



EVALUATION OF EFFECT OF POLYMERIZATION TIME ON CURING DEPTH OF VARIOUS BULK FILL COMPOSITES-AN IN VITRO STUDY

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ABSTRACT

Objectives: From the time composite has been developed, it has been subjected to various changes in composition like addition of filler and initiators to yield better result. One such modification is the new bulk fill composite. However an acceptable polymerization time to maintain adequate curing depth has to be evaluated. So the aim of this in vitro study was to evaluate the effect of polymerization time on curing depth of four bulk fill flowable composites.

Methods: Total of 80 cylindrical specimens were prepared, and divided into 4 groups comprising of 20 specimens in each group. These selected groups were again subdivided into 4 groups comprising of 5 specimens in each group, based on their polymerisation time. The four specimens were Group A: SDR flow (DENTSPLY), Group B: Tetric N' Flow (Ivoclar Vivadent), Group C: Filtek bulk fill (3M) and Group D: Venus bulk fill (Heraeus Kulzer). The aluminium mold of 4mm depth and 5mm diameter was filled with composite and top surface was irradiated using Valo LED curing light with polymerisation time of 10, 20, 40, and 60seconds on respective subgroup. The microhardness of top and bottom surfaces were performed using Vickers Hardness tester under the load of 200gram for 15seconds. The depth of cure of each specimen was determined by hardness ratio method using the formula $\text{Hardness ratio} = \text{VK of bottom surface} / \text{VK of top surface}$. The results were statistically analysed.

Results: The study results showed that all the tested bulkfill composites can be cured to an acceptable depth. Tetric N' Flow exhibits maximum curing depth (95.28 ± 0.26) at 10sec polymerisation time. Venus bulk fill showed higher depth of cure at 20 and 40sec curing time (96.79 ± 0.39 and 97.56 ± 0.20). SDR flow showed adequate curing depth at 20 and 40sec curing time (92.94 ± 0.49 and 93.62 ± 0.22 respectively). Filtek bulk fill has maximum depth of cure at 40sec curing (89.10 ± 0.79).

Conclusion: Increasing polymerisation time increases the top as well as bottom micro-hardness of all tested bulk fill composites. Tetric N' Flow maximum curing depth at 10sec curing time. Filtek bulk fill is the material with least depth of cure.

KEYWORDS: Bulk fill, Resin composite, Curing.

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INTRODUCTION

Restoration of a carious primary tooth is important for the normal architecture of tooth. It helps in the physiologic development of permanent dentition and also for the normal psychological development of child.¹ There has been an expansion in the range of restorative materials available in Pediatric Dentistry. American Dental Association (ADA) Council on Dental Materials, Instruments and Equipment (CDMIE) provisionally approved the use of composites for primary molars in 1984.² But the main disadvantage of using a conventional composite material in pediatric dentistry is its incremental curing, which leads to incorporation of voids or contamination between composite layers, failure in bonding between layers, placement difficulties and extends of treatment time.³

Recently new category of flowable resin based composites (RBCs) called bulk fill was introduced by various manufacturers¹ The advantage of this newer material is that, it can be placed in 4mm depth rather than the current incremental placement technique, without negatively affecting the polymerization shrinkage, cavity adaptation or the depth of cure.^{4,5}

The depth of cure (DOC) is the depth to which light is able to harden the material.⁶ Inadequate depth of curing affects the physical as well as biological properties such as water absorption, discoloration, wear resistance, hardness and strength, leaching out of uncured monomer, marginal breakdown etc.^{7,8} In order to prevent these untoward effects composite resin has to be adequately cured in a proper depth.

There are different factors which influence the curing depth of visible light activated dental resin which include material filler content, shade, and translucency, intensity of curing light, exposure time, and curing tip distance. The mechanical properties of the resin composites are determined by total energy irradiation, which is clearly related to irradiation time chosen by the operator.^{9,10}

Several methods have been available to determine the depth of cure. One such technique is measuring the top and bottom surface microhardness of specimen.¹¹ Surface micro hardness has been used to evaluate indirectly, the extent of polymerisation and also efficiency of light cure.⁸ Vickers hardness measurement is one of the several suitable methods available for determination of surface microhardness. According to Professor David Watts of university of Manchester, an acceptable curing depth is achieved when bottom hardness corresponds to at least 80% of top surface hardness.¹²

Finishing the treatment within a short period of time with the aid of a reliable restorative material, helps to reduce patient's fear to a greater extent especially in pediatric dental patients. So in this in vitro study we focused on influence of curing time on depth of cure of four commercially available bulk fill flowable composites by using Vickers microhardness test.

MATERIALS AND METHODS

This invitro study was conducted in department of Pedodontics and preventive dentistry, Kannur Dental College in association with National Institute of Technology, Calicut. For the evaluation of depth of cure of bulk fill

flowable composites, 80 cylindrical shaped aluminum mold samples of 5mm diameter and 4mm height was prepared. These were randomly divided into four groups, comprising of 20 samples of each group. Group A (Surefil SDR Flow (DENTSPLY). Group B (Tetric N Flow bulk fill, Ivoclar Vivadent), Group C (Filtek bulk fill flowable composite (3M) and Group D (Venus bulk fill (Heraeus Kulzer). Each group was again divided into four subgroup based on different polymerisation time (10sec, 20sec, 40sec, 60sec).

During the sample preparation, the aluminium mold was positioned over an acetate strip on a glass plate. After composite resin insertion, a second acetate strip was placed on top of the mold with slight pressure to remove the excess material from the mold. Only the top side of the specimen was irradiated with LED device (VALO® LED Curing Lights) for polymerization time of 10, 20, 40 and 60sec for each material and will remove the acetate strip. Then samples was stored for 24hr in complete darkness at 37°C and 100% humidity before performing Vickers hardness test. The top and the bottom Vicker's hardness number of the samples was measured using micro hardness tester. A 200g load was applied through the indenter with a time of 15s. For each sample, four VHN readings will be recorded for the irradiated top and non irradiated bottom surfaces. The corresponding mean value and standard deviation was taken. The depth of cure for each specimen was determined by hardness ratio method using the formula, Hardness ratio = $\frac{VK}{VK}$ (Vicker's hardness) of bottom surface / VK of top surface.

Table 1: microhardness measurement of top surface

	SDR flow Mean(SD)	Tetric N' Flow Mean(SD)	Filtek bulkfill Mean(SD)	Venus bulkfill Mean(SD)	F#	P value
10sec	31.48(.148)	33.10(.255)	30.14(.182)	27.20(.255)	675.26	<0.001**
20sec	32.14(.195)	34.18(.277)	30.80(.20)	29.22(.286)	372.75	<0.001**
40sec	33.04(.152)	35.56(.305)	31.24(.207)	30.78(.192)	514.146	<0.001**
60sec	33.22(.164)	35.74(.241)	32.12(.179)	32.48(.179)	357.601	<0.001**

**Significant at 0.001 SD-Standard deviation #-Fisher exact test

Table 2: microhardness measurement of bottom surface

	SDR flow Mean(SD)	Tetric N' Flow Mean(SD)	Filtek bulkfill Mean(SD)	Venus bulkfill Mean(SD)	F	P value
10sec	23.12(.148)	31.54(.297)	21.34(.207)	23.38(.148)	2287.615	<0.001**
20sec	29.88(.164)	32.04(.305)	23.18(.148)	28.26(.309)	1196.905	<0.001**
40sec	30.96(.134)	33.06(.365)	27.78(.217)	30.04(.240)	374.188	<0.001**
60sec	31.08(.130)	33.14(.288)	28.72(.492)	31.58(.192)	176.837	<0.001**

**significant at 0.001

Table 3: comparison of depth of cure of different composites at polymerisation time 10sec

Composites	Mean	SD	N	F	Sig.	Tukeys HSD test		
						Pair	Mean difference	P value
SDR flow (A)	73.42	0.217	5	4,880.45**	0.001	A & B	21.86	<0.001 vhs**
Tetric N' Flow (B)	95.28	0.268	5			A & C	2.34	<0.001 vhs**
Filtek bulk fill (C)	71.08	0.531	5			A & D	16.2	<0.001 vhs**
Venus bulk fill (D)	89.62	0.427	5			B & C	24.2	<0.001 vhs**
						B & D	5.66	<0.001 vhs**
					C & D	18.54	<0.001 vhs**	

** -significant at 0.001 vhs-very highly significant

STATISTICAL ANALYSIS

The data was statistically analysed using descriptive statistics, Analysis of Variance(ANOVA) with

Fisher exact test”, “Bonferonni t test” and “Tukeys HSD test” in SPSS software version 17.0. The results were considered statistically significant at 0.001 probability level.

RESULTS

SURFACE MICROHARDNESS

The mean top microhardness value and standard deviation of each group measured at different polymerization time are presented in table 1. Statistical significant differences (P<0.001) between the mean value was observed with Tetric N' Flow showing highest hardness value. Top hardness value of the materials were in the following order. Tetric N' Flow > SDR flow > Filtek bulkfill > Venus bulk fill.

Table 2 shows mean microhardness value of bottom surface of all four bulk fill flowable composite at different polymerisation time (10, 20, 40 and 60 sec). At 10sec curing, mean bottom surface hardness values were in the order Tetric N' Flow > Venus bulkfill = SDR flow > Filtek bulk fill. At 20sec the mean value was Tetric N' Flow > SDR flow > Venus bulk fill > Filtek bulk fill. At 40sec Tetric N' Flow > SDR flow = Venus bulk fill > Filtek bulk fill and at 60sec it was found that Tetric N' Flow > Venus bulk fill = SDR flow > Filtek bulk fill.

DEPTH OF CURE

The results of depth of cure of different bulkfill flowable composites at 10sec are shown in table 3. While comparing the mean depth of cure of different groups at 10sec, the mean depth of Filtek bulk fill is 71.08, and that of Tetric N' Flow has the maximum of 95.28 and the difference of the mean depth of cure among the four groups was found to be statistically highly significant. Inter comparison between the groups was done by Tukeys test and all the inter comparisons between the groups are significant

Table 4: comparison of depth of cure of different composites at polymerisation– time 20sec

Composites	Mean	SD	N	F	Sig.	Tukeys HSD test		
						Pair	Mean difference	P value
SDR flow (A)	92.94	0.498	5	3,196.243**	0.001	A & B	0.78	0.178
Tetric N' Flow (B)	93.72	0.342	5			A & C	17.68	<0.001 vhs**
Filtek bulk fill(C)	75.26	0.195	5			A & D	3.85	<0.001 vhs**
Venus bulkfill (D)	96.79	0.439	5			B & C	18.46	<0.001 vhs**
						B & D	3.07	<0.001 vhs**
						C & D	21.53	<0.001 vhs**

**-Significant at 0.001 vhs-very highly significant

Table 5: comparison of depth of cure of different composites at polymerisation– time 20sec

Composites	Mean	SD	N	F	Sig.	Tukeys HSD test		
						Pair	Mean difference	P value
SDR flow (A)	92.94	0.498	5	3,196.243**	0.001	A & B	0.78	0.178
Tetric N' Flow (B)	93.72	0.342	5			A & C	17.68	<0.001 vhs**
Filtek bulk fill(C)	75.26	0.195	5			A & D	3.85	<0.001 vhs**
Venus bulkfill (D)	96.79	0.439	5			B & C	18.46	<0.001 vhs**
						B & D	3.07	<0.001 vhs**
						C & D	21.53	<0.001 vhs**

**-Significant at 0.001 vhs-very highly significant

Table 6. Comparison of depth of cure in different composites at polymerisation time 60 sec

Composites	Mean	SD	N	F	Sig.	Tukeys HSD test		
						Pair	Mean difference	P value
SDR flow (A)	93.54	0.182	5	90.922*	0.001	A & B	0.74	0.418
Tetric N' Flow (B)	92.80	0.274	5			A & C	4.52	<0.001 vhs*
Filtek bulk fill(C)	89.38	1.44	5			A & D	3.69	<0.001 vhs*
Venus bulk fill (D)	97.23	0.305	5			B & C	3.9	<0.001 vhs*
						B & D	4.43	<0.001 vhs*
						C & D	7.85	<0.001 vhs*

* - Significant at 0.001 level

(p<0.001). Depth of cure are in the following order Tetric N' Flow>Venus bulk fill>SDR flow>Filtek bulk fill.

Table 4 showing depth of cure at 20sec, When comparing the mean depth of cure between groups, the least value is

for Filtek bulk fill (75.26) and highest value for Venus bulk fill (96.79) The mean value and the difference of mean depth of cure among the four groups by applying analysis of variance (ANOVA) was found to be statistically significant.

Later inter comparison between the groups was done by Tukeys test and difference between SDR flow and Tetric N' Flow was only 0.78 and it was found to be statistically not significant. But inter comparison between all other groups were found to be highly significant. Depth of cure were found to be in the following order. Venus bulk fill>Tetric N' Flow=SDR flow>Filtek bulk fill.

Results from table 5, shows the comparison of depth of cure of different composites at polymerisation time 40sec. and the least value is for Filtek bulk fill (89.10) and highest value for Venus bulk fill (97.58). The mean value and the difference of mean depth of cure among the four groups by applying analysis of variance (ANOVA) was found to be statistically significant. Later inter comparison between the groups was done by Tukeys test and difference between SDR flow and Tetric n' flow was only 0.62 and it was found to be statistically not significant (p=0.268).But inter comparison between all other group was found to be highly significant. Depth of cure were found to be in the following order Venus bulk fill>SDR flow= Tetric N' Flow >Filtek bulk fill.

Comparison of depth of cure at 60sec is presenting at table 6. The mean depth of cure between four groups at 60sec curing time we could see that Filtek bulk fill is having the least value of 89.38 and maximum value for Venus bulk fill (97.23). The mean value and the difference of mean depth of cure among the four groups by applying analysis of variance (ANOVA) was found to be statistically significant. Later inter comparison between the groups was done by Tukeys test and difference between SDR flow and Tetric N' Flow was only 0.74 and it was found to be statistically not significant (p=0.418). But

intercomparison between all other group was found to be highly significant. Depth of cure were found to be in the following order Venus bulk fill>SDR flow= Tetric N' Flow >Filtek bulk fill.

DISCUSSION

Bulk fill flowable composites with improved mechanical and chemical characteristics have recently introduced to advance the restorative technique. Important physical properties of resin composites are surface microhardness and depth of cure and it plays an important role in characterizing dental restorative materials with a curing depth between 4-10mm.^{13,14} Surefil SDR flow, Tetric N' Flow bulk fill, Filtek bulk fill and Venus bulk fill were the composite materials used in this study.

The present study showed Irrespective of curing time used in this study, bottom surface microhardness value was found to be lower than the top surface for all tested materials. Flury et al¹⁵ measured the Vickers hardness of different composite at different distances ranging from 0.5 mm to 13mm and found that there was a gradual decrease in microhardness from the top towards the bottom. Cabellos et al¹⁶ also reported that a decrease in microhardness value with increased thickness of composite restoration This may be due to the reduction of light traveling through the composite material or may be due to light scattering through filler particles.¹⁷

With respect to polymerisation time, our study showed increased microhardness on increased polymerisation time. Previous studies¹⁸⁻²¹ also reports that increase in polymerisation time increases the

microhardness of composite which was similar to our study.

Comparative evaluation of microhardness shows that Tetric N' Flow bulk fill composite having significantly higher microhardness value at the top surface at all tested curing time This may be attributed to increased filler content (68.2%) when compared to others. Ilie and Stark et al found that mechanical properties of resin based composites are directly proportional to filler content.^{10,2} Increased filler loading has been shown to result in lower water sorption and higher resistance to toothbrush abrasion.²² Kim KH et al found that composite with highest filler by volume exhibited the highest flexural strength, flexural modulus and hardness.²³

With regards to top microhardnes value, our study observed that SDR Flow follows the Tetric N' Flow with all tested curing . The difference in hardness value between the materials can be due to composition of organic matrix, differences in the density of the polymer network or low filler content (as in Filtek and Venus) or increased particle size using other photo initiators, or greater percentage of filler. Differences in the equipment used, molds used during the sample preparation could be the reason for obtaining different results in different studies. Leprince et al²³ stated that there is a linear correlation between the surface microhardness and filler content and this correlation was highlighted by the result of this study.

In this study it was found that Tetric N' Flow bulk fill flowable composite has the highest depth of cure even at 10sec curing time when compared to other material and it achieves the standard depth of cure as stated by ISO 4049.²⁴ The highest depth of cure may be due to the presence of an

“initiator booster” (Ivocerin), besides having a regular camphoroquinone/amine initiator system. However, studies explaining the polymerisation mechanism or chemical nature of the initiator are few in number. When compared to tetric N-Ceram and Tetric Evo Ceram, Tetric N' Flow has the highest curing depth. When comparing to other bulk fill having the same initiator showed less curing depth. Inorder to achieve an adequate curing depth of 2mm, it has been reported that composite filling materials should exhibit a minimum of 80% bottom/top hardness percentage.¹¹ Similarly, in the current study, a similar percentage at 4mm depth was considered acceptable curing, and above 90% was considered high curing efficiency. In this regard, Tetric N' Flow shows high curing efficiency even at 10sec.

Venus bulk fill in this study also showed an acceptable depth of cure, because it exceeds the HV-80% even at 10sec polymerisation time. According to studies by Jang JH et al²⁵, among the bulk-fill composites, the bottom surface HV of SDR and Venus Bulk Filled composites , exceeded HV-80%. The favorable depth of cure result of SDR and Venus bulk fill might be attributed to the translucent matrix being highly conducive to light transmission and the incorporation of a functional photoactive group in the methacrylate matrix.

Filtek bulk fill (FBF) showed lower curing depth at 10sec and 20sec, but it had an acceptable curing depth at 40sec and 60sec curing time. Filtek Bulk Fill contains additional zirconia fillers which are said to improve mechanical properties. However due to its high refractive index zirconia is also said to reduce the transmittance of light in the

restorative materials thus may affect the depth of cure.²⁶

The variation in depth of cure between bulk fill composites may also be attributed to high percentage of wave lengths being absorbed near the top surface of the resin composite and not used to stimulate co-initiators at greater depths or because light scattering at particle interfaces and the difference in the ability of the photo initiators and any pigments to absorb the light.²⁷ Pigments are opaque particles which will limit the light penetration into the restoration and decrease the degree of polymerisation at greater depth.²⁸ Leprince et al²⁹ noted that adequate curing of the composite depends on the initiator receiving sufficient energy at correct wavelength.³⁰ In addition, filler content and size of the filler in resin composites may affect light penetration and it has a direct relationship with depth of cure.³¹

The polymerisation reaction of the dental composites is depends on deep penetration of light source to ensure adequate mechanical properties. There may be some barrier that prevents this penetration, including scattering and absorption of the light by the restorative material attenuating its potential to cure. The photo initiators also have an effect on penetration of the light as they act as a filter to specific wavelengths.³² All of these factors may explain the variation in depth of cure between the bulk fill composite and conventional composite and these variations have been reported specifically regarding bulk-fill composites.³³

It is very rare that the manufacturers and the suppliers of the materials provide a basic recommendation about depth of cure

and light intensities but usually they only provide the light exposure time. It is very important for the clinician to be aware of the depth of cure at specific activation times and light intensities that can help in planning placement technique that will ensure adequate cure of the bulk of the restoration.³⁴ Since it has been shown that some residual monomers can elute even from a well polymerized resin it can be assumed that more substances would be released from poorly polymerized resin at the bottom of the restoration.³⁵ These substances can harm the soft tissue; promote allergic reactions as well as stimulating bacterial growth.³⁶

Reduction of time and improvement of convenience associated with Bulk fill resin composite is a clear advantage of this particular class of material. However mechanical properties when compared to conventional composites are seems to be lower. Eventhough Bulk fill flowable resin composites have high curing depth, mechanical properties of most of them are lower when compared to high filled nanocomposite.^{37,38} So their use for restorations under high occlusal load is subjected to caution.

LIMITATIONS OF THE STUDY

This study was done under laboratory condition where the curing light was in direct contact with the restoration, which may not be possible to apply on the tooth as the anatomy of the tooth plays a role in affecting the amount of light entering the restoration. The storage of the specimens in this study may be differing from the clinical situation; where the material was used dry in this in vitro study.

Other limitations of study include non-comparison with a conventional composite. A further study has to be carried out in an in vivo condition to check the surface microhardness as well as depth of cure.

CONCLUSION

The study result showed that all four tested flowable bulk fill composite can be cured to a standard curing depth of 80% bottom to top ratio. Tetric N' Flow is the material having adequate curing depth with minimum polymerisation time Venus bulk fill is the material with highest curing depth. The study also shows increasing polymerisation time after 40sec has no effect on curing depth.

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