

# The Comparative Evaluation of Depth of Cure of Bulk-Fill Composites: An in Vitro Study

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## Abstract

**Context:** The application of composite resins has greatly increased due to their optimal physical, mechanical, and aesthetic properties. Recently bulkfill composites are introduced which simplifies the restorative procedures and saves time.

**Aims:** The aim of the present study is to evaluate and compare the depth of cure of resin-based composites restorations: Sculptable bulk-fill composite Filtek Z250 Xt (3M, ESPE), Flowable bulk-fill composites G-aenial Universal Flo (GC America) and Dual cure bulk fill Multi Core Flow (Ivoclar Vivadent).

**Methods and Material:** Thirty black opaque silicone hollow cylindrical molds were divided into 3 groups (n=10) and bulk filled with each of the three composites and light cured for 20 s followed by 24 h storage in water. The surface hardness was measured on the top and the bottom by recording Vickers hardness number by Vickers hardness indenter.

**Statistical analysis used:** ANOVA and POST HOC Scheffe

**Results:** Oneway ANOVA and Post hoc Scheffe's test was used to calculate p value. MultiCore Flow (IvoclarVivadent) showed least mean of difference between top and bottom surface followed by of Filtek Z250 Xt (3M, ESPE) and G-aenial Universal Flo (GC America). There was significant difference between all groups. (p<0.05)

**Conclusions:** Thus, MultiCore Flow showed maximum depth of cure when compared to other two bulk-fill resin composites.

**Keywords:** Bulk-fill composites; Depth of cure; Dual-cure; Flowable; Microhardness; Sculptable.

## Introduction

Composites, as restorative dental materials have given a new aspect to conservative and esthetic dentistry with their improved mechanical properties, clinical handling, and ability to mimic the natural appearance of teeth. Composites are classified by initiation techniques into 3 types i.e. chemically activated, light activated and dual cure.<sup>1</sup> The introduction of chemically cured resin-based com-

posites (RBCs) paved the way for the beginnings of esthetic restorative dentistry. These composites can build up the lost tooth structure at one time, and have better marginal adaptation and present less damage to the integrity of the restored tooth.<sup>1</sup> Nevertheless they have the drawback of requiring a prolonged setting time that is not under the control of the clinician. It is not only slower but also is less effective than photoactivation with regard to monomer conversion. These lead to the introduc-



tion of light cured resin composites.<sup>2</sup> Light cured resin-composites are the most commonly used direct dental restorative materials nowadays. They are supplied as a single paste in a light proof syringe containing the free radical initiating system, which consists of a photosensitizer and an amine initiator. They interact when exposed to light. It allows the operator to complete insertion and contouring before curing is initiated. They are not as sensitive to oxygen inhibition as chemically cured systems. Because of these advantages, visible light-activated composites are more widely used.<sup>1</sup>

The placement of composite restorations is technique-sensitive and requires adequate light-curing to ensure a thorough cure. If the composite is not sufficiently cured, then the function and longevity of the restoration will be compromised.<sup>3</sup>

A combination of chemical and light curing is used to overcome some of the drawbacks of light curing. Dual cure resins are supplied as two pastes. When mixed together a slow setting reaction is initiated. These resins are used for cementing crowns or bulk restorations where there is limited or no light penetration. After the initial light cure, the remainder of the resin cures over a period of time by the chemical process.<sup>1</sup>

Dental composite restorations have a major drawback regarding the degree of cure, which is proportional to the amount of light they are exposed. So, they polymerize to a certain depth which varies with the penetration of a light beam in the bulk material. This extent of cure has been termed (depth of cure) and has significant influence on both physical and biological properties of restorations. The depth of cure is the depth to which the light is able to harden the material. So that layering technique for resin composite has been a central point in teaching direct resin composite restorations, to ensure their curing.<sup>4</sup>

Compromised depth of cure of a resin-composite material results in insufficient polymerization of deeper portions with subsequent degradation, poor physical properties and adverse biological reactions owing to leaching of the monomeric components of the uncured resin composite.<sup>5</sup>

When restoring cavities with light curing resin composites, the incremental placement technique (maximum 2 mm) has been regarded as the gold standard to apply and cure the resin composite in increments of limited thickness.<sup>3</sup> The intensity of the light source decreases as the distance between the tip of the light and the surface of the composite increases. Hence to obtain maximum polymerization, depth of cure becomes important.<sup>1</sup>

Penetration of the light through the material depends mainly on two factors. Composite related factors include shade, translucency, filler particle size, load, and distribution. Light-related factors include light intensity, irradiance, spectral distribu-

tion, and exposure time.<sup>1</sup> In incremental filling technique there is a risk of incorporating air bubbles or failure to maintain adequate isolation which leads to contaminations between the increments. Other disadvantages of this technique include increased clinical time, technical complexities, reduced bond strengths, voids, and bond failures between adjacent RBC layers.<sup>3</sup>

With advances in polymer chemistry, photo-activation, and curing light technologies, a new “class” of composites called bulk-fill composites has emerged, that enable the restoration to be placed in 4–5 mm thick layer and cured easily and thus replacing both enamel and dentin. The placement of larger increments of RBC may reduce the time and the formation of a gap in the restoration material, thereby reducing the technique sensitivity.<sup>3,6</sup> They should possess reduced polymerization shrinkage, a reasonable depth of cure (DOC), flowable enough to reach all the areas of the preparation without creating voids, excellent physical properties in terms of wear and function and esthetics.<sup>3</sup> Microhardness has been suggested as a way to examine the Depth of cure (DOC) of photo activated resin composite. A value over 0.80 in bottom to top surface microhardness indicates adequate DOC.<sup>3,6</sup>

Filtek Z250 (3M, ESPE) is posterior light cure bulk fill sculptable composite that provides excellent strength and is claimed to achieve 5 mm depth of cure. G-aenial Universal Flo (GC America) represents the next advancement in the light cure bulk fill flowable composites category. It has higher strength, higher wear resistance, and higher gloss retention.

A new bulk-fill composite, Multi Core Flow (IvoclarVivadent) is a dual curing, radiopaque, flowable composite that demonstrates excellent mechanical properties for core build-ups.

Rezaei S. et al compared the curing depth and degree of conversion of five bulk-fill composite resins compared to a conventional composite. Bulk-fill composites evaluated in this study are adequately polymerized at 4 mm depth. Their DC (Degree of Conversion) was optimal and within the range of conventional composites.<sup>8</sup>

The null hypothesis evaluated was that there would be no differences in depth of cure and microhardness properties between the materials.

Limited literature is available comparing the depth of cure of Filtek Z250 Xt (3M, ESPE), G-aenial Universal Flo (GC America) and MultiCore Flow (IvoclarVivadent), hence the aim of the present study was to group different bulk-fill RBCs (sculptable, dual cure, and flowable) for posterior use in this study and to compare the depth of cure and micro-hardness under optimal curing conditions to those of conventional composite materials.

## Materials and Methods

Thirty black opaque silicone hollow cylindrical molds of 4 mm height and 6 mm internal diameter were prepared. The glass slide (1.2 mm thick) was covered with a mylar strip and the mold was placed on it. The silicone molds were divided into 3 groups (n=10) and the complete depth of the mold was bulk filled with the respective composites. (Figure 2).

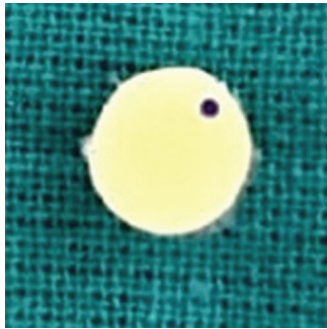


Fig. 1: Prepared Sample.

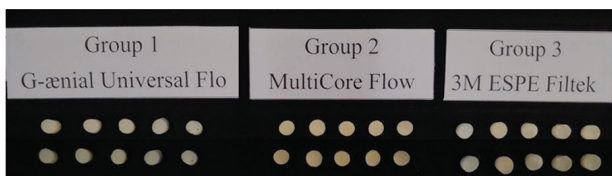


Fig. 2: Thirty samples divided into 3 groups (n = 10).

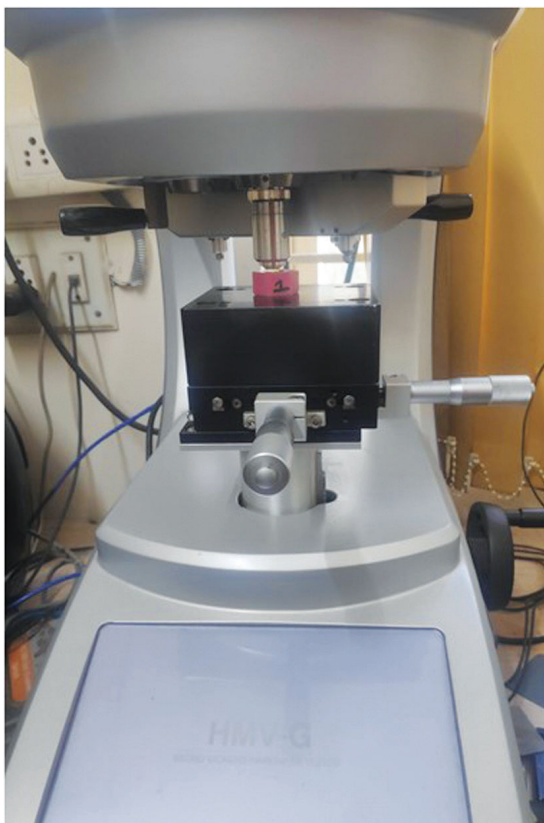


Fig. 3: Vicker's Microhardness Testing.

Group 1: Filtek Z250 Xt (shade A2) (3M, ESPE)

Group 2: G-aenial Universal Flo (GC America)

Group 3: MultiCore Flow (IvoclarVivadent)

In group 1 (Filtek Z250 Xt) the material was bulk filled with the help of Teflon coated instrument while group 2 and 3 were filled with syringe and dispensing tip. After the mold was slightly over-filled, another mylar strip was placed on top and glass slide (1.2 mm thick) was pressed firmly, permitting the excess material to extrude from the mold and to form a flat surface.

The molds were irradiated from one end. The specimen was polymerized for 20s keeping the tip of light-curing unit (Woodpecker LED D) in contact with the glass slide and positioned concentrically with the mold to ensure a constant distance from the specimen. All light-curing procedures were performed with the same curing unit operating in a continuous mode while emitting a light-intensity maintained at full charge before use.

After polymerization, the sample was gently pushed out from the mold, excess flash removed and the top surface (closer to the light source) of the sample was marked with a permanent marker. (Figure 1).

All specimens were stored in water for 24 h at room temperature so that the unreacted monomer would leach out and not affect hardness value (HV). After this, the microhardness test for the specimens was done. In order to prevent operator bias, this test was carried out by another operator (other than who had done the curing of composite specimens).

### Microhardness test

The fabricated samples were subjected to Vickers microhardness test and the top and bottom surface hardness of each 4-mm high specimen were measured using the Vickers microhardness instrument (Shimadzu microhardness tester HMV).

The measuring diamond Indenter, the Vickers pyramid, was pressed to the composite specimen using load 0.25 Kgf for 5 seconds such that the indenter applied load to each surface 3 times with at least 3 diagonal widths of 20  $\mu$ m between indentations. (Figure 3)

The surface Vickers hardness was measured at three points of each specimen. This was done to minimize measurement errors within a specimen. The three measurement microhardness values on the top and bottom were averaged to obtain a single value of Vickers microhardness of each specimen. Each specimen hardness value (HV) of the lower surface was compared with the upper surface value.

**Statistical Analysis**

One way ANOVA and Post hoc Scheffe’s test was used to calculate p value among different groups using Statistical Package for the Social Sciences (SPSS) software version 20. p value ≤ 0.05 was considered to be statistically significant.

**Results**

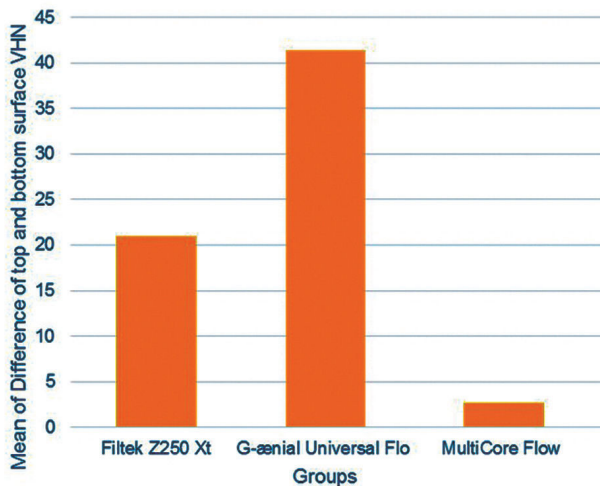
DOC of all three materials was evaluated with the help of Vicker’s microhardness test. Values obtained were subjected to statistical analysis. Analysis of variance with Scheffe’s post hoc test was done. P <0.05 was considered statistically significant.

**Table 1:** comparison of mean of difference of Vicker’s hardness value for each test group.

Group (n=10)	Material	Mean Vickers Hardness Value		Mean of Difference
		Top	Bottom	
1	Filtek Z250 Xt (3M, ESPE)	67.39	46.42	20.97
2	G-aenial Universal Flo (GC America)	64.45	23.02	41.43
3	MultiCore Flow (IvoclarVivadent)	55.60	52.98	2.66

All of the composites showed significantly lower HV values for the bottom compared with the top surface (P < 0.05). (Table 1) A ratio of bottom-to-top surface microhardness over 0.80 indicates adequate DOC.

MultiCore Flow showed least mean of difference between top and bottom surface followed by of Filtek Z250 Xt and G-aenial Universal Flo which suggested maximum depth of cure. There was significant difference between all groups. (p<0.05) (Figure 4).



**Fig. 4:** Graph showing comparison of mean of difference of vickers hardness number of all 3 groups.

Post hoc Scheffe’s test showed that there was statistical significant difference between all three groups. (p=0.000) (Table 2).

**Table 2:** Intergroup comparison of mean of difference of Vicker’s hardness value.

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	p value
1	2	-20.460*	1.156	0.000
	3	18.310*	1.156	0.000
2	3	38.770*	1.156	0.000

\*. The mean difference is significant at the 0.05 level.

**Discussion**

Adequate polymerization is a prerequisite for overall clinical success, longevity and biocompatibility of resin based composite restorations.<sup>2</sup> It is influenced by intrinsic and extrinsic factors. Intrinsic factors include the photoinitiator system, matrix, type and filler content, viscosity, color, and thickness. Extrinsic factors include intensity, light exposure time, the light spectrum, and tip distance of the light curing unit (LCU) to material.<sup>6</sup>

Inadequate polymerization reduces the physical/mechanical and biological properties of composite resins such as decreased elastic modulus and hardness, increased water absorption, discoloration elution of the possible irritant, toxicity, marginal breakdown, and edge leakage.<sup>4</sup> It has also been associated with postoperative sensitivity, microleakage, recurrent caries and pulpal irritation caused by residual monomers.<sup>2,9</sup> Poor physical properties and adverse biological reactions owes to leaching of the monomeric components of the uncured resin-composite.<sup>5</sup> Microhardness has been suggested as a way to examine the Depth of cure (DOC) of photo-activated resin composite. A value over 0.80 in bottom-to-top surface microhardness indicates adequate DOC.<sup>6</sup> Hardness is defined as the resistance of a material to indentation or penetration. It has been used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing teeth. The HV values are highly dependent on the size, weight, and volume of the filler particles.<sup>3</sup>

The polymerization quality of the composite resin can be assessed directly or indirectly. The direct method for assessing a degree of conversion include resonance imaging, optical microscopy, and Raman or Fourier transform infrared spectroscopy whereas indirect methods include visual inspection, surface hardness consisting of ISO 4049 scraping method, and Vickers microhardness ratio.<sup>6</sup>

In this study the Vickers microhardness method was used to evaluate the depth of cure of bulk-fill composite resin because it is easier to apply than other methods. The diamond indenter used in this

procedure does not deform over time and is reportedly suitable for measuring the hardness of fragile brittle materials. When Vickers hardness values are obtained, mean bottom/top ratio hardness value is determined to establish the depth of cure. This reflects the relative extent of conversion of the deeper surfaces in relation to the top surface.<sup>7</sup>

The DOC is the depth to which the light is able to cure the material. The use of thicker increments in bulk-fill resin composites is due to both developments in photoinitiator dynamics and their increased translucency, which allows additional light penetration and a deeper cure. DOC is dependent on filler (type, size, and load), chemical formulation of the organic matrix, distribution and amount of inorganic filler, light irradiance, the light source used, intensity, wavelength, light tip size, exposure time, and also resin composition and shade.<sup>6</sup>

The bulk-fill composite materials can be cured up to a depth of 4 to 5 mm. The increased depth of cure may be achieved through the use of novel proprietary resins, special modulators and unique fillers. The higher light transmission properties of the bulk fill composite is due to reduction of light scattering at the filler-matrix interface by either decreasing the filler amount or increasing the filler size. Moreover, differences in the refractive indices between the fillers and the organic matrix of the RBC materials also affect their translucency.<sup>7</sup>

Low DOC indicates the low polymerization quality that a lot of free or unreacted monomer during the polymerization process. The presence of unreacted monomer and their insufficient conversion at depth within the RBC bulk may also attenuate the irradiating light, preventing the formation of free radicals and thus reducing the DOC.<sup>3,6</sup>

MultiCore Flow showed least mean of difference between top and bottom surface followed by of Filtek Z250 Xt and G-aenial Universal Flo which suggested maximum depth of cure.

MultiCore Flow is a dual curing flowable composite. It is used for fabricating core build-ups using matrices up to 5 mm, in a single increment. The dual curing mechanism is: light curing and self-curing. It is a micro-hybrid 2-component composite consisting of a base and a catalyst paste. The two components are mixed during extrusion in a static mixer and the curing starts with a defined delay after the components are brought together. The benefit of dual-cure resin materials is the ability to bulk fill the core buildup material and/or lutes an opaque restoration while minimizing the risk of light attenuation that would disrupt the setting of the deepest portions of the resin material.<sup>3</sup>

Dual-cured resin-based composites were developed in an attempt to overcome limitations of both self-cured and light-cured materials by incorporating an oxidation-reduction (also known as "redox") initiator system in addition to photo initia-

tors. Superficial areas polymerize mainly through photoactivation, which results in rapid hardening of the resin for initial stabilization of the restoration, whereas the chemical setting modality is designed to ensure complete polymerization, even at deep portions of the material that have received insufficient light intensity.<sup>2</sup>

According to Vandewalker JP et al, the dual-cure resins which are not exposed to the appropriate amount of light or limited to only chemical curing may not obtain maximum mechanical properties due to a lower DC of the monomer. The chemical curing starts with a delay when the base and catalyst get mixed on extrusion in a static mixer. The material gets cured from the center and only 5-10 s light curing is required. Therefore, high levels of chemical curing compensate for attenuation of light energy in the deepest part of the restoration. The equal degree of polymerization within the entire depth of the materials may result in a uniform distribution of stress along tooth-material interfaces under load.<sup>3</sup>

The results of a study by Moraes and colleagues showed that for both activation modes, monomer conversion of dual-cured materials increased with time. Lee and colleagues described curing speeds that were up to about 320 times slower with chemical curing than with light curing. Deep portions of specimens relied mainly on self-curing might have benefited from the slow progressive hardening, even 24 hours after initiation of the polymerization reaction. Fonseca and colleagues reported a substantial increase in the degree of cure after 24 hours or more of self-curing for dual-cured resin cements. To ensure maximal double-bond conversion and to gain insight into the full extent of the progressive hardening potential of the self-curing mode, we extended setting times in the present study to one week. The dominant self-curing mode allows placement of the tested materials in areas that are inaccessible to a curing light.<sup>2</sup>

In the present study, Filtek Bulk Fill showed the second highest depth of cure value. Filtek Bulk-Fill posterior restorative material is a visible, light-activated restorative composite with special filler loading technology. In this material, the fillers are a combination of a non-agglomerated/non-aggregated silica filler, zirconia filler, and a ytterbium trifluoride filler. Filtek Bulk Fill was formulated with aromatic resins, which allowed the refractive index to more closely match the filler so the light would not significantly bend, and successfully transmitted through the material that increased the material's depth of cure. It is a Nanohybrid resin composites which have high translucency because the particles are smaller than the wavelength of light and cause minimal or zero scattering of photons.<sup>7,10</sup>

Filtek Z250 conventional composite also showed high microhardness; this may be due to the com-

position of its matrix that contains glass, quartz, and ceramic particles as fillers, enhancing its microhardness. The organic matrix of Z250 includes bis-GMA, bis-EMA, and UDMA. The latter has a higher content than the other two, which may explain the high DC of this composite.<sup>8</sup>

G-aenial Universal Flo, bulk-fill flowable exhibited large filler size with dominant polygonally shaped features compared with conventional flowable resin composites, as seen with a scanning electron microscope. The filler load was slightly increased, but the filler matrix interface was assumed to be decreased, due to the bigger size of the filler particle. Hence, it allows more curing light to transmit through the composite and improve the DOC.<sup>3,6</sup>

MultiCore Flow (IvoclarVivadent) showed least mean of difference between top and bottom surface followed by of Filtek Z250 Xt (3M, ESPE) and G-aenial Universal Flo (GC America) which suggested maximum depth of cure. ( $p < 0.05$ ).

Future studies are recommended to evaluate other properties of bulk-fill composite resins such as their wear resistance, fracture toughness, and effect of thickness on the passage of light.

## Conclusion

Multi Core Flow (IvoclarVivadent) showed maximum depth of cure when compared to other two bulk-fill resin composites. The materials used in this study are promising composite materials because they are useful in clinics for adhesive filling of deep cavities when time consuming incremental techniques are not possible due to low patients' compliance.

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