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Dentin Bond Strength of Composites with Self-etching Adhesives Using LED Curing Lights

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Abstract

Aim: The purpose of this study was to investigate the effect of light-emitting diode (LED) light curing units (LCUs) compared with halogen LCUs on the shear bond strength (SBS) of one nanofill composite (Filtek Supreme) and one microhibrid composite (Artemis) with self-etch adhesives.

Methods and Materials: The buccal surfaces of 60 non-carious extracted human molars were flattened to expose dentin and, subsequently, polished for 60 seconds with 600-grit wet silicon carbide abrasive paper. Specimens were assigned into six groups (n=10) according to composite material, self-etch adhesive, and curing light used as follows: *Group 1:* Adper Prompt L-Pop (AP) and Filtek Supreme (FS) using an Elipar Free Light (EFL); *Group 2:* AP and FS using an Elipar Free Light 2 (EFL2); *Group 3:* AP and FS using a Hilux Expert (HE) light, *Group 4:* AdheSE (AS)+Artemis (AR) using an EFL; *Group 5:* AS+AR using an EFL2; and *Group 6:* AS+AR using a HE light. The specimens were thermocycled for 500 cycles (5ºC–55ºC) and then loaded to failure in a Zwick universal testing machine at a crosshead speed of 5 mm/minute. SBS values were calculated as megapascals (MPa) and statistically analyzed using the one-way analysis of variance (ANOVA) test at a significance level of 0.05.

Results: Mean SBS (± standard deviations) values were as follows: *Group1:* 15.99±5.18; *Group 2:* 18.76±6.71; *Group 3:* 17.70±5.04; *Group 4:* 16.93±3.99; *Group 5:* 18.01±5.19, and *Group 6:* 17.46±5.40. There were no statistically significant differences for SBS to dentin among the groups tested.

Conclusion: The LED curing lights used in the study seem to be comparable with the halogen curing light for nanofill and microhybrid composites used in conjunction with self-etching systems in dentin. The EFL2 reduces curing time, which can be considered as an advantage.

Keywords: LED curing lights, halogen curing lights, self-etching primers, composites, shear bond strength

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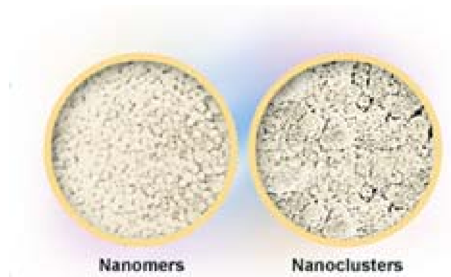
Introduction

Scientific interest in polymerization issues has increased in the wake of the increased use of improved composite resin formulations in dentistry.[1] The first light cured resin composites were polymerized by UV light and later by blue light commonly found in halogen bulbs.[2,3]

The innovative light-emitting diode (LED) technology has been improved for light curing dental materials to overcome the production of excessive heat during curing cycles. LEDs also avoid the drawback of bulb/reflector/filter degradation associated with halogen light curing units (LCUs).[4] Such degradation occurs over time due to high operating temperatures.[4] LED LCUs are inexpensive and last for thousands of hours in contrast to the 30 to 50 hour lifespan of a conventional halogen bulb.[5] In addition these LCUs are very compact and can be designed to emit specific light waves. They have better resistance to shock and vibration and are portable and safe to use.[4,6] Although they have a low level of light emission, LEDs are capable of polymerizing composite resin in a manner qualitatively comparable (or slightly less) with other light sources.[6,7] Furthermore, the temperature increase is significantly lower than other LCUs, thus, reducing a potential hazard to the pulp tissue.[8,9] It should also be noted saving time during the light curing process is one of the important benefits for clinicians who prefer using the incremental filling technique.[10]

Another new approach in dental materials research is the evaluation of self-etching adhesives as an alternative to conventional total-etch systems. Self-etching adhesives require a less sensitive technique and are easier to apply than total-etch adhesives.[11] Two step self-etching primers eliminate the conditioning and rinsing steps, however, a separate bonding system is required to couple the primed tooth substrate to the resin composite.[12] The most recent innovation in dental adhesive technology involves the introduction of one step self-etch adhesives ("all-in-one adhesives") in which the conditioner, primer, and bonding resin are combined to facilitate a single step adhesive application.[13,14] All-in-one adhesives are the most user friendly adhesive systems currently available.[15]



Currently available composite resins referred to as "nanofilled composites" are produced with nanofiller technology and formulated with nanomer and nanocluster filler particles.[16] Nanomers are discrete nanoagglomerated particles of 20–75 nm in size, and nanoclusters are loosely bound agglomerates of nano-sized particles. The manufacturer suggests the combination of nanomer-sized particles and nanocluster formulations reduces the interstitial spacing of the filler particles providing increased filler loading, better physical properties, and improved retention of a polished surface.[16]

This study evaluated the shear bond strength (SBS) of one nanofill composite (Filtek Supreme, 3M ESPE, St. Paul, MN, USA) used with a one step, self-etch adhesive (Adper Prompt L-Pop, 3M ESPE, St. Paul, MN, USA) and one microhybrid composite (Artemis, Ivoclar Vivadent, Schaan, Liechtenstein) used with a two step, self-etch adhesive (AdheSE, Ivoclar Vivadent, Schaan, Liechtenstein) and cured using two different LED LCUs in comparison with a single halogen LCU.

Methods and Materials

Sixty, non-carious, extracted human third molars were stored in distilled water and used within one month after extraction. The criteria for tooth selection included the presence of intact buccal enamel with no cracks caused by the extraction forceps. The buccal surface of each tooth was ground to remove the enamel and then exposed dentin was polished for 60 seconds with 600-grit silicon carbide abrasives paper under tap water. The prepared teeth were embedded with self curing acrylic resin and randomly divided into six groups (n=10) according to composite material, self-etch adhesive, and curing light used.

The composition of adhesive systems, restorative materials, and LCUs used in this study are listed in Tables 1, 2, and 3, respectively, while Table 4 shows the make up of the experimental groups.

Table 1. Composition of adhesive systems used in this study.

Materials	Manufacturer	Chemical Composition	Batch Number	pH
Adper Prompt L-Pop (One step self-etch adhesive)	3M ESPE St. Paul, MN, USA	**Liquid 1 (red blister)** • Methacrylated phosphoric esters • Bis-GMA • Initiators based on camphorquinone • Stabilizers **Liquid 2 (yellow blister)** • Water • 2-Hydroxyethyl methacrylate (HEMA) • Polyalkenoic acid • Stabilizers	201130	1.5
AdheSE (Two step self-etch adhesive)	Ivoclar- Vivadent Schaan, Liechtenstein	**Primer:** • Dimethacrylate • Phosporic acid acrylate • Initiators • Stabilizers • Water **Bond:** • HEMA • Dimethacrylate • Silicon dioxide • Initiators	F22511	1.7

Sources:
3M ESPE (2002) Adper Prompt L-Pop Self Etch Adhesive Technical Product Profile St. Paul, MN, USA
Ivoclar/Vivadent (2002) Scientific documentation AdheSE Liechtenstein

Table 2. Composition of the restorative materials used in this study.

Material	Matrix	Manufacturer	Batch Number	Filler Weight	Average Filler Size (μm)
Artemis A2 Dentin Mycrohybrid composite	Bis-GMA, Urethane Dimethacrylate, Triethyleneglycol, Dimetacrylate, Barium Glass, ytterbium trifloride, Ba-Al Fluorsilicate glass	Ivoclar Vivadent AG, Schaan, Liechtenstein	F29912	75-77%	0.6 μm
Filtek Supreme A2 Dentin Shade Nanofilled composite	Triethyleneglycol, dimethacrylate, urethane, Dimethacrylate Bis-EMA	3M ESPE Dental Products, St. Paul, MN, USA	3910A2D	78.5%	20nm (silica) 5-20nm (zirconia/ silica)

Table 3. Light curing units (LCU) used in this study.

LCU	Type	Manufacturer	Intensity (approx.)
Elipar Free Light (EFL)	LED	3M ESPE Dental Products, St. Paul, MN, USA	$400mW/cm^2$
Elipar Free Light 2 (EFL2)	LED	3M ESPE Dental Products, St. Paul, MN, USA	$1200mW/cm^2$
Hilux Expert (HE)	Halogen	Benliog˘lu Dental, Ankara, Turkey	$500mW/cm^2$

Table 4. Experimental groups.

Group	Adhesive	Composite	LCU	N
1	Adper Prompt L-Pop	Filtek Supreme	Elipar Free Light	10
2	Adper Prompt L-Pop	Filtek Supreme	Elipar Free Light 2	10
3	Adper Prompt L-Pop	Filtek Supreme	Hilux Expert	10
4	AdheSE	Artemis	Elipar Free Light	10
5	AdheSE	Artemis	Elipar Free Light 2	10
6	AdheSE	Artemis	Hilux Expert	10

Preparation of Groups 1, 2, and 3

The Adper Prompt L-Pop (AP) one step self-etch adhesive was used in all three of these groups and applied with active bluster and rubbed for 15 seconds than gently air blown dry and light cured as follows:

- *Group 1:* Exposure of 10 seconds using the Elipar Free Light (EFL).
- *Group 2:* Exposure of 5 seconds using the Elipar Free Light 2 (EFL2).
- *Group 3:* Exposure of 10 seconds using the Hilux Expert (HE).

Filtek Supreme (FS) composite (dentin shade A2) was then applied using a special mold whose diameter was 1.7 mm and cured as follows:

- *Group 1:* Exposure of 40 seconds using the EFL.
- *Group 2:* Exposure of 20 seconds using the EFL2.

- *Group 3:* Exposure of 40 seconds using the HE.

Preparation of Groups 4, 5, and 6

The AdheSE (AS) two step self-etch adhesive was used in Groups 4, 5, and 6. The primer was applied for 30 seconds and gently air dried then the bonding agent was applied and light cured as follows:

- *Group 4:* Exposure of 10 seconds using the EFL.
- *Group 5:* Exposure of 5 seconds using the EFL2.
- *Group 6:* Exposure of 10 seconds using the HE.

Artemis (AR) composite (dentin shade A2) was then applied using a special mold whose diameter was 1.7 mm and cured as follows:

- *Group 4:* Exposure of 40 seconds using the EFL.
- *Group 5:* Exposure of 20 seconds using the EFL2.
- *Group 6:* Exposure of 40 seconds using the HE.

All specimens were subjected to thermocycling for 500 cycles in two water baths held at 5°C and 55°C with a dwell time in each bath of 30 seconds with a transfer time of three seconds between baths.

A Zwick universal testing machine (Zwick GmbH & Co., KG, Ulm, Germany) was used for the shear bond test at a crosshead speed of 5 mm/min. Force was directly applied to the composite-dentin interface using a single-ended flattened steel rod. The load at failure was recorded by a personal computer connected to the Zwick test machine. The SBS values were calculated in megapasqals (MPa) by dividing the force by the area of the mold. After testing the SBS, the fracture sites were then examined with a Leica MS5 microscrope (Leica, Wetzlar, Germany) under 16X maginification to determine if the mode of failure was either adhesive or cohesive.

The statistical analysis was performed using the two-way anaylsis of variance (ANOVA) test at a significance level of 0.05.

Results

The mean SBS values and standard deviations in MPa are shown in Table 5. Mean SBS (± standard deviations) values were as follows:

- *Group 1:* 15.99±5.18
- *Group 2:* 18.76±6.71
- *Group 3:* 17.70±5.04
- *Group 4:* 16.93±3.99
- *Group 5:* 18.01± 5.19
- *Group 6:* 17.46±5.40

The two-way ANOVA indicated there were no statistically significant differences for bond strength to dentin between the LCUs used (p=.511), composites (p=.968), and interaction

Table 5. Shear bond strength (MPa) for the different groups.

Group	n	Mean	SD
Adper Prompt L- Pop, Filtek Supreme, (Elipar Free Light)	10	15.99	5.18
Adper Prompt L- Pop, Filtek Supreme, (Elipar Free Light 2)	10	18.76	6.71
Adper Prompt L- Pop, Filtek Supreme, (Hilux Expert)	10	17.70	5.04
AdheSe, Artemis, (Elipar Free Light)	10	16.93	3.99
AdheSe, Artemis, (Elipar Free Light 2)	10	18.01	5.19
AdheSe, Artemis, (Hilux Expert)	10	17.46	5.40

Table 6. Distribution of modes of failure.

Group	n	Adhesive %	Cohesive %
Adper Prompt L- Pop, Filtek Supreme, (Elipar Free Light)	10	50	50
Adper Prompt L- Pop, Filtek Supreme, (Elipar Free Light 2)	10	40	60
Adper Prompt L- Pop, Filtek Supreme, (Hilux Expert)	10	30	70
AdheSe, Artemis, (Elipar Free Light)	10	40	60
AdheSe, Artemis, (Elipar Free Light 2)	10	50	50
AdheSe, Artemis, (Hilux Expert)	10	40	60

between LCUs and composites (p=.891) (p>0.05). For both composite materials used, the least shear bond mean values were obtained with EFL LCU, and the highest shear bond mean values were observed using the EFL2 LCU but there was no statistically significant difference (p>0.05).

The mode of failure is shown in Table 6. The highest percentage of cohesive failure was observed with FS composite using the HE halogen curing light.

Discussion

Control of contour during restoration placement, improved color stability, and improved polymerization compared to chemically activated materials are some of the advantages of light cured resin composites. The quality of polymerization is one of the important factors affecting the longevity of any composite resin restoration. Using LED LCUs to achieve maximum polymerization of composite resins is important from a practical and fundamental view point since these LCUs are gaining popularity.[17] However, conflicting results are often reported in the literature when the effects of different LCUs on composite restorative materials are reported. This might be explained by the differences between irradiation protocols used, especially the intensity of the light used.[18] For this reason, a standard halogen LCU (HE) was used as a control. In the present study the SBS of a nanofill and a microhybrid composite cured with LED LCUs (EFL and EFL2) were not statistically significantly different than the values produced with a halogen LCU (p>0.05).

Successful adhesion to dentin, a more simplified restorative technique using fewer clinical steps, and lower technique sensitivity are some of the important requirements to consider when selecting a resin based composite as a restorative material. Self-etching primers which simplify and shorten bonding procedures are available.[19,20] Studies have been conducted on the bond strengths of composites to dentin using self-etch adhesives.[21,22] A few of the studies have shown self-etch adhesives bond sufficiently to dentin where as others demonstrated bonding was inadequate.[14,23,24]

In the present study an “all-in-one” self-etch adhesive (AP) and a two step self etch adhesive (AS) were used with two different LED LCUs compared with a halogen LCU. No significant difference was found between the groups.

The AP adhesive evolved from the original AP adhesive and is composed of methacrylated phosphoric esters, Bis-GMA, initiators, stabilizers, water, HEMA, and polyalkenoic acid. Naughton et al.[25] found similar mean SBS values (15.3 MPa) with AP as was found in the present study.

AS is a relatively new self-etching system containing a primer consisting of phosphoric acid acrylate, bis-acrylate, bis acrylamide, water, initiators and stabilizers along with a bonding component consisting of dimethacrylate, hydroxyethyl methacrylate, highly dispersed silicon dioxide, initiators, and stabilizers. AP and AS adhesive systems achieved similar bond strengths with the three LCUs tested in the present study.

The shear bond test is a simple method used for the laboratory evaluation of adhesive systems.[26] Other bond strength tests including tensile and fracture toughness tests have also been suggested.[27-29] However, whether any test resulting in the fracture and removal of dentin is truly measuring the strength of the dentin substrate is moot.[30] In order to predict the performance of adhesive systems bonding tests are necessary and useful. These test results may correlate with clinical conditions, but clinical success cannot be obtained by relying on *in vitro* investigations alone.[31] It should be noted in most studies dentin surfaces are standardized by grinding them with 600 grid silicon carbide abrasives.[25,32]

It has been hypothesized a minimum bond strength to dentin of 17 to 20 MPa is required to withstand contraction forces of resin composite materials.[33] The lowest SBS values (15.99 MPa) were obtained by Group 1 (AP and FS with using an EFL curing light). The highest SBS values (18.76 MPa) were obtained by Group 2 (AP and FS with using an EFL2 curing light).

The cohesive failure in resin composite indicates adhesion forces between the dentin and the self-etching are stronger than the cohesive forces of the composites themselves. The highest percentage cohesive failure mode was observed with FS using the halogen LCU (HE).

Conclusion

Under the conditions of this *in vitro* study, LED curing lights used in the study seem to be comparable with the halogen curing light for nanofill and microhybrid composites used in conjunction with self-etching systems in dentin. The EFL2 curing light reduces curing time, which can be considered as an advantage. However, in order to confirm these *in vitro* results, clinical evaluations are obviously necessary.

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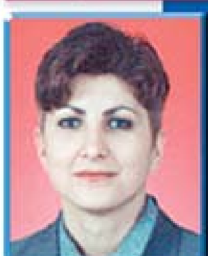

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