite resin. The nanocomposites were used in the present study because they have a low shrinkage relative to the high filler content,^{16,17} good mechanical properties, a better polish and gloss, an excellent surface finish, and an increased resistance to wear.¹⁸

 Table 4: The mean and standard deviation (SD) of surface

 roughness values of the analyzed data. The values are expressed

 in Ra

			Post hoc
Groups ($n = 2$)	Mean	SD	analysis*
Tetric EvoCeram	51.66	3.93	А
Filtek Bulk Fill	54.83	2.78	А
Grandio	88.66	2.42	В
Filtek Z350XT	93.40	3.25	В

*Means with same capital letters imply no statistically significant values (p < 0.05)



Figs 1A to D: The representative surface roughness image measured by AFM at right, center, and left portions of (A) Tetric EvoCeram; (B) Filtek Bulk Fill; (C) Grandio; and (D) Filtek Z350 XT specimen

Nanoindentation is a widely accepted technique for determining the mechanical properties of a material from the derived indentation load–displacement response.¹⁹ Nanoindentation is unique from other measurement techniques as there is no need to image the indentation area to determine the mechanical properties, such as hardness. These can be obtained directly from the indentation load and displacement measurements.²⁰

Hardness is an indirect measurement of the degree of conversion (%) of the material and gives valuable information on the depth of polymerization when such measurements are carried out on the top and bottom surfaces of cured samples.²¹⁻²³

The elastic modulus describes the relative stiffness of a material. A high elastic modulus is required to withstand deformation and cuspal fracture especially in stress-bearing occlusal contact areas. The materials with a low modulus deform more under masticatory stresses and may cause a catastrophic failure.^{11,24,25}

The nanohardness and elastic moduli (Graph 1) of two bulk fill and two incremental fill resin composites evaluated in this study showed Filtek Bulk Fill with good hardness and elastic modulus values compared with other tested materials. This observation was contradictory to the outcome of elastic modulus values of the previous study for Filtek Bulk Fill. The present study presented with values of 18.2 GPa as compared with 3.7 GPa in the previous study. This difference may be due to the method of testing; the previous study had used a three-point bending test for testing the elastic modulus of the material.²⁶

The elastic modulus values and nanohardness values for Grandio recorded in our study were very similar to the values obtained in the previous studies. The nanohardness values for Grandio in our study were 0.55 GPa as compared with 0.72 GPa in the previous studies.^{26,27} However, Grandio presented with excellent hardness and modulus values in both the previous studies as compared with our study. A study on hardness of composite resins confirmed that Tetric EvoCeram Bulk Fill enabled curing up to 4 mm in one step, which is similar to the results of this study.²⁸

Flexural strength is the material property that indicates the quantity of flaws within the material that may have the ability to cause catastrophic failure due to loading.^{29,30} The compressive strength plays an important role in the



mastication process.^{31,32} The compressive and flexural strength values of the incremental fill resin used in the study were very similar to the values reported earlier by Rosa et al,²⁷ except for the compressive strength of Filtek Z350 XT, which was comparatively a high value of 245.13 MPa as compared with 184.67 MPa in the previous study.

Another study by Leprince et al,²⁶ evaluated the flexural strengths of Tetric EvoCeram, Filtek Bulk Fill, and Grandio, and the values obtained were 94.5, 88.4, and 125 MPa as compared with 115, 130, and 110 MPa in our study. On the contrary, Ilie et al³³ showed that the flexural strength of Tetric EvoCeram and Filtek Bulk Fill was very similar and in agreement with the outcome of the present study.

Among the exhibited properties of the nanocomposites, surface roughness is of utmost importance as it may predict the outcome of the final restoration. Surface roughness on the composite restoration affects the esthetic appearance and discoloration of restorations, plaque accumulation, secondary caries, gingival irritation, and increases tooth wear of opposing or adjacent teeth.^{18,34,35} In the present study, Tetric EvoCeram showed least roughness values of 51.66 nm followed by Filtek Bulk Fill. Both of the bulk fill composite resins showed significantly lower surface roughness values as compared with the incremental fill composite resin. The highest roughness value was found in Filtek Z350 XT. The three-dimensional AFM image (Figs 1A to D) showed uniform surface texture on all the measured points for Tetric EvoCeram and Filtek Bulk fill composite resin materials. However, all the tested composites had roughness values below 200 nm. Surface roughness (Ra) values above 200 nm result in increased plaque accumulation and increased risk of secondary caries and periodontal inflammation.³⁴ This is advantageous from the clinical point of view, because there will be no risk of plaque accumulation on the composite surfaces.³⁶

The limitation of this study was that it was performed under ideal or laboratory conditions; the test results could be even less in clinical situations depending on the technique, isolation, and many other variables. Further research should emphasize on testing the degree of conversion and marginal integrity of Bulk Fill and Incremental Fill composite resins.

CONCLUSION

In the present study, the following conclusions are drawn:

- Filtek Bulk Fill showed better mechanical properties than all other materials compared, except for a low compressive strength.
- Filtek Bulk Fill exhibited better mechanical properties than Tetric EvoCeram among the bulk fill materials, and Filtek Z350 XT was better than Grandio.

 Tetric EvoCeram had the least surface roughness compared with other composite materials, but was not statistically significant.

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