AJDS ALAZHAR
Al-Azhar Journal of Dental Science
Vol. 27- No. 2- 161:169- April 2024
Print ISSN 1110-6751 | online ISSN 2682 - 3314
https://ajdsm.journals.ekb.eg
Restorative Dentistry Issue *(Dental Biomaterials, Operative Dentistry, Endodontics, Removable & Fixed Prosthodontics)*

OPTICAL CHARACTERIZATION OF BULK-FILL RESIN COMPOSITES

Rana Abdelrehim Sedky[1*], Dena Safwat Mustafa [1]

ABSTRACT

Objective: This study was conducted to evaluate the light irradiance travelling through bulk-fill resin composites of different filler volumes and incremental thicknesses cured at different light curing distances. **Materials and Methods:** Forty specimens (n=5) were prepared according to the three levels of the study; filler volume of the bulk-fill resin composite; Filtek™ Bulk Fill Flowable Restorative (BF) and Filtek™ Bulk Fill Posterior Restorative (BP) (3M ESPE, St. Paul, MN, USA), increment thickness (2- or 4-mm thick increment) and light curing distance (zero mm or at 10 mm). Light intensity transmitted to the bottom of the specimen was monitored using a radiometer (P-9710, Gigahertz Optik GmbH, Germany) for 30 seconds from the start of light curing. Additionally, the light attenuation coefficient of bulk-fill materials was calculated. Specimens of 20mm diameter and 1 mm thickness were prepared to measure the contrast ratio for both bulk fill resin composite materials(n=5). Data was statistically analyzed at (α = .05). **Results:** Three- way ANOVA showed statistically significant difference of the three levels of the study for both transmitted light irradiance and light attenuation coefficient($p<0.0001$). The highest light intensity transmitted was for BR-2mm at Zero Distance (116.20±3.18) while least light transmitted with in BF-4mm. The mean contrast ratio calculated for BF was 0.95± 0.01 and for BR 0.97± 0.01(*p*=0.0170). **Conclusions:** Filler volume contributes to light attenuation of bulk-fill composites. Increment thickness and light curing distance should be accounted for to ensure proper curing of resin composite at its deepest portion.

KEYWORDS: Light attenuation, Bulk-fill Composites, Light curing, Filler volume, Increment thickness

INTRODUCTION

Bulk-fill composites were introduced to simplify restorative procedures. They allow clinicians to place composite resin in a quick and less tiring process than traditional incrementation. However, a valid concern regarding their depth of cure as well as degree of conversion surfaced, as they are both essential clinical standards to fulfill [(1)]. Any material without proper cure or conversion from monomer to polymer will not reach its optimum properties nor serve its purpose. Nevertheless, ongoing development brought about this family of materials based on the understanding of the interaction of light and matter as well as how polymerization shrinkage in resin composite works [(2)].

As a general rule, curing light that passes or is transmitted unrestricted through the resin composite can help the composite develop its properties, given compatible wavelength and efficient excitation. However, inherent to the formulation of any composite material, are two constituents of unequal refractive indices [(3)]. Both organic resin matrix and inorganic filler are included with varying proportions and sizes therefore affecting their viscosities as

1. Lecturer, Department of Operative Dentistry, Faculty of Dentistry, Ain-Shams University, Cairo, Egypt.

• ***Corresponding author***: ranaabdelrehim@asfd.asu.edu.eg

DOI: *10.21608/AJDSM.2023.253022.1479*

well as during the curing process, curing light will be absorbed and/or scattered based on the specific composition of the material [4,5].

In line with our understanding of polymerization kinetics, material translucency was proclaimed pivotal and key to the evolution of bulk-fill materials and the enhancement of cure depth. This was achieved by several intricate modifications to the resin composite to balance light transmission, cure and final mechanical and physical properties. Fundamentally, these modifications include the use of larger filler particle size, lower filler volume load, incorporation of highly light-reactive photo-initiator systems. Also, high molecular weight monomers and polymerization modulators were incorporated to help decrease polymerization shrinkage and resultant stresses [1,6].

Nevertheless, attenuation is inevitable by effect of material thickness or curing through hard dental tissues [7]. Basically, attenuation is the decrease in intensity of light as it travels away farther from the light source. Attenuation coefficient is a measure of the total loss of intensity of the light cure beam, as a function of scattering, and the interaction of light with the specific material composition [8].

Seldom do ideal clinical situations prevail when the light cure tip is in direct contact with the composite being cured. Instead, the norm involves a distance larger than zero from light source to surface due to tooth morphology and varying complexity of cavity preparation. This is further complicated by limited convenience and accessibility to the sites requiring cure, and light cure tip angulation to follow. Concerns are legitimate at the bottom surface of the increment farthest from light source rather than top surface. This is demonstrated clinically in multiple areas with the most common being gingival seat of proximal Class II cavity preparations as illustrated by Price et al.[9] Lack of cure can manifest in poor physical properties as well as multiple clinical failure sequelae as microleakage, secondary caries and fractures [10].

Frequently, focus goes to material property testing as well as long-term performance, yet the basic light emittance and how much is received by composite surfaces is overlooked. Therefore, it is worth investigating what lies beyond resin composite properties and performance, specifically bulk-fill materials of different viscosity and filler volume. Furthermore, the interplay of factors like increment thickness and distance from light curing tip to restoration surface are of special interest. The null hypothesis is that different filler volumes and incremental thicknesses of bulk-fill resin composites cured at different light curing distances does not influence the light irradiance through the material.

MATERIALS AND METHODS

Forty specimens (n=5) were prepared in this study according to the three levels of the study; filler volume of the bulk-fill resin composite, increment thickness and light curing distance. Two bulk-fill resin composites with different filler volume; hence viscosities, were used to prepare the specimens: Filtek™ Bulk Fill Flowable Restorative (BF) and Filtek™ Bulk Fill Posterior Restorative (BP); (3M ESPE, St. Paul, MN, USA). Two incremental thicknesses were used; 2- or 4-mm thick increment. Light curing of the resin composite was done either at zero mm curing distance from the top of the specimen or at 10 mm curing distances from the base of the specimen (i.e. 8mm away from the top of 2mm increment specimens and 6mm away from the top of 4mm increment specimens). Table 1 shows the materials used, their compositions and manufacturer.

TABLE (1) Chemical composition and manufacturer for materials used in this study

Material	Type	Shade	Resin	Filler loading (%)	Manufacturer
Filtek™ Bulk Fill Flowable Restorative, (BF)	Bulk-fill flowable/ low viscosity	A2	bisGMA, UDMA, bisEMA, Procrylat resins	64.5 (wt) 42.5 (vol)	3M ESPE, St Paul, MN, USA
Filtek™ Bulk Fill Posterior Restorative (BR)	Bulk-fill paste/ high viscosity	A2	AUDMA, UDMA and 1, 12-dodecane-DMA	76.5 (wt) 58.4 (vol)	3M ESPE, St Paul, MN, USA

Specimen preparation

An adjustable cylindrical white Teflon mold was used for 10mm curing distance specimens' preparation. The mold bottom 2- or 4-mm had a diameter of 4mm and was adjusted to accommodate resin composite specimens with 2- or 4- mm increment thickness respectively, while allowing a maximum height of 10mm from the light curing tip to the base of the mold. The top of the cylindrical mold had diameter of 10 mm to allow for accurate and reproducible positioning of the 10mm light curing tip. Another adjustable cylindrical white Teflon mold was used for zero curing distance specimens' preparation that allowed zero distance between the tip of the light curing unit and the top of the specimen.

Specimens were prepared using high-viscosity, bulk-fill composite (Filtek™ Bulk Fill Posterior Restorative) by carefully packing the resin composite inside the mold using a small ball burnisher in one 2- or 4-mm increment. Specimens prepared for low-viscosity, bulk-fill composite (Filtek™ Bulk Fill Flowable Restorative) by injecting the resin composite material inside the mold, using the manufacturer-provided injecting tip in single 2- or 4-mm thick increment.

Measurement of light irradiance transmitted during curing:

The uncured resin composite specimens were light cured using an LED light curing unit (Elipar™S10, 3M ESPE, St.Paul, USA), operating at 1200 mW/cm^2, turned on for 30seconds) either at zero distance (from the top of the specimen; ZD) or at 6mm from the top of the specimen in 4mm thick increment or 8mm from the top of the specimen for 2mm thick increments (10mm from the base of the increment;10mm). The light irradiance exiting at the base of the specimens during photopolymerization was continuously monitored for 30s from the start of light-activated curing of resin composite using a radiometer (P-9710, Gigahertz-Optik, Munnich, Germany). In order to record the measured irradiance, the radiometer was connected to virtual engineering software (LabVIEW, National Instruments Corporation, Austin, Texas, USA).

The light irradiance measured for all groups were analyzed using three-way ANOVA followed by student's unpaired t-test and One-way ANOVA with Tukey's multiple comparison test. The level of statistical significance was set at α=0.05.

Light attenuation coefficient of Bulk-fill resin composite:

In order to calculate the decay in light intensity within the specimen, attenuation coefficient (AC) was calculated using the measured light intensities at different thicknesses. AC was calculated based on Beer-Lambert law after modification to account for the specular reflection that occurs at the composite-air interface [11].

$$\mathbf{I=I_0(1-r)^2 \exp(-ki.x)}$$

I is the irradiance exiting from the bottom of the specimen, $\mathbf{I_0}$ is the output from the curing light in the absence of the specimen, **r** is the proportion of irradiance loss due to specular reflection, **ki** is the coefficient of light attenuation through scattering and absorption and **x** is the thickness of the composite material.

The value of r was determined to be 48.3% based on a previous study (11). I_0 was calculated by measuring the intensity of the light cure output at different distances (2mm, 4mm, 10mm) through the mold with no resin composite inside for 30 seconds (12). Moreover, the maximum intensity of the light cure output at zero distance was measured using the same radiometer. Three readings were recorded at each distance and the average was calculated.

The light attenuation coefficient was calculated for all groups and were analyzed using Three-way ANOVA followed by One-way ANOVA with Tukey's multiple comparison test. The level of statistical significance was set at α=0.05.

Contrast ratio of Bulk-fill resin composite:

Specimens of 20mm diameter and 1 mm thickness (n=5) were prepared by packing the resin composite material inside the mold. Mylar strips was placed on top of the uncured specimen and slightly pressed with a glass slide to produce uniform thickness. The specimen was light cured in three overlapping cycles to ensure adequate cure of the specimen. Specimens were stored in a lightproof container in deionized water and stored in an incubator at 37ºC for 24 hours. Color parameters for all specimens were measured using an instrumental spectrophotometer (Ocean Optics, Orlando, FL, USA) against white and black backgrounds under D65 light. Three measurements were recorded for each sample, and their average was calculated.

The contrast ratio for both Bulk-fill materials was calculated using the following equation:

CR = Yb/Yw

As the ratio of reflectance when the specimens were placed on the black background **(Yb)** to that of the same specimen when it was placed over the white background **(Yw)**. Contrast ratio ranges from 0 to 1 where 0 indicates a transparent material and 1 indicates an opaque material (13).

RESULTS

Light irradiance transmitted during bulk-fill resin composite curing:

Three-way ANOVA showed statistical significant difference to the three level of the study; filler volume, increment thickness and distance of light curing unit and their interactions (p<0.0001). Table 2 shows the mean, standard deviation, and statistical significance of the exiting light irradiance for all the groups. Highest light intensity transmitted was for BR-2mm at Zero Distance (116.20±3.18 mW/cm^2), while least light irradiance exiting was in BF-4mm cured at both zero and 10mm distance. A statistically significant decrease in the exiting light irradiance in all groups was noted when the increment thickness increased from 2mm to 4mm thick increment. At 2mm increment thickness there was a statistically significant higher light irradiance for BR compared to BF at both curing distances, while at 4mm increment thickness the difference was statistically significant only at zero distance. (BR: 36.45±3.30 mW/cm^2 vs BF: 20.65±2.79 mW/cm^2).

TABLE (2) Mean (standard deviation) and statistical significance of light irradiance transmitted in all groups of different filler volume, increment thicknesses, and light curing distances

Filler volume / Increment Thickness		BR		BF		p-value
		2mm	4mm	2mm	4mm	
Light Curing Distance	Zero Distance	116.20±3.18[a]	36.45±3.30[b]	58.54±1.00[c]	20.65±2.79[d]	<0.0001
	10mm Distance	63.33±3.73[a]	33.00±6.16[b]	50.34±3.35[c]	26.23±3.54[b]	0.0189
p-value		<0.0001	0.7653	0.0964	0.5289	

Different superscripts represent statistically significant difference within rows at p=0.05

Light attenuation coefficient of Bulk-fill resin composite:

Mean light cure intensity at 0mm (maximum light cure output), 2mm, 4mm and 10mm was as follows: 1274.11±3.93, 390.29±15.86, 287.48±5.14 and 205.35±0.24mW/cm^2 respectively. Figure 1 shows the recorded light irradiance exiting the bottom of the specimens throughout the 30sec curing time.

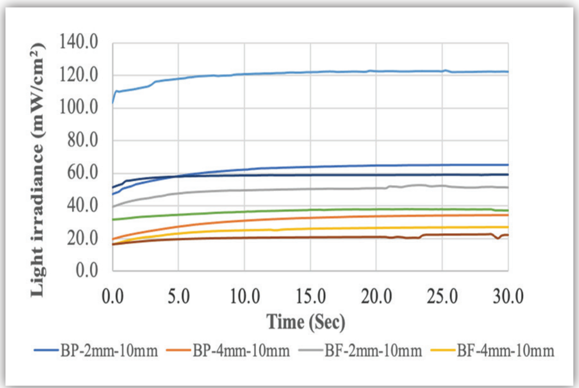

FIG (1) Light irradiance exiting the bottom of Bulk-fill resin composite specimens measured in mW/cm2

Three- way ANOVA showed statistical significant difference for the three level of the study; filler volume, increment thickness and distance of light curing unit and their interactions (p<0.0001). Table 3 shows the mean, standard deviation, and statistical significance of the light attenuation coefficient for all the groups. The estimated highest light attenuation coefficient (AC, mm^{-1}) was for 4mm increment thickness with no significance between both materials. While the lowest light attenuation coefficient was for BR-2mm group (0.949±0.001 and 0.947±0.003 mm^{-1}) at zero- and 10-mm curing distance respectively.

The light attenuation coefficient decreased as specimen's increment thickness increased from 2 to 4mm thick at both light curing distances. Given that the attenuation of light travelling in air was accounted for, the light curing distance did not have a statistically significant effect on the light attenuation coefficient for both materials except for BF-2mm group.

TABLE (3) Mean (standard deviation) and statistical significance of light attenuation coefficient in all groups of different filler volume, increment thickness, and light curing distance.

Filler volume		**BR**		**BF**		**p-value**
Increment Thickness		2mm	4mm	2mm	4mm	
Light curing distance	**Zero Distance**	0.949±0.001 a	0.997±0.000 b	0.974±0.000 c	0.998±0.000 b	<0.0001*
	10mm Distance	0.947±0.003a	0.996±0.000 b	0.958±0.002c	0.997±0.000b	<0.0001*
p-value		0.6105	0.9638	<0.0001*	0.8680	

Different superscripts represent statistically significant difference within rows at p=0.05

Contrast ratio of Bulk-fill resin composite:

The mean contrast ratio calculated for BF and BR was 0.95±0.01 and 0.97±0.01 respectively.Student t-test showed statistically significant difference between the contrast ratio of both materials (p=0.0170).

DISCUSSION

Curing resin-based restorations has always been an aspect of modern-day dentistry. A deeper understanding of polymerization and its true impact on material properties and performance puts huge emphasis on the quality of cure: how well a material '*can*' cure, and how well it '*actually has*' cured [5]. Bulk-fill materials promise curing in increments thicker than usual, in times shorter than expected, and even with polymerization shrinkage control [6].

Three-way ANOVA showed statistically significant effect of different filler volume, increment thickness and distance of light curing tip ($p<0.0001$). Therefore, the null hypothesis of this study can be rejected. Light intensity as recorded by the radiometer showed a consistent percent decrease relevant to material thickness, as shown in Table 2. Within 0mm distance from light curing tip groups, light intensity decreased by 65-68% from 2mm to 4mm thick increments. Meanwhile, within 10mm distance groups, intensity dropped by a constant 47%.

The highest light intensity recorded was for BP, in 2mm thickness, at zero distance, while least transmitted was with BF in 4mm thickness. Optical behavior of a material are a function of structural determinants like refractive index per filler and resin, the more different, the more the scattering of light. However, as composites cure, refractive indices of both constituents may approximate each other, allowing for better transmission of light [14]. Moreover, current results highlight the effect of resin monomer. According to Azzopardi et al, Bis-GMA, more readily available in Filtek™ Bulk-Fill Flowable Restorative, has been found to be significantly more translucent than TEGDMA & UDMA [15]. It seems that the novel resin system in BP (AUDMA, UDMA and 1, 12-dodecane-DMA) may have considerable translucency thus regulating depth of cure as claimed by manufacturer [16,17].

Light intensity readings exhibited are despite numerically higher filler volume %; (BP total inorganic filler load 58.4% vol, and BF at 42.5%vol.) creating multiple interfaces and consequent scattering. This differentiation was highlighted by Perieria et al stating that the amount of light transmitted is influenced by filler particles with regards to their size, shape as well as filler load [18]. Similarly, Rezaie et al explained that BP includes a combination of fillers of different sizes ranging from 4-11, 20 and 100nm [19,20]. Furthermore, this study demonstrates BF giving less intensity readings as attenuation is a function of both absorption and scattering. In this case, absorption by higher resin volume of BF compared to BP may have contributed to more light irradiance consumed and hence less light irradiance reaching the bottom of the specimen[14].

The mold used in this study allowed for reproducible measurement of radiant exposure actually received by the surface rather than full power density of the light curing unit (LCU). This simulates the clinical condition where narrow dimensions may occur in the proximal cavity portion in conjunction with buccal and lingual walls[21]. According to Haenel et al, the center of the light cure tip exhibits different light output compared to the sides or periphery of the same tip, hence best receiving to energy [10,21].

Still, the minimum threshold of light intensity must be fulfilled per increment, both top and bottom surfaces. Increasing LCU intensity alone regardless of other factors may cause rapid conversion and crosslinking within the polymer network but only at top surface without reaching the bottom[22]. Comparatively, increasing curing time may not necessarily compensate for proper power density received by surface or radiant exposure[5,23]. Our findings for the diminished output of LCU received by specimen as well as decay of light as it travels through air are consistent with previous studies. Price et al recorded a marked drop of light cure intensity from >1200 to <200 mW/cm^2 when measured at 0 and 10-mm distance respectively[9,24,25]. It was demanded that manufacturers clearly state the 'true power density' of their light curing units at both top and bottom surface, as the average distance between the light cure tip and the bottom of the cavity with a proximal portion to be in the depth range of 6.3±0.7mm. Displaying an inverse relation, power density decreases as the distance increases [9,24,26].

Increment thickness had a significant effect in all situations, where 2mm increment thickness recorded higher light intensity compared to 4mm increment thickness, for both BP and BF. This confirms the direct proportionality between increment thickness and light attenuation. In line, Gou et al stated that resin composite has different light attenuation coefficient between its cured and uncured states referring to a composite becoming more translucent as it cures[27]. This is similarly demonstrated in Figure 1, plotting light irradiance exiting the bottom of bulk-fill specimens against time, showing the characteristic initial increase in light intensity followed by plateau. Furthermore, the amount of energy photons reaching the radiometer can also be the function of the efficiency of the photo-initiator system relative to increment thickness as verified by Halvorson et al. [28,29].

Although bulk-fill resin composites are notorious for high translucency parameters [12], marked attenuation coefficient (AC) was shown in this study. A statistically significant difference was recorded between BP and BF in both increment thicknesses ($p<0.0001$), consistent with the light irradiance measured. Moreover, increasing the material thickness from 2mm to 4mm exponentially decreases the light irradiance travelling through the specimen and hence increasing its attenuation coefficient [12,27,30].

Meanwhile, no statistically significant difference was revealed between a single material AC at 0mm and 10mm ($p>0.05$), as the decrease in the light cure output by distance was accounted for by using the actual light cure intensity output reaching the radiometer in the absence of the specimen and hence ensure measuring the inherent material property solely [12]. It is worth noting that when assessing contrast ratio, a statistically significant difference was recorded between BP and BF. However, both materials recorded values close to 1 which indicates an almost opaque material when cured, this might have enhanced the light attenuation coefficient of the tested materials [31].

In this study, distance showed a statistically significant effect on transmitted light irradiance and light attenuation which is also clinically meaningful**.** Clinically, it is important during curing to take into consideration factors like cavity wall inclination, light cure tip angulation, as well as distance in the way of sufficient light reaching the composite with a homogenous beam profile [9,24,26].

In conclusion, curing is a complex and multi-faceted process. Sufficient curing must be ensured, taking into consideration all clinical challenges. Any high-end brand of light curing device or bulk-fill composite will not compensate for operator diligence to deliver proper power density and to keep distance to the minimum relative to restoration surface. The clinician is advised to choose the appropriate restorative material and ensure its light requirements are fulfilled as they change with material composition [32,33]. Last, increasing time is not a solution especially as it contributes to polymerization exotherm which may jeopardize pulpal health. Thus, manufacturer instructions must always be clear, specific, and appreciative of clinical situations and recommendations [4].

CONCLUSION

Within the limitations of this study, the following can be concluded:

1. Total filler volume as well as specific compositional and filler size characteristics contribute to light attenuation of bulk-fill composites.
2. Increment thickness and light curing distance should always be accounted for to ensure proper curing of resin composite at its deepest portion.

ACKNOWLEDGMENTS

Dr. Rana Sedky would like to sincerely thank 3Mgives for supporting her visit at University of Minnesota, USA through their sponsorship of the Key Opinion Leaders (KOLs) Program. Additionally, she is grateful to 3M OCSD (St. Paul, MN, USA) for providing the necessary materials for research. Dr. Rana Sedky would also like to express her deep appreciation to the dedicated staff at MDRCBB, University of Minnesota for their invaluable assistance throughout the completion of this project, especially Prof. Alex Fok (Director of MDRCBB), Dr. Wondwosen Aregawi and Dr. Brian Holmes.

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