

Micro-Hardness of Bulk-Fill versus Layered Resin- Based Composites

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Abstract

Objectives: This study was conducted to evaluate surface micro-hardness of bulk-fill versus incremental layered resin-based composites.

Materials and Methods: Forty cylindrical split molds (10 mm diameter and 2 mm thick) were constructed from Teflon. Four groups of specimens were prepared, ten per each material (n=10): two groups for different bulk-fill composites, one group nano-hybrid composites and one group micro-hybrid composites. Micro hardness measurements were performed using a micro hardness tester with a Vickers indenter.

Results: There were high statistically significant difference between all the tested restorative materials (P<.0001).

Conclusions: SonicFill bulk-fill and microhybrid Filtek z250 resin composite showed high Vickers micro-hardness values.

Keywords: Bulk Fill, Incremental layering, nano-hybrid resin composite, micro-hybrid resin composite, micro-hardness.

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I. Introduction

The smoothness of restorative material's surfaces has a great importance in the success and clinical longevity of the restorations¹⁻³. The surface roughness of a resin composite relates to the composition, porosity of the material, the instruments and procedures used in polishing^{4, 5-8}. In addition, the surface roughness of a resin composite has been recognized as a parameter of high clinical relevance for wear resistance, plaque accumulation, gingival inflammation, material discoloration (especially in Class V restorations), and surface gloss⁹⁻¹¹.

The most smooth and glossy surface is generally obtained under a Mylar strip without subsequent finishing or polishing, but unfortunately intra-oral finishing is always required¹². The mylar strip finished surface has higher resin content and will reduce the wear resistance of the restoration over time. Therefore, finishing and polishing of tooth-colored restoration after placement are inevitable procedures that will improve esthetics; early wear resistance, color stability and marginal integrity^{1,13}. Several investigations have shown that removal of the polymer-rich, outermost resin layer is essential to achieving a stain-resistant, more esthetically stable surface¹³⁻¹⁵.

Knowledge of the physical properties of composite restorative materials is important to aid our understanding of their behavior under clinical conditions. Hardness is considered one of the most important properties of these materials.^{16,17} The most used methods to evaluate the elastic properties of composite resins are the Knoop and Vickers micro-hardness.^{16, 18} These are considered indirect methods to evaluate the degree of polymerization of resin composites which have already been reported to correlate with the degree of conversion of carbon double bonds. Furthermore; hardness profiles can be used to alternatively measure the depth of cure of such resinous materials.¹⁹ It has been related to strength, proportional limit and ductility of materials and has been used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing tooth structure and materials.²⁰ Therefore; the objective of this study was intended to evaluate surface micro hardness of different bulk-fill resin based composites. The null hypothesis was that there were significant differences among restorative materials tested.

II. Materials & Methods

Two commercially available bulk-fill resin composites and two incremental-fill resin composites; one nano-hybrid and one micro-hybrid resin composite, were used in the study. Materials specification, composition and manufacturers were listed in table 1.

Table 1. Materials specification, composition and manufacturers

Restorative system	Manufacturer	Composition
SonicFill	Kerr, Orange, CA, USA	Bis-GMA, TEGDMA, EBPDMA Silica, glass, oxide (83.5wt%, 69 vol%)
Tetric N Ceram (Nanohybrid)	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, Bis-EMA, UDMA Barium glass, PrePolymerized Fillers, YbF3, Oxide 75-77 wt%
Filtek Bulk Fill	3M, ESPE, St. Paul, MN, USA	Bis-GMA, UDMA, Bis-EMA, procrilate resins Ytterbium trifluoride, zirconia, silica (64.5wt%, 42.5 vol%)
Filtek Z250 (Microhybrid)	3M, ESPE, St. Paul, MN, USA	Bis-GMA, Bis-EMA, TEGDMA, UDMA zirconia, silica (82wt%, 60 vol%)

Cylindrical split mold (50 mm diameter and 2 mm thick) was constructed from Teflon. In the center of the mold a circular recess (10 mm diameter) was constructed and used for preparing the composite specimens.²¹ Four groups of specimens were prepared, ten per each material (n=10): two groups for bulk-fill composites; group I Sonic fill and group II for Filtek bulk fill and two groups for incremental-fill composites; group III for nano-hybrid Tetric-N ceram and group IV for micro-hybrid Filtek z250 resin composites. For bulk-fill composites, each restorative material was placed in bulk in the mold using Optra Sculp modeling instrument (Ivoclar Vivadent AG, Schaan, Liechtenstein) over a transparent, 0.051 mm thick Mylar strip (Universal strip of acetate foil, Italy) and a glass slide. For incremental-fill composites each restorative material was applied incrementally in two horizontal increments with approximately 1-mm thickness. Each increment was gently condensed with clean non sticky composite condenser. Black paper was placed between the glass slide and Mylar strip to prevent reflection of light during polymerization.²² Effort was made to prevent the inclusion of air voids while inserting the material in the mold. Another Mylar strip and a glass slide one mm thick were placed over the inserted material. A 500 gm stainless steel weight was applied for 30 s over the specimen, allowing the composite to flow in order to obtain a smoother and standardized surface.

After removal of the weight, curing was performed according to manufacturer's instructions. Only one operator performed all the procedures of specimen's preparations. A light emitting diode (LED) visible-light curing unit (bluephase C8, Ivoclar Vivadent AG, Schaan, Liechtenstein) was used, and the power density of the light (800 mW/cm²) was checked every 10 specimens with a digital readout dental radiometer (bluephase meter,

Ivoclar Vivadent AG, Schaan, Liechtenstein). The distance between light source and specimen was standardized by curing through the glass slide. The tip of the light curing unit was in contact with the covering glass slide. Finally the specimens were removed from the mold.

All the specimens were notched on their reverse side to serve as an orientation aid for the finishing procedures, each disc was notched at two locations 180° apart to ensure consistent orientation of specimens during polishing procedures (double notch at one edge; single notch at the opposite edge) 23, which were carried out perpendicular to the notch.

Specimens were finished with 600 grit silicon carbide paper 24 (standard finishing) then polished with Sof-Lex discs (3 MESPE, Seefeld, Germany) following a decreasing sequence of abrasiveness (Coarse 55µm, medium 40 µm, fine 24 µm and ultrafine 8 µm) using a low-speed hand piece at approximately 4,000- 5,000 rpm. Uniform light pressure and a circular pattern for 10 s for each abrasive step were used to polish the specimens 25. Sof-Lex discs were discarded following each use.

After the finishing procedures, the specimens were washed with air-water spray for 5 s and examined under a stereomicroscope (Nikon model SMZ-IB, Tokyo, Japan) for grinding debris or surface defects and then stored in distilled water at room temperature for 24 h 26 then were dried with oil-free air. The upper surfaces of the specimens were marked with waterproof pen. The prepared specimens were stored in distilled water in dark at room temperature for 24 h to assure complete polymerization. 27

Micro-hardness measurements were performed using a micro-hardness tester (Durimet, Leitz, Wetzlar, Germany) with a Vickers indenter (Fig 1). The specimen was placed flat on a glass slide and mounted on a holder on the microscope stage. The specimen surface was examined microscopically and the indenter was then moved into position and the microscope stage raised steadily until the required load was applied by the indenter upon the specimen.

The Vickers micro-hardness test uses a square based diamond pyramid as the indenter. The included angle between nonadjacent faces of the pyramid are 136°, and Vickers hardness number (VHN) is equal to the applied force in kg divided by the actual area of the impression in mm². The applied load was 50 gm for 5 s. Under an optical microscope, each indentation was measured diagonally from one edge of the diamond shaped impression to the other edge. The average diagonal lengths of the indentations were then measured.

Three indentations were performed to the top irradiated surface and three corresponding indentations were made in the bottom surface. Mean VHNs of the top and bottom surfaces were calculated. VHN was calculated using the following equation: 28

$$\text{VHN (Kg/mm}^2\text{)} = 1854.4 \cdot P/d^2$$

Where P = the force in kg.

d = the diagonal length of the impression.

and 1854.4 is a constant.

The results of microhardness values were statistically analyzed with one way analysis of variance (ANOVA) at P <0.05 level of significance. Tukey Post Hoc multiple comparison test was used to determine the significant intra-group differences.

Results

One-way ANOVA (Table 2) revealed statistically significant difference between the tested composite materials (P <0.000). The Tukey Post Hoc test was then performed to determine the significant intra-group differences and showed that, significant differences were found between group I and group II, group I and group III, group I and IV, group II and group III and group III and group IV (P <0.0001). While no significant difference was found between group I and group IV resin composite specimens (P=0.997) with group I demonstrated the highest micro-hardness values.

Fig 1: Vicker's microhardness tester.



Table 2: One way ANOVA test results of comparison of micro-hardness of the tested composite materials.

By material	Sum of Squares	df	Mean Square	F value	P value
Between Groups	1578.133	2	789.066	26.424	<.0001
Within Groups	806.266	27	29.862		
Total	2384.399	29			

III. Discussion

Nowadays, the development of the esthetic dentistry resulted in increasing interest of using resin composites in high stress dental bearing areas. The most important factor that limits their use in these areas is that they are not hard enough to withstand mastication strength. The improvements in the currently available composite materials include the increase of filler content, variations in size, type and morphology of the particles, in addition to changes in the organic matrix. 29 These changes have conferred better mechanical properties to these materials, thus, allowing them to be used in areas subjected to great masticatory efforts.30

Adequate surface hardness of the resin composites is important to obtain optimum clinical performance of the restoratives in stress dental bearing areas. It has been reported that the hardness of inorganic fillers has a direct effect on the material’s hardness. In general, the increase of particle size increases the strength as well as the surface hardness of composite. Moreover, after polymerization, the solidified polymer matrix that is formed plays a role in hardness development.27 A positive correlation has been established between the hardness and inorganic filler content of resin composites. 29

Therefore, the present study investigated Vickers micro-hardness of composite restoratives based on different resin matrix and different filler size, type, and content. Vickers micro-hardness test was selected for this study because it is relatively a simple technique, very popular and reliable for obtaining the results. Additionally, it is considered by several authors as an indicator for the degree of polymerization of resin materials and used commonly as indirect method to evaluate degree of cure.31 Surface micro-hardness is considered as an indicative factor of the mechanical strength of a resin and correlates well to the material’s rigidity. 32

In the current study, all test samples were submitted to the same parameters of light curing method and finishing. Finishing and polishing were performed for the specimen’s surface after polymerization in order to remove the softer resin rich layer of material and exposing the hardest one. Removal of this weak superficial layer is essential to produce a relatively stable surface with increasing predictability of developing high surface hardness. In this study, 2mm specimen’s thickness of resin composites may be sufficient to allow favorable depth of cure for light penetration and performing the hardness test. Hardness measurements were performed at top- irradiated and base non -irradiated surfaces of the specimens to ensure proper cure of the resin.

The results of the present study revealed that SonicFill bulk-fill resin composite demonstrated the highest VHN which was not significantly differed than micro-hybrid Filtek z250 resin composite. This may be due to the increase in the inorganic filler content; as SonicFill bulk-fill contains about 83.5% inorganic fillers of silicate glass, while Filtek z250 contains about 82% inorganic fillers of silicate zirconium which could be a possible consequence of increasing hardness.

Both nano-hybrid Tetric N ceram and Filtek bulk-fill resin composite demonstrated a lower values than the micro-hybrid composite. This may be attributed to presence of small filler particles that causes a light scattering, thus, decreasing the effectiveness of the curing light.33 In addition to the reduced inorganic filler content of these resin composites. From previous studies, increasing the volumetric content of inorganic particles and enhancing the degree of conversion of the methacrylate-based composites produced higher surface hardness. 34,35

IV. Conclusion

Based on the findings of this study, it can be concluded that Sonic fill bulk-fill and micro-hybrid Filtek z250 restorative materials showed high surface micro-hardness.

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