10.5005/jp-journals-10015-1563

ORIGINAL ARTICLE

Curing Effectiveness of Bulk-fill Composites

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ABSTRACT

Aim: The aim of this study was to assess the effectiveness of composite cure of many newly introduced bulk-fill composites

Materials and methods: Five types of dental composite materials of A2 shade were selected for this study. Three packable bulk-fill composites: Tetric N Ceram (Ivoclar Vivadent), Filtek bulk-fill posterior composite (3M ESPE) and X-trafill bulk-fill packable composite (VOCO) and two conventional composites: Quadrant Universal L.C Composites (CAVEX) and Composan Bio-esthetic Nano-ceram Composite (PROMEDICA). Ten specimens were prepared from each type of composite material, each composite specimen was prepared by compressing sufficient amount of composite into a mold (6 mm in diameter and 4 mm in thickness) for bulk-fill composites and (6 mm in diameter and 2 mm in thickness) for conventional composites. The total fifty composite specimens (n = 50) were cured for 20 seconds from the top surface only with Woodpecker LED light curing unit by making the curing tip in intimate contact with the acetate celluloid strips covering the composite surface. After that, the composite specimens were incubated in distilled water at 37°C for 24 hours. Then the 50 composite specimens were tested with Vickers microhardness tester (Microhardness tester FM-800, FUTURE-TEC H, Japan) at 300 g load and 15 seconds according to ISO 4049 for both top and bottom surfaces by making three indentations of both surfaces and considering the mean microhardness value for each surface. The hardness ratio of each specimen was calculated by using the formula ($HR = \frac{\text{Mean Vicker's hardness of the bottom}}{\text{Mean Vicker's hardness of the top}} \times 100$). Data were statistically analyzed using mean, standard deviation, one way Analysis of variance (ANOVA) and T-test at 5% level of significance.

Results: Statistical analysis of the data revealed, there was a significant difference between the five groups being tested (ANOVA, $p \leq 0.05$). The material with the highest hardness ratio was Cavex followed by X-trafill, Composan, Filtek bulk-fill packable and Tetric N ceram which did not achieve adequate curing (80% hardness ratio).

Conclusion: Composite formulation in general significantly affects the effectiveness of composite cure.

Clinical significance: Inadequate curing of composite might be considered one of the main causes behind composite restoration failure by negatively affecting its physical and mechanical properties including solubility, bonding qualities and more residual monomers production that are considered highly toxic to pulpal tissue. All these problems can be avoided clinically by former assessment the effectiveness of curing related to that specific composite material being selected.

Keywords: Bulk-fill composite, Conventional composite, Microhardness, Surface hardness.

How to cite this article: Al-Shekhli AAR, Al-Aubi I. Curing Effectiveness of Bulk-fill Composites. World J Dent 2018;9(5):361-365.

Source of support: Nil

Conflict of interest: None

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INTRODUCTION

Direct composite restorations and adhesive techniques revolutionized modern dentistry by maximizing working time, minimizing setting time as well as maintaining patient's esthetic.

Setting reaction of dental composite resulted from a chemical reaction between dimethacrylate resin monomers that produces a rigid and heavily cross-linked polymer network surrounding the inert filler particles.[1] The extent of this reaction often referred to adequate curing or degree (effectiveness) of cure which became an important issue in photocuring that dictates many physical and mechanical properties of the composite restoration.[2] Inadequate curing has been associated with inferior mechanical properties, higher solubility, retention failures and adverse pulpal responses due to residual monomers.[3] Packable composites might be an alternative to conventional composite in terms of the convenience of the placement, but no evidence shows clinical properties better than that of conventional composites.[4,5] Bulk-fill composites are new composite materials aimed to decrease the time taken to place the composite in the cavity by reducing the layers that have to be cured. They are also intended to decrease the shrinkage and the resulting stress by using the same exposure time and light intensity used for the regular composites.[6] This is made possible by either a reduction in the filler content (Bulk-fill flowable composites), altering the filler matrix composition to improve the translucency of the material or by improving the photoinitiator system.[7] In spite of the several advantages inherited from bulk-fill composites in avoiding all the clinical problems that might be associated with the use of incremental layering technique like bonding failure, contamination between composite layers, limitation to access in the small cavities leads to difficulty in placement, time-consuming including placement of the composite in increments and curing it,[8] there are many disadvantages associated with the use of bulk-fill composites ranging from shrinkage stress that might be more when bulk-fill composites are used, to incomplete polymerization of these composites in relatively deep cavities[9] probably because of light intensity reduction as the light penetrates through the thickness of the restorative material (4–5 mm for a single composite increment) as a result of its scattering, absorption, and reflection by the restoration, thus, decreasing the degree of cure at the bottom surface and its adverse effect on the general properties of the bulk-fill restorations that tend to be similar or lower than the traditional composites even when cured in 2 mm thickness.[7]

The degree of composite cure may be evaluated in two ways direct and indirect. Direct ways that evaluate the degree of composite cure, such as infrared spectroscopy and laser Raman spectroscopy, have not been considered for routine use since these ways are considered complicated[10] in comparison with indirect ways like visual, scrapping and microhardness testing. Incremental microhardness testing has been shown to be an effective way to assess the degree of curing.[11] Hardness ratio is equal to bottom divided by top multiplied by 100. A hardness ratio (HR) above 80% has been suggested as a minimum threshold accepted value.[12] Briefly, the minimum acceptable Hardness ratio is that the bottom surface can be about 80% of the upper hardness value to consider that the composite is being adequately cured.

The aim of the current study was to assess the degree (effectiveness) of composite cure of bulk-fill (4 mm thickness composite specimen) and traditional composites (2 mm thickness composite specimen) by using Vicker's micro-hardness testing devise and micro-hardness bottom to top ratio (HR).

MATERIALS AND METHODS

Five types of direct composite restorative materials (A2) shade was selected in the current study. Three packable bulk-fill composites: Tetric N Ceram (Ivoclar Vivadent AG, Schaan/Liechtenstein), Filtek Bulk fill Posterior composite (3M ESPE, USA) and X-trafill Bulk-fill packable composite (VOCO, Germany) and two traditional composites: Quadrant Universal L.C Composites (CAVEX, Netherlands) and Composan Bio-esthetic Nano-ceram Composite (PROMEDICA, Germany). Ten specimens were prepared from each type of composite resin brand, each composite specimen was prepared by compressing a sufficient amount of composite into a metal mold (inner diameter was 6 mm, and thickness was 4mm) for bulk-fill composites and (inner diameter was 6 mm and thickness was 2 mm) for traditional composites (Fig. 1). The composite material was placed in the mold with a plastic instrument and pressed between two cover slides and two acetate celluloid strips in between. Any excess material was extruded by a gentile pressure hand to ensure that the exposed surface of the composite was flat and parallel to the surface of the mold. The total fifty composite specimens (n = 50) were cured for 20 seconds at 1700 mW/cm^2 (calibrated always with radiometer before each curing procedure) from the top surface only with Woodpecker LED light curing unit (Guilin, Guangxi, China) by making the curing tip end in intimate contact with the acetate celluloid strips covering the composite surface. After that, the composite samples were stored in distilled water at 37° C for 24 hours. Then the 50 composite specimens were tested with Vickers microhardness tester (Microhardness tester FM-800, FUTURE-TEH, Japan) at 300 g weight for 15 seconds according to ISO 4049[13] for the top and bottom surfaces by making three indentations of both surfaces and considering the mean microhardness value for each surface. Each specimen placed on the stage of the micro-hardness tester and a magnification of 40 X was used to bring the center of the composite disc into focus to locate a smooth surface, devoid of voids or other irregularities. The indenter was automatically moved to indent the specimens, indentations were made on top and the bottom surface of each specimen and Vicker's hardness number (VHN) was calculated accordingly (Fig. 2).

The hardness ratio of each specimen was calculated by using the formula ($HR = \frac{\text{Mean Vicker's hardness of the bottom}}{\text{Mean Vicker's hardness of the top}} \times 100$). Data were statistically analyzed using mean, standard deviation, One Way ANOVA and T-test at 5% level of significance.

Fig. 1: The metal mold of 6 mm in diameter and 4 mm in thickness used with bulk-fill composites in this study with a small groove indicating the top surface

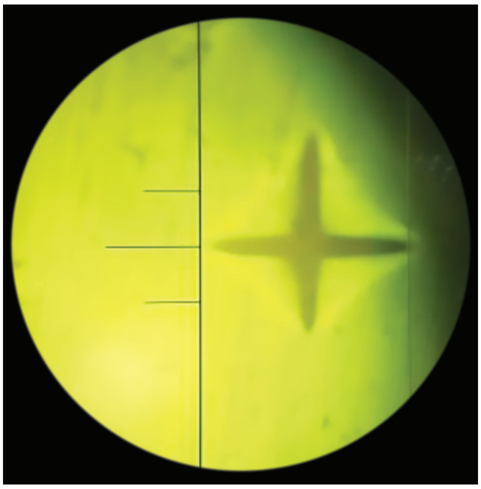
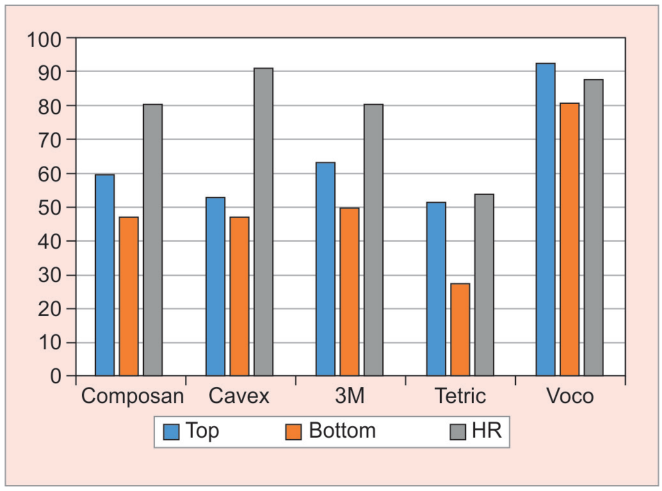


Fig. 2: Surface indentation on composite specimen surface under magnification of 40X



Graph 1: Mean VHN at the top, bottom and mean hardness ratio of the five composite groups being tested

RESULTS

Mean VHN and standard deviation at the top, bottom and mean hardness ratio of the five composite groups being tested in this study are listed in Table 1. Graph 1 represents mean VHN at the top, bottom and mean hardness ratio of the five composite groups being tested.

One-way analysis of variance (ANOVA) for hardness ratios of all the five composite groups being tested revealed that there was a statistically significant difference ($p \leq 0.05$) in hardness ratios as shown in Table 2.

Further analysis of the data with t-test indicated that there was a statistically significant difference in hardness ratios between all the 10 pairs of the five groups ($p \leq 0.05$) except between pairs no. 2, 4, 5, 7 and 9 that showed no significant differences between them (Table 3).

DISCUSSION

Surface micro-hardness and depth of cure are considered to be important physical properties of resin composites and play a significant role in the final clinical success of any composite restoration. The possibility of insufficient monomer conversion and the limitations of depth of curing are considered serious problems associated with photo-polymerized resin composites.[14] Depth of cure measurements has shown an indication to the clinical aspects of composite curing.[15] With the newer bulk-fill composites that are specifically designed to be placed in increments of 4mm might not achieve adequate depth of cure.

Several factors related to composite formulation were reported to affect the surface hardness of a resin composite restorative material. It was reported that the size and distribution of filler particles have a significant effect on some physical and mechanical properties, including surface hardness.[16] It was also mentioned that other parameters such as particle shape and density, monomer type and ratio, the degree of polymers crosslinking, and

Table 1: Mean VHN and standard deviation at the top, bottom and mean hardness ratio of the five composite groups

Groups	*G1*	*G2*	*G3*	*G4*	*G5*
Composite	*Composan*	*Cavex*	*3M*	*Tetric*	*Voco*
		Top			
Mean	59.38	52.44	63.18	50.99	92.28
SD	6.19	4.99	7.59	3.54	7.29
		Bottom			
Mean	46.99	47.4	49.58	27.22	80.62
SD	2.92	3.41	8.6	3.85	5.79
		Hardness ratio (HR)			
Mean	79.95	90.86	79.83	53.61	87.53
SD	10.56	8.25	17.19	8.82	5.85

Table 2: One-way analysis of variance (ANOVA) for hardness ratios of all the five composite groups

Source	*SS*	*df*	*MS*	*F*	*p*
Between-treatments	8575.9032	4	2143.9758	18.25192	< 0.00001
Within-treatments	5285.96	45	117.4658		
Total	13861.8632	49			

Table 3: t-test of the hardness ratio between different pairs of the five groups

Pair No.	*Pair of groups*	*t*	*Critical value*
			2.101
1	G1 X G2	–2.5742	Sig.
2	G1 X G3	0.0188	Not sig.
3	G1 X G4	6.0537	Sig.
4	G1 X G5	–1.985	Not sig.
5	G2 X G3	1.829	Not sig.
6	G2 X G4	9.7516	Sig.
7	G2 X G5	1.0405	Not sig.
8	G3 X G4	4.2909	Sig.
9	G3 X G5	–1.3406	Not sig.
10	G4 X G5	–10.130	Sig.

photo-initiators seem to have a significant influence on surface hardness.[17] The findings of this study indicated that, there was a strong relationship between hardness, hardness ratio and the amount of filler loading by volume (Table 4) since Voco composite used in this study exhibited the highest hardness values and hardness ratio and was the highest composite in the amount of filler loading (70.1% by volume) on one hand and Tetric composite in this study exhibited the lowest hardness values and hardness ratio and was the lowest composite in the amount of filler loading (53–55% by volume) on the other hand and the other three composites being tested in this study showed intermediate hardness values and hardness ratios related to their intermediate amount of filler loading by volume (56- 60%) (Table 4). Bulk-fill composites consist of ceramic fiber resin incorporated into the elongated filler network of about 100 nm in length.These materials have an increased depth of cure of up to 5 mm.[18] They are composed of light cured, dimethacrylate resins with a higher percentage of irregular (a mixture of irregular particles and glass rods) or porous fillers loading [19] in these composite resins varies from 60-80% by volume (Table 4). The ceramic fiber resin and glass rods might be the main source of light transmission through the bulk of the material in addition to the improved photo-initiator system of such composites.[7] As a general observation in this study, and regardless the character of the composite whether bulk fill or conventional, four out of the five composites being tested in this study, have approximately achieved the minimum accepted effectiveness of composite cure through their hardness ratios (Two of them approximately 80%) in spite of the differences in composite specimen thickness between conventional and bulk filled composites by 2 mm. Tetric N ceram in this study, has failed to achieve the minimum accepted effectiveness of composite cure through its hardness ratio (53.61%) that might be attributed mainly to the very low mean VHN of the bottom surfaces (Table 1) in comparison with the top VHN means indicating that the light intensity was inadequate to cure the bottoms of the specimens within the 20 seconds time interval (although the recommended curing time by the manufacturer is 20 seconds when light intensity output ≥ 500 mW/cm^2 and 10 seconds when light intensity output ≥1000 mW/cm^2) due to high composite resistance to light penetration since there was no possibility for light intensity fluctuations in photo-curing between the groups or individual specimens of specific group because of continuous calibration with radiometer before any curing procedure. The reduced light intensity reaching the bottom surfaces of tetric N ceram bulk-fill composite might be related to the incorporation of barium aluminum silicate glass and spherical mixed oxides in its filler composition as mentioned in Table 4. Clinically there are two options to overcome this problem either by using it in 2 mm composite increments and to be treated clinically as conventional composites and in this way it will not be eligible to continue as bulk-fill composite or by doubling or tripling the curing time (40–60 seconds for 4 mm composite increment) from buccal or the lingual/palatal aspects beside the occlusal after removing the matrix, if no Bluephase polymerization light was used or the light probe cannot be ideally positioned as recommended from manufacturer's instructions or combination of both approaches. Because of ivocerin new photoinitiator incorporated in the material as polymerization booster (manufacturer's data) has failed to achieve adequate curing for the specimens bottom surfaces and our findings were not in agreement with the findings of Moszner [20] who stated that "Ivocerin gives a depth of cure of up to 4mm and a less curing time of 10 seconds (>1,000 mW/cm^2)". Our findings also were not in agreement with the findings of Al-Mansour [21] in the mean hardness ratio obtained for Tetric N Ceram composite material in their study (83.6) which achieved the adequate minimum degree of curing. However, our date agreed with the Garcia [22] who stated that "All materials tested had significantly less depth of cure than either manufacturers' claims or ISO 4049 scrape test results". The only possible excuse for tetric N ceram for not achieving the minimum accepted effectiveness of composite cure in our study is that, the ivocerin new

Table 4: Comparison of materials properties provided by manufacturers

Property	*Composan*	*Cavex*	*3M*	*Tetric*	*Voco*
Filler vol %	56%	60%	58.4%	53–55 %	70.1%
Filler size	Not mentioned	Microfill	Nanofill	Nanofill	Microfill
Filler type and particle size	Special Nano and Ceramic filler particles	Silica, silicate glass and fluoride containing filler	20 nm silica filler, zirconia filler, an aggregated zirconia/ silica cluster filler, ytterbium tri-fluoride filler consisting of agglomerate 100 nm particles	Barium aluminium silicate glass, Ytterbium Fluoride, spherical mixed oxides	SiO2, glass, oxide
Resin type	Not mentioned	Methacrylate-based monomers	AUDMA, UDMA and 1, 12-dodecane-DMA	Bis-GMA, UDMA, Bis-EMA	Bis-GMA , UDMA, TEGDMA

photoinitiator wavelength was not compatible with that of Woodpecker LED light curing unit which was used for photo-curing all the composite specimens used in this study and tetric N ceram composite should be always cured with Bluephase polymerization light curing unit that seems to be more compatible for photo-curing the material from the aspect of wavelength or other secrets in the material because it is from the same manufacturer (Ivoclar Vivadent), and the manufacturer should recommend this important note in material instructions for use.

CONCLUSION

Within the limits of this study, we can conclude:

- The amount and type of composite filler loading significantly affect the effectiveness of composite cure.
- All the composite materials being tested in this study whether bulk-fill or conventional, had achieved adequate curing (80% hardness ratio) except tetric N ceram bulk-filled composite.

CLINICAL SIGNIFICANCE

Inadequate curing of composite might be considered one of the main causes behind composite restoration failure by negatively affecting its physical and mechanical properties including solubility, bonding qualities, and more residual monomers production that are considered highly toxic to pulpal tissue. All these problems can be avoided clinically by former assessment the effectiveness of curing related to that specific composite material being selected.

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