Effects of Curing Modes of Light Emitting Diode on Shear Bond Strength of Resin Composite

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#### **ABSTRACT**

Aim: To evaluated the SBS of three resin composites (Tetric Flow, Tetric and Heliomolar) bonded to dentin and cured by different curing modes utilizing light emitting diode (LED) light curing unit. **Materials and Method:** Buccal dentin of 90 upper premolars was expose and Clearfil SE Bond applied and cured prior to restorative procedure. Samples divided into 9 groups (3 groups for each composite type), photoactivation of groups of each composite was carried out using three modes: Continuous (CO: 600 mW/cm² for 40 s), Soft-Start (SS:  $100 \text{ mW/cm}^2$  for  $10 \text{ s} + 600 \text{ mW/cm}^2$  for 3 s and Pulse-Delay (PD:  $100 \text{ mW/cm}^2$  for  $3 \text{ s} + 3 \text{ min wait} + 600 \text{ mW/cm}^2$  for 3 s). Samples thermocycled and loaded at tooth-composite interface at 1 mm/min cross head speed until failure. **Results:** Two-way ANOVA ( $p \le 0.05$ ) followed by Duncan multiple range test revealed that SBS of SS mode (21.5 Kg) is significantly higher than SBS of both CO (18.1 Kg) and PD (18.7 Kg) and its also revealed that although SBS of Tetric Flow (21.2 Kg) is higher than that of Tetric (20.1 Kg) and both are significantly higher than that of Heliomolar (16.9 Kg). **Conclusions:** Less material's rigidity along with slower polymerization reaction by SS mode result in higher SBS due to lesser contraction stress at the adhesive interface.

Key Words: Light emitting diode, curing modes, shear bond strength.

Mohammad KA, Hamdoon MGh, Rasheed AM. Effects of Curing Modes of Light Emitting Diode on Shear Bond Strength of Resin Composite.. *Al–Rafidain Dent J.* 2008; 8(2):168–176.

Received: 18/10/2006 Sent to Referees: 18/10/2006 Accepted for Publication: 7/12/2006

### INTRODUCTION

Composite materials are well suited for repair of damaged or decayed tooth structure because in addition to an esthetic appearance, they can easily be adapted to a wide variety of direct placement applications and be bonded to the tooth. Liquid resins can be converted to solid, by simple exposure to visible light activation. The polymerization process involving conventional dimethacrylate monomers used in dental composites is quite complex and the final polymers are with intrinsic problems that mainly result from polymerization shrinkage. (1, 2)

The polymerization shrinkage of a resin composite can create stresses that compete with the adhesive—dentin bond, potentially causing failure of the bond and eventually, the restoration itself.<sup>(3, 4)</sup> Oth-

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erwise, if the bonding interface remains intact, residual forces create stresses to surrounding tooth structure, and may result in apparent tooth strain or fracture. (5)

It has been proved that degree of conversion (DC) of resin composite is directly proportional with curing light intensity. So, increasing light intensity lead to superior physical and mechanical properties. But unfortunately there is a linear relationship between polymerization shrinkage strain and DC. (6) Therefore, the maintenance of the bond integrity and sufficient shear bond strength (SBS) of the restoration without the loss of the ultimate physical and mechanical properties can be obtained through the relaxation of the stress through the composite flow. (7, 8)

Studies have demonstrated that prepolymerization with low light intensity, followed by final polymerization at high intensity (soft-start or referred to as stepcuring) could reduce the stress generated by shrinkage. (9) This photoactivation mode results in slower contraction and more flow time to compensate for shrinkage strain while maintaining the degree of cure. (8,10-11) Through the soft-start mode, obtained a better marginal adaptation than with the conventional technique, without the loss of the physical and mechanical properties of the composites. (6, 9, 10, 13, 14) To allow more time for composite flow, the mode of pulse-delay was proposed, where the polymerization is initiated by a short flash of light (3 seconds) followed by a waiting time of several minutes (3 minutes) before the final cure is performed allowed for stress relaxation by flow, before curing is completed with a second irradiation at high intensity. (15-17)

Regarding material properties, a resin composite that has the ability to flow during the early phases of polymerization would allow shrinkage to occur without substantial stress. As many resin composites used for posterior restorations are not able to flow easily, the resin-dentin interface is subject to high stress. (18) Less rigid materials were observed to be better capable of reducing the contraction stresses than rigid materials. (18, 19) Application of a flowable composite prior to placement of the resin composite might function as an elastic liner preserving good bonding as well as preventing gap formation at the internal margin. (20)

Due to apparent drawbacks of tungsten-halogen light curing units, lightemitting diode (LED) has been developed and operating at 470 nm wavelength as an alternative to standard tungsten-halogen units. (21)

This study evaluated the SBS of three resin composite restorative materials bonded to dentin and cured by different curing modes utilizing LED light curing unit

# MATERIALS AND METHODS

Ninety upper premolars free of cracks and caries, extracted for orthodontic reasons were collected, cleaned, polished and stored in distilled water, used in this study. Roots of teeth were cut and discarded, then each crown was embedded in self-cure acrylic resin (Major Repair 2, 10024 Moncalieri, Italy) within cylindrical plastic tube (2×2 cm) in such away that palatal and middle third are embedded within acrylic leaving the buccal third extruded out of acrylic and above the level of plastic ring rim to enable subsequent grinding, restorative materials application and testing. After that, Buccal surface of each tooth was grinded to remove buccal enamel and exposing dentin at least 4 mm diameter at middle third of tooth using 600 grit abrasive carbide papers mounted on grinding wheel of rotary pregrinder machine (Metasery, Surry, England) with copious amount of water to create standardized dentin surface. (22)

To conduct this study, three resin composite restorative materials: Tetric Flow (Vivadent), Tetric (Vivadent) and Heliomolar (Vivadent), with same shade (A2) were selected and self-etching primer adhesive system (Clearfil SE Bond, Kuraray). Materials, manufacturers, composition and batch numbers are listed in Table (1).

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Materials	Manufacturer	Composition	Filler	Batch
Tetric Flow	Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, TEG- DMA (35%wt)	64 wt % 0.04-3 μm (mean 0.70 μm)	C31580
Tetric	Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, TEG- DMA (18.8% wt)	81% wt (mean 0.70 μm)	C24789
Heliomolar	Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, decandiol dimethacrylate (22% wt)	77.8% wt 0.04-0.2 μm	C35631
Clearfil (SE Bond)	Umeda, Kita-Ku. Kuraray, Osaka, Japan	Primer: MPD, HEMA, DMA, DPT, water Bonding: MDP, Bis-GMA, HEMA, DMA, CQ, DPT, silanated colloidal silica		Lot 125

**Bis-GMA**: bisphenol A glycidyl methacrylate; **UDMA**: Urethane dimethacrylate; **TEGDMA**: triethylene glycol dimethacrylate; **HEMA**: Hydroxyethyl methacrylate; **DMA**: hydrophilic dimethacrylate; **DPT**: N,N-diethanol p-toluidine; **MPD**:10-Methacryloyloxydecyl dihydrogen phophate; **QC:** Camphoroquinone

A commercial light-cure unit that allowed independent command over time and intensity (VIP, BISCO, Inc., Schaumburg, IL) was selected for this study.

For composite photoactivation, three irradiation modes were utilized. The continuous or conventional (CO) involves irradiation at 600 mW/cm<sup>2</sup> for 40 seconds. The soft-start (SS) initially uses low intensity (100 mW/ cm<sup>2</sup> for 10 seconds) followed by a final cure at high intensity (600 mW/cm<sup>2</sup> for 30 seconds). The pulse-delay (PD) mode employs an initial low intensity exposure (100 mW/cm<sup>2</sup> for 3 seconds) followed by 3 min waiting time and a final cure at high intensity (600 mW/cm<sup>2</sup> for 37 seconds), (23) Light intensity was verified using a radiometer (CROMATEST 7041, Curing Radiometer, Germany). The light exposure period was kept constant (40 seconds) for all photoactivation modes.

Samples were randomly assigned to nine experimental groups of 10 teeth each as follows:

Group 1: Continuous irradiation mode, Tetric Flow resin composite (CO-TF).

Group 2: Continuous irradiation mode, Tetric resin composite (CO-T).

Group 3: Continuous irradiation mode, Heliomolar resin composite (CO-H).

Group 4: Soft-Start irradiation mode, Tetric Flow resin composite (SS-TF).

Group 5: Soft-Start irradiation mode, Tetric resin composite (SS-T).

Group 6: Soft-Start irradiation mode, Heliomolar resin composite, (SS-H).

Group 7: Pulse-Delay irradiation mode, Tetric Flow resin composite (PD-TF).

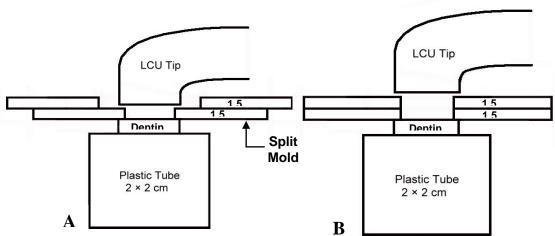
Group 8: Pulse-Delay irradiation mode, Tetric resin composite (PD-T).

Group 9: Pulse-Delay irradiation mode, Heliomolar resin composite (PD-H).

Before application of restorative materials, bonding agent applied according to manufacturer instructions and cured (20 seconds, Continuous mode) with the tip of curing unit being perpendicular on the buccal surface with distance of 1 mm that controlled by using Teflon spacer which is a Teflon plate of 2×4 cm and 1 mm thickness with a 5 mm diameter hole at the center of the plate in order not to interfere with the light beam irradiating the 4 mm diameter of exposed dentin, on the other hand this 5 mm hole is smaller than the tip of curing units, so keeping the tip constantly 1 mm distant from dentin.

Then, each restorative material was applied by the aid of a Teflon split mold with cylindrical cavity of 4 mm in diameter and 3 mm thickness, which perfectly adjusted on the exposed dentin. Composite applied by two increments of 1.5 mm thickness for each, this procedure was

guided by the thickness of split mold which is already composed of two layers of 1.5 mm for each layer as shown in Figure (1).



**Figure (1): A:** Split mold at first composite increment application. **B:** Split mold at second composite increment application.

Approximation of the two parts of first layer of the split mold over the exposed dentin will provide a cylindrical space of 4 mm diameter and 1.5mm depth (Figure 1A), first composite increment applied and cured by a specific irradiation mode. Then two parts of second layer of split mold approximated to each other creating a cylindrical space of 4mm diameter and 1.5 mm depth continuous with the first space (Figure 1B), second composite increment applied over the first one and cured by the same irradiation mode utilized for the first increment. Then samples thermocycled in a water bath for 300 times

at a temperature  $(5\pm2-55\pm2\,^{\circ}\text{C})$  with a dwell time of 30 seconds for each cycle, (24) then stored in tap water for 24 hours. After that the bond strength between tooth and composite was measured by using Universal Testing Machine (Soil Test Co. Inc., USA). The samples grasped by a holder that hold the plastic ring horizontally, so making the tooth-restoration interface vertical to the floor, then the interface between dentin surface and the core of composite subjected to a load with stainless steel knife edge head at a cross head speed of 1 mm/minute till failure occurred (Figure 2). (22)

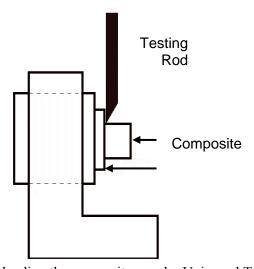


Figure (2): Testing rod loading the composite core by Universal Testing Machine.

The loads required to produce failure were recorded, calculated and statistically analyzed by SPSS for windows program (version 11.0.0, LEAD Technologies Inc.).

### **RESULTS**

Mean and standard deviation of SBS for the tested groups were as listed in Table (2) and represented by a histogram Figure (3)

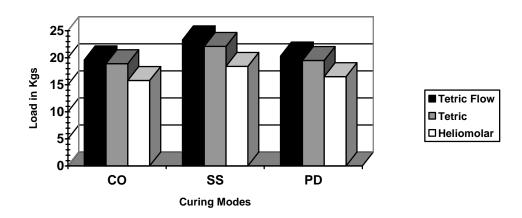


Figure (3): A histogram shows the means for the tested groups.

Tow-way analyses of variance (ANOVA) was performed to show if there are significant differences among groups and their interactions, and its revealed that there are significant differences at a level

of curing modes and composite types but there are no significant differences at the level of mode-composite interaction, at probability value of (p < 0.05) as shown in Table (3).

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	SS	df	MS	F-value	Sig.
Corrected Model	463.406	8	57.926	4.782	.000*
Intercept	33767.721	1	33767.721	2787.640	*000
MODE	164.189	2	82.094	6.777	.002*
COMPOSITE	295.699	2	147.849	12.205	.000*
MODE * COMPOSITE	3.519	4	.880	.073	.990
Error	981.183	81	12.113		
Total	35212.310	90			
Corrected Total	1444.589	89			

<sup>\*:</sup> Significant. SS: sum of squares. df= degree of freedom. MS= mean square.

Duncan multiple range test for curing modes revealed that SBS of SS mode (21.5 Kg) is significantly higher than SBS

of both CO (18.1 Kg) and PD (18.7 Kg) as shown in Table (4).

Table (4): Duncan Multiple Range Test for the Curing Modes.

MODE	N	Mean (Kg)	Duncan Grouping*
Continuous	30	18.1	В
Soft-Start	30	21.5	$\mathbf{A}$
<b>Pulse-Delay</b>	30	18.7	В

<sup>\*</sup> No significant differences in SBS among groups with the same letter; The error term is Mean Square (Error) = 12.113; Alpha = .05

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Duncan multiple range test for composite types revealed that although SBS of Tetric Flow (21.2 Kg) is higher than that of Tetric (20.1 Kg) but no statistical sig-

nificant difference present between them and both are significantly higher than that of Heliomolar (16.9 Kg) as shown in Table (5).

Table (5): Duncan Multiple Range Test for the Tested Resin Composites.

COMPOSITE	N	Mean (Kg)	Duncan Grouping*
Tetric Flow	30	21.2	A
Tetric	30	20.1	$\mathbf{A}$
Heliomolar	30	16.9	В

\* No significant differences in SBS among groups with the same letter; The error term is Mean Square (Error) = 12.113; Alpha = .05

## **DISCUSSION**

In an attempt to standardize the factors that may have effects on this experiment, A2 shade was selected to minimize the effects of colorant and light polymerization. (25) Light intensity in this study was above the minimum intensity of 400 mW/cm2 that has been suggested for routine polymerization. (26) Manufacturer's recommended curing time (40 seconds) followed, distance of light tip was constant. Clearfil SE Bond was chosen as an adhesive, because it ensures not only good adhesion to enamel, but also to superficial and deep dentin.(27-29) Thus, the influence of the polymerization stress and not the adhesive quality will play the primary role with regard to bond strength. (30)

However, in spite of surface treatments that provide improved adhesion of composites to dentin, as well as multi-step composite layering techniques, a reliable adhesion without marginal gap formation has proven elusive due to development of polymerization shrinkage stress and strain that may compromise the competitive formation of an adequate adhesive bond between the composite and the tooth. (31, 32)

During the initial phase of polymerization, when the early polymer is still in a flexible and fluid state, composite flow can be used to prevent stress development at the tooth/resin interface. (33, 34) The point at which the polymer acquires a higher modulus and can transfer polymerization stress to various interfaces is called the gel point. Many dental materials studies have rightly pointed out that shrinkage strain that occurs prior to gelation does not contribute to stress since this involves viscous but unrestricted flow. (23, 35) Above this

point, further polymerization results in the transmission of stress to the composite boundaries (the bonding interface and the tooth tissue itself). (33, 34, 36)

Polymerization shrinkage during photoactivation of a composite by CO is not uniform. It occurs very rapidly during the first 20 seconds and it slows down in the final 20 seconds. (17) Reduction of this initial speed can be obtained through the use of low intensity units that produce few free radicals. (37) This lower speed gives the composite more time for molecular rearrangement, reducing the stress caused by polymerization shrinkage. (38) So, the SS reduced the initial shrinkage, enabling the material to flow in this period. This resulted in a reduction of stress at the bond interface, demonstrated through the higher SBS values obtained by this technique. These findings are in agreement with other studies that had obtained higher microtensile bond strength, (23) less post-gel shrinkage, (39) and better sealing of the restoration margins and thus, less marginal microleakage through composite photoactivation using the SS mode. (20, 40)

In PD, one seeks to reduce the polymerization reaction speed and, therefore, to relieve part of the stress generated by polymerization shrinkage. In this study, however, this effect was not obtained and the bond values for this technique did not differ from the ones achieved with CO, and this is supported by findings of Silva and others. (23) Yap *et al.* (17) through the PD in which the initial activation was carried out for 3 seconds at an intensity of 100 mW/cm2, like in the one used in this study, did not detect any shrinkage of the composite during photoactivation and the

waiting period. The authors believe that the energy density of the initial photoactivation (the intensity multiplied by the exposure period) was not enough to effectively initiate a polymerization reaction and all the shrinkage the composite underwent, was compensated for by the flow of the material. The reaction only became effective when the second high intensity exposure occurred.

Modifications to stress and strain development can also be made based on the fillers used in a dental composite. It is well known that maximized filler loading is one avenue to reduced shrinkage by moderating the heat rise during cure. But surprisingly, there has been little systematic work conducted on the direct effect of filler volume on shrinkage stress. (42)

However, Chen et al.(43) found that hybrid composite can be better in reducing the contraction stresses during the early setting stage than the highly filled small sized resin composites, as the highly filled small-sized interlocking filler particles may, to some extent, obstruct the composite to change shape during polymerization, resulting in an overall higher stress build-up. These findings agree with the current study as highly filled Heliomolar (microfilled composite) showed significantly less SBS values compared with Tetric (hybrid composite).

On the other hand, the resin matrix also has an important influence on the properties of the composite materials, besides the filler system. (43) In most dental resins, a low viscosity diluent monomer, commonly triethylene glycol dimethacrylate (TEGDMA), is added to a bulkier and structurally rigid base monomer, such as bisphenol A glycidyl methacrylate (Bis-GMA) or urethane dimethacrylate (UD-MA). The higher molecular weights or more precisely, the lower reactive group concentrations, associated with these base monomers contribute to reduced polymerization shrinkage compared with the smaller diluent comonomer.(44) The overall shrinkage that occurs during polymerization is proportional to the degree of conversion. (45) Thus, it is not particularly meaningful to report shrinkage values of different materials with relatively the same base monomers, or even values for the

same material polymerized under different curing modes, in the absence of a coordinated measurement of conversion. (2)

Less rigid materials and slower polymerization reaction may provide better ability to undergo plastic flow during the early phases of polymerization, which causes reduction in polymerization contraction stress and less damage at the adhesive interface. (9, 10, 19, 31, 46-48) Leevailoj et al. (49) reported a decrease in marginal microleakage when flowable composites were used. Current study agreed with these facts as the SBS of Tetric Flow (flowable composite) was higher than that of Tetric (hybrid composite) and significantly higher than highly filled high viscosity Heliomolar composite, as increasing the resin content or decreasing filler load produces a composite that is easily adapted to preparation walls. (50) This may advocate the use of less rigid materials to enhance SBS of resin composite filled restorations.

## **CONCLUSIONS**

Within the limitations of this in vitro study, less material's rigidity along with slower polymerization reaction by SS mode result in higher SBS of resin composite, due to that it may provide better ability to undergo plastic flow during the early phases of polymerization resulting in lesser contraction stress at the adhesive interface.

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