

**Microhardness study on bulk fill restorative materials - An In-vitro Evaluation**

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**Abstract**

**Aim:** To evaluate the microhardness of four bulkfill restorative materials.1) Filtek Bulk Fill, 2) Cention N, 3)SDR plus Bulk Fill flowable, and 4) Tetric N-Ceram Bulk Fill.

**Materials and method:** All the four restorative samples were procured commercially. Four empty strips of spent tablets were also collected as moulds. The Bulk fill restorative materials were packed in each of these moulds. The samples were then polymerized with LED curing device. All the samples were stored in distilled water for

24 hrs. The samples so prepared were divided into 4 groups and subjected to microhardness indentation test.

**Result:** The result showed statistically significant difference in mean microhardness between the groups with p value <0.001.

Among all the samples tested, Cention N showed highest microhardness value followed by Tetric N-Ceram Bulk Fill, Filtek Bulk Fill and SDR plus Bulk Fill flowable respectively.

**Conclusion:** All the samples tested showed surface hardness within acceptable limits to withstand masticatory load. Cention N showed the highest microhardness

strength compared to all other materials tested. SDR plus Bulk Fill flowable showed the least wear resistance amongst the groups.

**Keywords:** SDR plus, Tetric N-Ceram

### Introduction

Resin based restorative materials are in vogue as materials of choice currently, due to properties such as superior esthetics, better mechanical properties and command set function. But these materials still suffer from poor wear resistance properties when used in high stress bearing areas. Another area of concern is the limited depth of cure when used in increments of more than 2mm in thickness. Over the last several years research has been focused in overcoming these disadvantages through the use of better filler particles and other modifications in the chemical structure. This has led to the development of several bulk fill posterior restorative materials. Bulk fill materials have high viscosity, contains high filler loading and capability of being placed in bulk increments of approximately 4-5 mm thickness. These materials can be applied and light cured in bulk. This resulted in reduced restorative procedure time, minimized air void entrapment, and improved quality of the final restoration.

Wear resistance properties of bulk fill restorative materials have not been extensively studied. Wear resistance is measured clinically as surface hardness of the material. The Metals Handbook defines hardness as "Resistance of metal to plastic deformation, usually by indentation. However, the term may also refer to stiffness or temper or to resistance to scratching, abrasion, or cutting. It is the property of a metal, which gives it the ability to resist being permanently, deformed (bent, broken, or have its shape changed), when a load is applied. The greater the hardness of the metal, the greater resistance it has to deformation.

In this in vitro study, Vickers microhardness test of four different bulk fill restorative materials, Filtek Bulk Fill, Cention N, SDR plus Bulk Fill flowable, and Tetric N-Ceram Bulk Fill were evaluated.

### Materials & Methods

Bulk fill restorative materials (3M™ ESPE™ Filtek™ Bulk Fill Posterior, Cention N Ivoclar-Vivadent, SDR plus Bulk Fill flowable, and Tetric® N-Ceram Bulk Fill) were procured commercially [Fig.1(a)]. The samples were made by packing each material in to ten bubble moulds of 4 empty tablet strips to get a total 40 samples. Each group consisted of 10 samples to make four groups. Each round sample measured 12mm in diameter and 4mm in height. The Bulk fill restorative materials were packed in each of these strips and light cured [Fig.1 (b)]. The restorative materials were divided into 4 groups (10 restorative samples of one restorative material in each group).

Group I: - Filtek Bulk Fill Posterior (3M ESPE) was packed in the mould using a plastic filling instrument and condensed with the help of a condenser.

Group II: - Cention N (Ivoclar-Vivadent) was packed in the mould using a plastic filling instrument and condensed with the help of a condenser.

Group III: - SDR plus Bulk Fill flowable (Dentsply Sirona) was packed in the mould using a compule gun and excess was removed.

Group IV: - The Tetric N-Ceram Bulk Fill (Ivoclar-Vivadent) was packed in the mould using a plastic filling instrument and condensed with the help of a condenser.

Restorative samples in each strip were polymerized with Woodpecker Led D Light Cure Unit (Light output 850-1000mW/cm<sup>2</sup>). All the samples were stored in distilled water for 24 hrs at 37°C. The group samples were mounted and subjected to indentations using microhardness indenter (Fig.2).



Fig.1(a) Bulk fill restorative materials used in this study. (b) Restorative samples made in empty medicine moulds.

Vickers hardness test<sup>1</sup>

The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a test force of between 1gf and 100kgf. The full load is normally applied for 10 to 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation.

HV = Vickers hardness

$$HV = \frac{2F \sin \frac{136^\circ}{2}}{d^2} \quad HV = 1.854 \frac{F}{d^2} \text{ approximately}$$

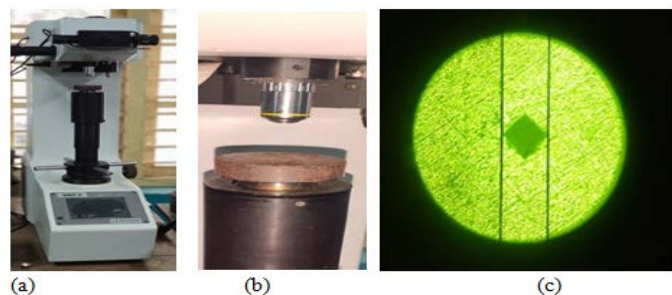


Fig 2: a) Vickers hardness testing machine b) Indenter c) Representative image of Vickers microhardness.

Results

Table 1: Microhardness values of each samples

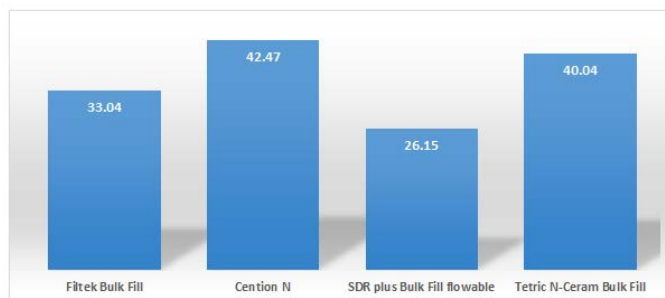
Statistical Analysis: The mean value and standard deviation were calculated. VHN data were subject to One - way ANOVA and Tukey’s Post hoc test were used to compare the microhardness between the groups. Significant differences were considered at p<0.05.

One - way ANOVA test

	N	Mean	Std. Deviation	p-value
Filtek Bulk Fill	10	33.0400	3.54595	<0.001
Centon N	10	42.4700	2.10821	
SDR pluUs Bulk Fill flowable	10	26.1500	1.58202	
Tetric N-Ceram Bulk Fill	10	40.0400	1.34759	
Total	40	35.4250	6.82844	

Table 2: Comparison of mean values with One- way ANOVA test.

The result show that there is statistically significant difference in mean microhardness between the groups with p<0.001. Microhardness is more in Centon N group followed by Tetric N-Ceram Bulk Fill, Filtek Bulk Fill and SDR plus Bulk Fill flowable respectively with mean microhardness (SD) value of 42.47(2.11), 40.04(1.35), 35.43 (6.83) and 33.04(3.56) respectively.



Tukey's Post hoc test for multiple comparisons:

(I) G	(J) G	Mean Difference (I-J)	Std. Error	p-value
Filtek Bulk Fill	Cention N	-9.43000*	1.03289	<0.001
	SDR plus Bulk Fill flowable	6.89000*	1.03289	<0.001
	Tetric N-Ceram Bulk Fill	-7.00000*	1.03289	<0.001
Cention N	Filtek Bulk Fill	9.43000*	1.03289	<0.001
	SDR plus Bulk Fill flowable	16.32000*	1.03289	<0.001
	Tetric N-Ceram Bulk Fill	2.43000*	1.03289	<0.024
SDR plus Bulk Fill flowable	Filtek Bulk Fill	-6.89000*	1.03289	<0.001
	Cention N	-16.32000*	1.03289	<0.001
	Tetric N-Ceram Bulk Fill	-13.89000*	1.03289	<0.001
Tetric N-Ceram Bulk Fill	Filtek Bulk Fill	7.00000*	1.03289	<0.001
	Cention N	-2.43000*	1.03289	<0.024
	SDR plus Bulk Fill flowable	13.89000*	1.03289	<0.001

Table 3: Multiple comparisons between groups using Tukey's Post hoc test.

**Discussion**

Bulk fill restorative materials offer many advantages over conventional composite restorative materials, mainly with regard to depth of cure and polymerization shrinkage. In bulk-fill composite resins, alternative and more reactive photo initiators are used to improve polymerization depth and to increase the translucency of the material. Further, the filler content is reduced and the size of the filler particles is increased (2). In addition, some chemical

These results show that with multiple comparisons of mean microhardness values with Tukey's Post hoc test, there were statistically significant values less than 0.05 in each comparison.

modifications have been made to reduce polymerization shrinkage stress, such as increasing the molecular weight of the monomers in their contents, adding new stress-relieving monomers, and including methacrylate monomers (3). The composition and properties of the bulk fill materials used in this study is represented below as table 4.

Materials	Type	Manufacturer	Components	Filler % by weight
3M/ ESPE Filtek One Bulk-fill Restorative	Packable 5mm	3M/ ESPE, St. Paul, MN, USA	AromaticUDMA, UDMA, 1,12-dodecanedimethacrylate. non-agglomerated/non-aggregated silica filler, non-agglomerated/nonaggregated filler, aggregated zirconia/silica cluster filler, ytterbium trifluoride filler	76.5 wt%
Cention N	Packable Alkasite (dual-cure) mechanism	Ivoclar Vivadent AG, Schaan, Liechtenstein	UDMA, DCP, Aromatic aliphatic-UDMA, PEG-400 DMA Barium aluminium silicate glass, Ytterbium trifluoride, Isofiller, Calcium barium aluminium fluorosilicate glass, calciumfluoro silicateglass	78.4% wt

SDR plus Bulk Fill flowable	Flowable 4 mm	Dentsply DeTrey	Proprietary modified urethane dimethacrylate resin; TEGDMA; polymerizable dimethacrylate resin; polymerizable trimethacrylate resin silanated barium-alumino-fluoro-borosilicate glass; silanated strontium alumino-fluoro-silicate glass; surface treated fume silicas; ytterbium fluoride.	70.5 wt%
Tetric N Ceram Bulk Fill (IVA)	Packable 4 mm	Ivoclar Ivoclar Vivadent, Schaan, Lichtenstein	Bis-EMA, UDMA, EBPADMA. Barium aluminium silicate glass, ytterbium trifluoride	80.0 wt%

Table 4: Composition of Bulk-fill restorative materials used in the study

Hardness for dental materials is really important because of high occlusal stress load in restored teeth. For posterior tooth restorative materials and for restorations involving proximal surface, a restorative material of high microhardness value is important as hardness determines its wear resistance.

In the present study, the Vickers hardness value was higher for restorative materials with higher filler loading. Also the size of the filler particles and the filler content influence the hardness of tested materials. Fillers are responsible for imparting restorative materials with the adequate strength to withstand the stresses and strains of the oral cavity and to achieve acceptable clinical longevity.<sup>(4)</sup>

Among the materials tested Cention N showed the highest microhardness value. Cention N has 78.4% wt of filler percentage.(Table 4). Increasing filler content minimize polymerization shrinkage and contraction stress. The increased microhardness of Cention N is probably related to the nanoparticle size of filler. Also, Cention N has an Isofiller (Tetric N-Ceram technology), which keeps shrinkage stress to a minimum. Isofiller minimizes

shrinkage force, whereas the organic/inorganic ratio, as well as monomer composition of the material, is responsible for the low volumetric shrinkage<sup>(5)</sup>.

Tetric N-Ceram Bulk Fill which has a filler percentage of 80.0 wt%<sup>(6)</sup>. contains Bis-GMA which has a strong intramolecular hydrogen bonding of hydroxyl groups, and it is considered the most viscous and least flexible monomer among dental resin monomers(9). It also has isofiller technology and higher filler load, which in turn increased its microhardness values compared to Filtek Bulk Fill group and SDR flow+ Bulk Fill. Filtek Bulk Fill also has high filler loading and contains non-agglomerated/non-aggregated silica filler particles which imparts for high microhardness values comparable with Tetric N-Ceram Bulk Fill.(7).

SDR plus Bulk Fill flowable showed lesser values for microhardness. This can be related to its low filler content and flowable consistency. It has been reported that flowable bulk-fill composites show lower surface microhardness values than high-viscosity bulk-fill composites (8).UDMA contains a hydrogen bond between the amine and carbonyl groups and is also a viscous resin

monomer. However, the viscosity of UDMA is much lower than that of Bis-GMA due to weak hydrogen bonding. UDMA and TEGDMA are the least viscous (9). The SDR group had the lowest filler content (68%) by weight, while it contained modified UDMA and TEGDMA (10). SDR flowable showed low microhardness values in studies comparing bulk fill composites (11, 12). Thus SDR plus Bulk Fill flowable marketed as a dentin replacement requires a veneering layer of micro-hybrid composite.

Higher microhardness values correlate with lower material wear, and thus durability and biocompatibility of composite restorations. All the materials tested here showed the minimum wear resistance or surface hardness required to withstand the intraoral masticatory load.

### Conclusions

Within the limitations of this study, the following conclusions were drawn:

All bulk fill materials used in the study showed a significant difference in their mean microhardness values when compared to each other.

Cention N showed the highest wear resistance compared to all others. SDR showed the least wear resistance amongst the groups.

All the materials showed acceptable wear resistance values to be used clinically in stress bearing areas.

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