

Affect of the Different Irradiation Time and Light Density on the Shear Bond Strength of the Bulk-fill Composites

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Abstract: *The objective of this study is in vitro examination of the dentin bonding values of bulk-fill composites under various light densities and irradiation times. Eighty-five extracted human molars were used. While Grandio (VOCO) was used in the control group, SDR (Dentsply), X-tra Base (VOCO), Quixfil (Dentsply) and X-tra Fil (VOCO) were used in the study groups. The bonding strength groups were divided based on the light density and irradiation time. The Max. Tensile values of control group were found statistically significantly higher than those of the studygroups ($p=0.042$, $p=0.005$) and no statistically significant between the study groups ($p=0.005$).*

Keywords: Dentin bonding, bulk-fill, dental composite, SDR, light density

1. Introduction

In today's restorative dentistry, conservative and esthetic restorations are made with composite restorative materials. Advancing technology has improved the physical and optical characteristics of composite materials. However, conventional composite materials should be applied in layers not exceeding 2 mm due to their polymerization properties. This procedure requires a large number of layering and polymerization particularly in deep cavities.

The factors causing failures in conventional composite restorations such as long treatment times due to layering techniques, formation of micro gaps between layers and risk of contamination are tried to be eliminated with the recently developed bulk-fill restorative materials (1). The layering technique allows bulk-fill applications of 4-5 mm at a time.

Due to reasons such as cross bond formation arising from polymerization kinetics, intermolecular gaps are reduced and polymerization shrinkage occurs (3). Polymerization shrinkage is one of the features of restorative materials that require improvement as it causes stress within the polymer formed and on the surface of the dental substrate as well as micro leaks.

Highly strong light and prolonged irradiation time increase the degrees of polymerization in the materials (2).

This study aims at testing the effect of the dynamics taking place on the adhesive surfaces and in the ingredients of the bulk-fill composite materials that were polymerized with a light density higher than that in the manufacturing company's instructions on dentin bonding strengths (DBS). The null hypotheses were tested as follows:

- 1) There is no difference between the dentin bonding performances of bulk-fill composites and conventional composites.
- 2) Increased light density will not affect the DBS values.

2. Materials and Methods

This study is a part of the doctoral thesis named "Evolution of the effects of the different light intensities and curing times on the shear-bond strength and microleakage of the bulk-fill composite systems" and was approved on 26.09.2014 by the Non-Interventional Clinical Trials Ethics Committee of T.R. Istanbul Medipol University with the committee decision numbered 10840098-245. Extracted molars were used in our study. Decayed, restored, or cracked teeth and those that have not completed root development were excluded from the study. After cleaning them from residues with an ultrasonic cleaner, the teeth collected were disinfected by keeping them in 0.5% chloramine T solution for 24 hours and were kept in distilled water. Prepared in this way, the teeth were used in the study within at most 6 months from the date of their extraction. The 85 molar teeth to undergo shear bond strength testing were embedded in self-curing cold acrylic (BMS Dental, Italy) in silicon molds with their crowns outside. The teeth were then taken out of the molds and prepared horizontally from 3 mm cervical of their tubercle tips with an Isomet precision cutting device. To form a homogeneous and standard smear layer on dentin surfaces, 300, 600, and 800 grid silicon carbide sandpapers were used. The prepared 85 teeth were randomly divided into 17 groups, each group containing 5 teeth.

Table 1: Study groups

Group	Adhesive	Material	Light Density	Irradiation Time
1	Futurabond DC	Grandio	1000 mW/cm ²	10 sec
2	Futurabond DC	X-tra Base	1000 mW/cm ²	10 sec
3	Futurabond DC	X-tra Base	1000 mW/cm ²	20 sec
4	Futurabond DC	X-tra Base	1400 mW/cm ²	10 sec
5	Futurabond DC	X-tra Base	1400 mW/cm ²	20 sec
6	Futurabond DC	X-tra Fil	1000 mW/cm ²	10 sec
7	Futurabond DC	X-tra Fil	1000 mW/cm ²	20 sec
8	Futurabond DC	X-tra Fil	1400 mW/cm ²	10 sec
9	Futurabond DC	X-tra Fil	1400 mW/cm ²	20 sec
10	Xeno V+	SDR	1000 mW/cm ²	10 sec
11	Xeno V+	SDR	1000 mW/cm ²	20 sec

12	Xeno V+	SDR	1400 mW/cm ²	10 sec
13	Xeno V+	SDR	1400 mW/cm ²	20 sec
14	Xeno V+	Quixfil	1000 mW/cm ²	10 sec
15	Xeno V+	Quixfil	1000 mW/cm ²	20 sec
16	Xeno V+	Quixfil	1400 mW/cm ²	10 sec
17	Xeno V+	Quixfil	1400 mW/cm ²	20 sec

A control group and 16 test groups were formed by randomly selecting the prepared extracted tooth samples. Futurabond DC (VOCO, Cuxhaven, Germany) was used as an adhesive for the samples where VOCO brand products were used and Xeno V⁺ (Dentsply, Kontaz, Germany) for the samples where Dentsply brand products were used. The bonding agents were applied in line with the manufacturing companies' instructions.

Conventional nanohybrid Grandio[®] A3 (VOCO, Cuxhaven, Germany) was used in the control group in our study. X-tra Fil (VOCO, Cuxhaven, Germany), X-tra Base (VOCO, Cuxhaven, Germany), QuixFil (Dentsply, Kontaz, Germany) and SDR (Dentsply, Kontaz, Germany) were used in the test groups in our study.

When preparing the shear bond strength samples, the conventional composite Grandio[®] A3 (VOCO, Cuxhaven, Germany), which was used in the control group, was placed in silicon molds in 2-mm layers, whereas the materials used in the test groups were placed in 4-mm layers with the bulk-fill technique.

The prepared dentin surfaces of the samples were washed with pressured water for 10 sec and were dried with dry air for another 10 sec.

The adhesive agent prepared for each group was applied on the dentin surface for 20 sec and polymerized with a VALO[®] (Ultradent) LED light polymerization device for 10 sec at its standard density mode, 1000 mW/cm².

The silicon mold was placed on the adhesive-applied surface and the test materials were placed in the mold in the form of 4-mm layers. After covering their outer surface with a transparent strip band, the composite resins were

polymerized for the combinations of 10 and 20 sec at the standard density mode, 1000 mW/cm², and the extra density mode, 1400 mW/cm², of the VALO[®] (Ultradent) LED light polymerization device.

After completion of their preparation, the shear bond strength samples were made subject to aging with a thermal cycler. The transition between the +5 and +55°C containers in the thermal aging device was done in 3 sec and the sample waiting time in the +5 and +55°C containers was 30 sec. The thermal aging process was completed in 10 000 cycles.

After thermal aging, the shear bond strength samples were tested with a Shimadzu AGS-5kNXD model universal testing device. The tests were applied with a 0.5 mm/min approach speed in line with the ISO standards. The values at the time of fracture were scored in units of Mpa. The dentin surfaces were also examined under a stereomicroscope with 40x magnification. The fracture types of the samples were classified as adhesive, cohesive, and mixed.

The statistical analyses were carried out with the Number Cruncher Statistical System (NCSS) 2007 Statistical Software (Utah, USA).

Alongside descriptive statistical methods (mean, standard deviation, median, and interquartile range), the Kruskal Wallis test was used for intergroup comparisons, the Dunn's multiple comparison test for subgroup comparisons, and the Mann-Whitney-U test for paired group comparisons. The results were assessed at p<0.05 significance level and 95% confidence interval.

3. Results

The mean values and standard deviations of all groups are shown in Tables 2 and 3. The highest dentin bonding strength (17.893±5.905 Mpa) was recorded in the control group (Group 1) and the lowest (5.16±3.12 Mpa) in the samples where X-tra Fil composite material was polymerized at 1400 mW/cm² light density for 10 sec (Group 8).

Table 2: Bonding strength assessments of the study groups.

Light density		10 (sec)	20 (sec)	p
SDR Group	1000 (mW/cm ²)	Mean±SD	6,9±2,08	0,494
		Median (IQR)	7,95 (5,17-8,12)	
	1400 (mW/cm ²)	Mean±SD	8,72±2,4	0,285
		Median (IQR)	8,18 (7,11-10,6)	
QuixFil Group	1000 (mW/cm ²)	Mean±SD	6,82±1,96	0,489
		Median (IQR)	6,01 (5,22-8,83)	
	1400 (mW/cm ²)	Mean±SD	5,81±1,95	0,016
		Median (IQR)	6,27 (4,08-7,32)	
X-tra Base Group	1000 (mW/cm ²)	Mean±SD	9,85±4,9	0,977
		Median (IQR)	8,5 (5,47-14,89)	
	1400 (mW/cm ²)	Mean±SD	8,88±1,46	0,422
		Median (IQR)	8,42 (7,63-10,36)	
X-tra Fil Group	1000 (mW/cm ²)	Mean±SD	4,79±1,95	0,422
		Median (IQR)	4,5 (3,23-6,5)	
	1400 (mW/cm ²)	Mean±SD	5,16±3,12	0,215
		Median (IQR)	3,9 (2,61-8,33)	

Table 3: Bonding strength assessment of the control group

			Entire Area Calculation	
20 sec	1000m W/cm ²	Control Group	Mean±SD	17,893±5,905
			Median (IQR)	19,14 (12,21-22,954)

When the density of the lighting device was set at 1000 mW/cm² and its irradiation time at 20 sec, a statistically significant difference was observed between the entire area calculation max tensile strength (Mpa) values of the SDR (Group 11), QuixFil (Group 15), X-tra Base (Group 3), X-tra Fil (Group 7), and Grandio (Group 1) groups (p=0.008). The entire area calculation max tensile strength (Mpa) values of Group 1 were found significantly higher statistically than those of Group 7, Group 11, and Group 15 (p=0.042,

p=0.005) but no statistically significant difference was seen between the Group 1 and Group 3 (p>0.05).

When the study groups were compared to each other on the basis of light density, Group 17 gave a significantly higher bonding value in statistical terms than Group 16 (p=0.016), whereas no statistically significant difference was found between the other groups (Tables 2 and 3).

When the study groups were compared on the basis of irradiation times, no statistically significant difference was found between the groups. The variable light density did not produce any statistically significant difference in the dentin bonding strength (Table 4).

Table 4: Assessment of bonding strength results with respect to polymerization times.

Material	Irradiation Time		1000 (mW/cm ²)	1400 (mW/cm ²)	p	
SDR Group	10 (sec)	Mean±SD	6,9±2,08	8,72±2,4	0,237	
		Median (IQR)	7,95 (5,17-8,12)	8,18 (7,11-10,6)		
	20 (sec)	Mean±SD	8,38±4,11	7,29±1,43	0,590	
		Median (IQR)	7,24 (5,88-11,45)	7,83 (5,83-8,47)		
	QuixFil Group	10 (sec)	Mean±SD	6,82±1,96	5,81±1,95	0,437
			Median (IQR)	6,01 (5,22-8,83)	6,27 (4,08-7,32)	
20 (sec)		Mean±SD	8,02±3,11	10,03±2,4	0,284	
		Median (IQR)	7,02 (5,78-10,75)	8,74 (8,36-12,35)		
X-tra Base Group	10 (sec)	Mean±SD	9,85±4,9	8,88±1,46	0,684	
		Median (IQR)	8,5 (5,47-14,89)	8,42 (7,63-10,36)		
	20 (sec)	Mean±SD	9,94±4,81	8,19±1,08	0,452	
		Median (IQR)	7,84 (5,81-15,1)	8,34 (7,13-9,18)		
X-tra Fil Group	10 (sec)	Mean±SD	4,79±1,95	5,16±3,12	0,829	
		Median (IQR)	4,5 (3,23-6,5)	3,9 (2,61-8,33)		
	20 (sec)	Mean±SD	5,97±2,42	7,38±1,98	0,341	
		Median (IQR)	6,78 (3,71-7,82)	6,79 (5,68-9,37)		

The groups were assessed with respect to fracture types as shown in Table 5.

Table 5: Distribution of fracture types of the materials by polymerization times and light densities.

			Control Group		SDR Group		QuixFil Group		X-tra Base Group		X-traFil Group		p
10sec	1000 mW/cm ²	Adhesive	0	0,00%	0	0,00%	2	40,00%	0	0,00%	1	16,67%	0,130
		Cohesive	0	0,00%	0	0,00%	2	40,00%	3	60,00%	2	33,33%	
		Mixed	0	0,00%	5	100,00%	1	20,00%	2	40,00%	3	50,00%	
	1400 mW/cm ²	Adhesive	0	0,00%	0	0,00%	3	60,00%	0	0,00%	0	0,00%	0,001
		Cohesive	0	0,00%	1	20,00%	0	0,00%	5	100,00%	1	20,00%	
		Mixed	0	0,00%	4	80,00%	2	40,00%	0	0,00%	4	80,00%	
20sec	1000 mW/cm ²	Adhesive	5	100,00%	1	20,00%	2	40,00%	0	0,00%	0	0,00%	0,0001
		Cohesive	0	0,00%	1	20,00%	0	0,00%	5	100,00%	1	20,00%	
		Mixed	0	0,00%	3	60,00%	3	60,00%	0	0,00%	4	80,00%	
	1400 mW/cm ²	Adhesive	0	0,00%	1	20,00%	0	0,00%	0	0,00%	0	0,00%	0,005
		Cohesive	0	0,00%	1	20,00%	0	0,00%	5	100,00%	0	0,00%	
		Mixed	0	0,00%	3	60,00%	5	100,00%	0	0,00%	4	100,00%	

When the irradiation time of the lighting device was set at 10 sec and its density at 1000 mW/cm², no statistically significant difference was observed between the fracture type distributions of the SDR, QuixFil, X-tra Base, and X-tra Fil groups (p=0.130).

When the irradiation time of the lighting device was set at 10 sec and its density at 1400 mW/cm², a statistically significant difference was observed between the fracture type distributions of the SDR, QuixFil, X-tra Base, and X-tra Fil groups (p=0.001). The presence of adhesive type was found high in the QuixFil group and the presence of mixed type in the SDR and X-traFil groups.

When the irradiation time of the lighting device was set at 20 sec and its density at 1000 mW/cm², a statistically significant difference was observed between the fracture type distributions of the Control, SDR, QuixFil, X-tra Base, and X-tra Fil groups (p=0.001). The presence of adhesive type was found high in the control group and the presence of cohesive type in the X-tra Base group.

When the irradiation time of the lighting device was set at 20 sec and its density at 1400 mW/cm², a statistically significant difference was found between the fracture type distributions of the SDR, QuixFil, X-tra Base, and X-tra Fil groups (p=0.005). The presence of adhesive type was found high in the SDR group and the presence of mixed type in the QuixFil and X-traFil groups.

4. Discussion

For composite resin to show an ideal polymerization, monomers need to convert into polymers at a maximum level. The residual monomers that did not turn into polymers will lead to imperfections in the material structure and the restoration will be a failure (4)(5)(6). Conversion of monomers to polymers depend on many factors including the thickness, chemical properties, transparency, and color of the composite resin placed into the cavity, the quality of the light source, and the method used (7)(8). The spectroscopic conversion rate analyses of the entire resin materials used in this study were at acceptable levels (9)(10).

The thickness of the composite resin placed into the cavity is among the main factors affecting the degree of polymerization. The upper surface of the resin receives sufficient light energy, but the light applied to the surface diffuses when passing through the composite resin bulk and its density, brightness, and curing efficiency diminish as it reaches the lower layers. Therefore, as the thickness of the composite resin increases, the irradiation of the light decreases depending on the distance it travels. Even minor increments in the restoration thickness create large differences in the amount of light energy transmitted through the restoration site, affecting the degree of polymerization. Placing composite resins into the cavity at most 2 mm in thickness is accepted as a standard to achieve a successful polymerization. It has been reported that when the thickness of the layer exceeds 2 mm in conventional composite resins the polymerization is endangered, the physical properties are worsened, and clinical life reduced (11)(12)(13)(14).

Referring to the sources in the literature, 4-mm Grandio condensable conventional nanohybrid composite samples were applied in this study in 2-mm layers for shear bond strength tests in the control groups. The LED light device was used for 20 sec at a standard density of 1000 mW/cm² for each layer during the preparation of control groups.

Another factor affecting polymerization is the light device used (15). We used a LED light device for the polymerization of samples in this thesis study due to its advantages such as having fixed output power throughout its life and not producing heat during light emission. Since the light it generates has the wavelength interval of 395-480 nm and it provides constant light for desired durations at 3 different light densities as Standard (1000 mW/cm²), High (1400 mW/cm²) and Extra (3200 mW/cm²), we preferred the polywave LED polymerization device of VALO in our study. The resin materials that were exposed to polymerization light at various densities and durations were examined for their shear bond strengths in this study.

The performances of light devices in polymerization formation depend on the density of the light they emit. Price et al. (16) have reported in their study that the light density required for sufficient polymerization of a 2-mm composite resin has to be at least 600 mW/cm² and Rueggeberg et al. (17) that it has to be at least 400 mW/cm². Recently, LED devices with light densities increased up to 3200 mW/cm² have been introduced to the market. With their high light densities, the new generation LED devices are argued to

improve the polymerization of composite resins (18). Rahiotis et al. (18) have reported that high density LED devices achieve better carbon double bond conversion than low density LED devices, leading to more successful physical properties in the composite resin. Al-Ahdal et al. (19) investigated the conversion rates and kinetics of 7 different bulk-fill composite resins after polymerization with Elipar™ S10, which allows polymerization under a light density of 1200 mW/cm². The materials, whose conversion rates were measured with FTIR after 20-sec polymerization times, produced different processes and percentages depending on the type of the material. They reported that all material samples prepared 4 mm in thickness showed conversion rates between 50 and 72% at the end of a 24-hour period. The densities of the LED light device used in this thesis study were 1000 and 1400 mW/cm², which were sufficient for a satisfactory polymerization as reported in the previous studies.

Another major factor determining the polymerization quality of a composite resin is the irradiation time. It has been reported that at least 20 sec of exposure to light is required for adequate polymerization of a 2-mm composite resin (20)(21). Haenel et al. (22) evaluated the effect of light density on the microhardness of resin surfaces and have reported that maximum polymerization is achieved in the 2nd second and 40% of the polymerization is completed between 3.6 and 5.7 seconds regardless of the light irradiation time. They have also reported that increasing polymerization time using light does not contribute to the homogeneity of hardness distribution, but increases the level of hardness. The light irradiation time proposed by the manufacturing companies for the composite resins used in this study, SDR, QuixFil™, X-tra Fil, and X-tra Base, was at least 10 seconds. Based on this information and for the purpose of setting a standard among the groups, all adhesives were exposed to a standard light power of 1000 mW/cm² for 20 sec. In order to see the effects of the differences in light density and duration, the restorative materials were exposed to light at standard and extra density modes for 10 and 20 sec.

The clinical success of a composite restoration depends on the adhesive system (23). Bonding of dental materials to the dentin effectively and for a long time has been investigated in depth in recent years, but the bonding strengths of adhesive materials to dental structures could not escape being criticized by investigators who used shear bond strength tests (24). A shear bond strength test is a simple method for testing the adhesion performance of dental adhesives (25). In this study, the dentin adhesion performances of moderately acidic 7th generation self-etch adhesives and bulk-fill composite restoration materials resulting from polymerization at various light densities and durations were tested using the shear bond strength testing method.

The assessment was made by measuring the amount of load falling on each unit area at the moment of fracture after applying stress or shear strength on the bonding surface. A review of the literature shows that the shear bond strength has been widely made subject to investigation (26)(27). The

shear strengths are also agreed to reflect the clinical setting better (28).

In their study where they investigated the microhardness and dentin bonding strength of bulk-fill composites in different thicknesses, Flury, Peutzfeldt and Lussi (29) tested the Filtek Supreme XTE conventional nanohybrid composite, SDR, Filtek Bulk Fill, X-tra Fil, and Tetric EvoCeram Bulk Fill composites in 2.4 and 6 mm thicknesses and found that a statistical decrease occurred in the microhardness values of the Filtek Supreme XTE and Tetric EvoCeram Bulk Fill groups when their thickness were increased ($p < 0.0001$), and that increased thicknesses did not produce any statistically significant difference in their microhardness values in the SDR, Filtek Bulk Fill, and X-tra Fil groups ($p = 0.10$, $p = 0.16$, $p = 0.18$). Similar to microhardness tests, a significant decline was seen with increased thickness in the dentin bonding strengths of the control groups ($p < 0.0001$). No statistically significant difference was observed in the dentin bonding strength in the SDR, Filtek Bulk Fill, X-tra Fil, and Tetric EvoCeram Bulk Fill study groups ($p = 0.26$), ($p = 0.11$), ($p = 0.55$), ($p = 0.11$); a decline in the dentin bonding strength with increasing thickness occurred only in the Tetric EvoCeram Bulk Fill material ($p = 0.11$) $21.0 = 20.7 = 19.0$ Mpa. As for fracture types, mostly cohesive fractures at the dentin level were observed. A cohesive type fracture indicates strong bonding between the materials. Especially the low-viscosity bulk-fill composite materials such as SDR that aim at preventing the initial polymerization shrinkage stress through polymerization modulators enable producing a high dentin bonding strength and a strong bonding with the adhesive layer. In this study, the samples were polymerized at 1000 mW/cm^2 light density and 20 sec polymerization time. The Optibond FL 3-stage etch-and-rinse system was used as adhesive. Lower values were obtained for the dentin bonding strengths of the 4-mm bulk-fill composite samples that were polymerized at 1000 mW/cm^2 for 20 sec in the groups where shear bond strength tests were applied in this thesis study. The fracture types constituted mostly the mixed and cohesive fractures. These results can be explained by the fact that the dentin bonding agents we used in this study were moderately strong 7th generation self-etch systems and bonding strengths were reduced through thermal aging. No statistically significant differences were seen in similar groups of the study with respect to intergroup dentin bonding strength ($p = 0.005$).

The elasticity module of the dentin and the enamel-dentin connection site being lower than that of the adhesive may lead to unequal stress distribution at the connection interface and thus increased cohesive and mixed type fractures at the dentin and the enamel-dentin connection site (30). The fracture type analyses of the 85 samples used in this study showed that there were adhesive fractures in 15 of the samples, cohesive fractures in 27 and mixed fractures in 43. There is no distinct fracture type distribution among the low-viscosity and condensable composites from the bulk-fill materials used in the study group of shear bond strength samples. However, the entire control group samples had adhesive fractures. This can be explained by the fact that low-viscosity composite materials were not used in the control group. Energy 3-6 times denser than normal, that is between 1000 and 2800 mW/cm^2 , is used in a short time at

the high energy modes of LED polymerization devices. There are opinions that polymerization with high energy technique has not been adequately investigated yet and they involve three potential concerns:

- Speedy application of energy may produce weak resin restorations with short polymers
- Speedy application of energy has the potential to reduce the diametric tensile strength
- There may be a threshold value at which the resin becomes a good quality one and high energy may result in more fragile resins (31).

In this study, the test samples were polymerized at light densities as high as 1000 and 1400 mW/cm^2 . A comparison of the bonding values obtained as a result of the shear bond strength tests showed that there were no distinct statistical differences in the density and duration combinations of the bulk-fill composite materials within themselves. However, we obtained lower values compared to the bonding strength values in units of Mpa in the literature. The majority of fracture types being cohesive and mixed types can be considered to occur as a result of increased internal stress and fragile resin formation due to speedy polymerization. It is obvious that further studies need to be carried out on this subject.

Nicoleta et al. (32) explored the shear bond strengths of bulk-fill resin composites in deciduous and permanent teeth. They used a polymerization device with an output of 1100 mW/cm^2 in their study. The SDR and Tetric Evo Ceram Bulk Fill testing samples 3 mm in diameter and 4 mm in thickness were applied to milk teeth and permanent teeth substrates and were aged at 5000 cycles. As adhesives, they used Xeno V and Adhes OneF single-stage self-etch systems. The highest shear bond strength value combination was found in the group where Xeno V and SDR were applied to permanent teeth. The adhesive and mixed fracture types occurred at the same rate in the Xeno V groups. Similar to our study, they used a polywave light device and performed aging with thermal cycles. Their shear bond strength values were found compatible with those of the groups that were polymerized at 1000 mW/cm^2 for 20 sec in our study. Their fracture type distribution was also similar.

5. Conclusion

The bulk-fill composite materials used in the study were polymerized with light densities and light irradiation times above those in the producer company instructions. When compared to the literature, the increased light density and irradiation time did not make any significant contribution to dentin bonding strength. The occurrence of cohesive fractures increased as the polymerization time of the SDR[®] bulk-fill flowable composite material was prolonged.

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