

**The effect of curing light source on shear bond strength and degree of conversion over time of an orthodontic adhesive**

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**A thesis presented by Barton L. Carter D.M.D.**

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## ABSTRACT

*Objective:* The purpose of this study is to evaluate over time the shear bond strength (SBS) and degree of conversion (DC%) of an orthodontic resin-composite adhesive activated with a quartz-tungsten halogen and a high-intensity (second-generation) light-emitting diode (LED) light-curing unit (LCU) in order to determine the effect of light-source type on the SBS and DC% of the adhesive at various time points after light exposure and to determine whether there is a correlation between SBS and DC%.

*Methods:* Brackets were bonded to permanent mandibular bovine incisors (N=138) with an orthodontic adhesive (Transbond XT, 3M Unitek, Monrovia, CA) and cured with either a second-generation LED light source (Ortholux LED, 3M Unitek) for 10 seconds or a quartz-tungsten halogen (Ortholux XT, 3M Unitek) for 20 seconds per manufacturer's instructions. SBS testing was done on a universal mechanical test instrument (Q Test, MTS Sintech, Research Triangle Park, NC) at 5 minutes, 60 minutes, and 24 hours after initial exposure. Immediately after bond failure, composite specimens were removed from the adhesive remaining on each tooth and were analyzed by Fourier Transform Infrared (FTIR) spectroscopy (DS-20/XAD, Analect Instruments, Irvine, CA) to determine the degree of conversion of carbon-carbon double bonds within the polymerized composite adhesive. Data were analyzed with two-way ANOVA and Bonferroni and Tukey post-hoc testing ( $p < 0.05$ ).

*Results:* There was no significant effect of light source type on the SBS or DC% at each time interval after photopolymerization ( $p \geq 0.05$ ). Time, however, was found to have a significant effect on the SBS of orthodontic brackets – SBS increased significantly

with time up to 24 hours ( $p < 0.0001$ ). DC%, however, did not change significantly over time ( $p \geq 0.05$ ).

*Significance:* In addition to its practical and operational advantages the Ortholux LED LCU offered similar bond strength in half the cure time than the Ortholux XT quartz-tungsten halogen. Though it is not fully understood why SBS increases with time up to 24 hours while DC% remains constant, crosslinking and polymer entanglement may be factors. Within the limitations of this study, the use of second-generation LED technology is well justified.

## **PREFACE**

In an attempt to present all information related to this thesis and to provide a more concise report suitable for publication, a standalone manuscript intended for submission is included within this thesis. A comprehensive literature review precedes the manuscript which contains the discussion and conclusion sections. A complete list of references concludes the thesis. As the manuscript summarizes some of the material found in the remainder of the thesis, some redundancy is introduced.

## LITERATURE REVIEW

In 1964, Newman (1964) introduced the bonding of orthodontic attachments directly to enamel. Since then, numerous innovations and improvements in materials and procedures have made the process of bonding attachments directly to teeth a universally accepted technique among orthodontists.

Compared to metal bands that completely encircle teeth, bonded brackets have the advantage of being more esthetic, causing less soft tissue irritation and hyperplastic gingivitis, not occupying space in the perimeter of the arches, requiring no dental separation, producing no band space after removal, providing the ability to detect and treat dental caries during orthodontic treatment, and allowing attachment to partially erupted teeth (Bishara *et al.*, 2002). The polymerization of orthodontic resin adhesives with visible-light curing (VLC) provides many additional advantages to bonding brackets to teeth. Compared to chemically cured composite adhesives, VLC adhesive systems have no mix time (Neugebauer *et al.*, 2004), more consistent handling characteristics (Cacciafesta *et al.*, 2005), minimal oxygen inhibition (Lekka *et al.*, 1989), longer working-time with easier removal of excess composite, and a setting time that can be completely controlled (Eliades *et al.*, 1995). Also, it has been shown that light cured composite provides a higher initial degree of conversion and a higher bond strength of orthodontic brackets compared to self-cured adhesive (Eliades *et al.*, 1991; Eliades *et al.*, 1995; Chamda and Stein, 1996).



A highlighted advantage, “the greatest advantage” according to Bishara *et al.* (1999), of photopolymerization is the allowance of time to accurately position a bracket without the constraints normally imposed by chemically-initiated adhesive systems (Mavropoulos *et al.*, 2005). This extra working time that VLC provides comes at a cost to the clinician in the additional monetary expense of the VLC units and their maintenance and the additional time needed to expose the adhesive (Bishara *et al.*, 2003). Therefore, manufacturers have made efforts to provide light-curing units that emit light with qualities that reduce the time necessary to fully cure composite without compromising its mechanical properties.

The polymerization of a light-activated composite is a chemical process that combines monomers into a polymer network. First, a photon of light activates a light sensitive photoinitiator molecule, usually camphorquinone (CPQ), charging it with energy. The energy from the photon changes the CPQ molecule into a high energy radical which can react either with a monomer or a reducing agent such as amine co-initiators, molecules that can help to propagate the polymerization reaction more efficiently (Emami and Soderholm, 2005). When the radicals react with the resin monomers, the monomers in turn react to form polymers of resin (Mills *et al.*, 1999). The extent of this polymerization process is dependent upon the number of activated molecules of the photoinitiator which is, in turn, determined by the number and wavelength of the photons delivered to the sample (Althoff and Hartung, 2000).

The number of photons delivered to a sample of composite depends upon the total light energy density delivered – a function of light source and irradiation time. Total light energy density is the product of the irradiance (or power density, units: mW/cm<sup>2</sup>) times the exposure time (Total light energy density = irradiance \* time). The wavelength of the photons is also determined by the light source and when the wavelength more closely matches the photoinitiator's spectral absorption, the range of energy within the light spectrum necessary for activation of the photoinitiator, initiation of polymerization is more efficient (Althoff and Hartung, 2000).

CPQ, the most commonly used photoinitiator of dental VLC systems (Wiggins *et al.*, 2004), is , therefore, the key player in the chemistry of photopolymerization. The molecule, a diketone, absorbs light in the blue region of the visible light spectrum (Blankenau *et al.*, 1991). Its absorption maximum, the wavelength of light that is absorbed more than any other, has been measured to be between 410 and 500nm (Cook, 1982) with a peak at 467nm (Nomoto, 1997; Teshima *et al.*, 2003). Activation of CPQ in dental composites is more likely when the photons have wavelengths that match the absorption maximum of CPQ (Mills *et al.*, 1999).

## LIGHT-CURING UNITS (LCUs)

Photopolymerization of dental composites started in the 1970s beginning with its use on pit and fissure sealants (Mills *et al.*, 1999). Thereafter, VLC technology progressed into the realm of restorative dental materials and orthodontic adhesives (Sfondrini *et*

*al.*, 2001). Exposure of ultra-violet light with relatively long cure times was the first method of photopolymerization (Cacciafesta *et al.*, 2000). In the 1980s, visible light-curing was introduced in order to avoid some of the potentially harmful effects of long-term exposure to UV light (Cacciafesta *et al.*, 2000). Thereafter, VLC was found to be more efficient at curing composite compared to UV systems (Sfondrini *et al.*, 2001). Unlike today's VLC units which emit blue light, original units emitted white light from the entire spectrum of visible and non-visible light. Filters were added to these LCUs in order to block out unnecessary light outside the blue region and to further increase safety for the patient and the clinician (Cook, 1982).

## HALOGEN

The most commonly used LCU is the quartz-tungsten halogen (Mills *et al.*, 1999). These units produce light by incandescence in which a small tungsten filament is heated to several thousand degrees Celsius as an electrical current is passed through it (Althoff and Hartung, 2000). The resistance of the filament causes excitation of atoms over a wide range of energy levels emitting light of a broad spectrum most of which is infrared (Mills *et al.*, 1999; Dunn and Bush, 2002). One percent of the electrical energy used to power halogen LCUs is converted to the blue region of light necessary for photopolymerization (Althoff and Hartung, 2000). The mechanism of a halogen LCUs and their inherent inefficiency, where most of the energy is given off as heat, is at the root of many of their shortcomings.

The filament of a halogen LCU has a useful lifetime of 50-100 hours (Rueggeberg *et al.*, 1996), is sensitive to shock (Silta *et al.*, 2005), and requires a noise-producing internal fan to cool the unit. Associated with the fan are air slots that can make the handheld unit difficult to properly disinfect (Wiggins *et al.*, 2004). It has been well documented that the many sensitive components of a halogen LCU, including the bulb, filter, and reflector, degrade over time due to the extreme heat generated by the element. This degradation causes decreased output that can result in decreased physical and mechanical properties of the cured composite (Barghi *et al.*, 1994; Miyazaki *et al.*, 1998).

In testing halogen units used in private dental offices, investigators have found a wide range of intensities from 28 to 1368 mW/cm<sup>2</sup> in one study by Miyazaki *et al.* (1998) and from 11 to 680 mW/cm<sup>2</sup> by Barghi *et al.* (1994). The intensities of the lights were discovered to have dropped 15-82% compared to new units (Miyazaki *et al.*, 1998) and 30% emitted less than 200 mW/cm<sup>2</sup> (Barghi *et al.*, 1994). Both studies concluded that the practitioners were either unaware of the reduction in irradiance of the halogen LCUs or unsatisfied with its performance. In fact, a positive correlation was found between satisfaction level of the dentist and the output of the light. Furthermore, a negative correlation was found between the age of the light and its irradiance and its performance in compressive strength tests; old lights were more likely to have poor output and inadequately cure composite (Barghi *et al.*, 1994). The irradiance of halogen LCUs increased by 322% when the lamps, filters, and light guides were replaced. Of those three maintenance procedures, lamp replacement was the most effective way to

keep halogen LCUs working properly (Miyazaki *et al.*, 1998). Dentists are advised to check and replace faulty parts and regularly measure output in order to ensure proper performance. Decreased longevity and increased maintenance requirements are disadvantages of halogen LCUs that have definite clinical and practice management implications.

## LASERS AND PLASMA-ARC

With increasing light intensity of an LCU, the same total light energy density can be delivered to a composite in a shorter time. Intensity, therefore, is “the key to faster curing times” (Kauppi and Combe, 2003). In a quest to make light-curing more efficient, manufacturers have developed innovative products to reduce the number of seconds required to cure photosensitive composite. In the late 1980’s, the argon laser (Light Amplification by Stimulation Emission of Radiation) was introduced as a new type of LCU able to cure composite in 5 to 10 seconds. In the mid 1990’s the xenon-arc LCU was developed as another high-output alternative to quartz-tungsten halogen technology (Christensen, 2004).

Lasers emit a highly concentrated beam of light that is very narrow in spectrum that peaks at about 480 nm, close to the optimal energy for the activation of most composites (Althoff and Hartung, 2000; James *et al.*, 2003). Laser LCUs boast cure times of 5 to 10 seconds and have been shown to cure, in one quarter the time, composite with superior physical properties and degree of polymerization compared to

quartz-tungsten halogen (Blankenau *et al.*, 1991). Laser units, however, are bulky, the power conversion is worse than halogen (Althoff and Hartung, 2000), and the price of the units is very high (Wiggins *et al.*, 2004). Furthermore, the use of lasers requires strict safety precautions for the clinician patient (Mills *et al.*, 1999).

Xenon plasma-arc curing (PAC) units produce light that is much more intense than quartz-tungsten halogen (Oesterle *et al.*, 2001) and even though the white light produced by PAC units is filtered similar to halogen, the light spectrum is much more narrow (440-490nm) and more closely matches the maximum absorbance of CPQ (Neugebauer *et al.*, 2004). Cure times of 2 to 5 seconds recommended by manufacturers have been shown to produce results equivalent to quartz-tungsten halogen (Cacciafesta *et al.*, 2000; Sfondrini *et al.*, 2001). Furthermore, the short exposure time does not increase pulpal temperatures as much as halogen LCUs (Cacciafesta *et al.*, 2000). PAC units are able to cure orthodontic adhesives in such a short time because the composite layer under a bracket is so thin and because the high intensity light emitted by PAC units simultaneously activates a greater number of molecules compared to halogen LCUs (Cacciafesta *et al.*, 2000). Like lasers, PAC LCUs are relatively expensive (Christensen, 2004).

## THE LIGHT-EMITTING DIODE

In 1995, Mills (1995) proposed the use of solid-state light-emitting diode (LED) technology for the polymerization of dental composites in order to overcome many of

the inherent disadvantages of halogen LCUs. The relatively new blue LEDs showed considerable promise for use in the activation of light sensitive composites because of their long lifetime and consistent output (Mills *et al.*, 1999).

LED technology is based on semiconductors. The lights are manufactured with layered metal-organic chemical vapors such as gallium nitride, gallium arsenide, or gallium arsenide phosphide deposited onto different semiconductor materials (Silta *et al.*, 2005). The latest dental LED curing units have a coating of indium gallium nitride (Swanson *et al.*, 2004) and produce a blue light that has a narrow emission spectrum between 400 and 500nm with a peak at 460nm (Mills *et al.*, 1999). This narrow spectral band occurs as light is produced by the process of electroluminescence wherein electrons recombine with a hole within the p-n junction of n-doped and p-doped semiconductors. LED light consists of photons of a specific wavelength that depends on the band gap width of these p-n junctions (Nakamura *et al.*, 1994; Mills *et al.*, 1999). LED light production is extremely efficient as electricity is converted directly into light (Swanson *et al.*, 2004).

The emission spectrum of LED light makes it ideal for the polymerization of dental composites because the absorption maximum of CPQ is very near the peak of LED light output (Nomoto, 1997). This may also increase the curing efficiency of LED units (Jandt *et al.*, 2000), reduce the cure time for each bracket, and, thereby, greatly reduce the time required to bond several teeth compared to halogen LCUs.

LED LCUs, usually battery powered and cordless, have a lifetime of 10,000 hours and are, compared halogen LCUs, more resistant to vibration (Silta *et al.*, 2005). With their efficient energy conversion, LEDs produce little heat and, therefore, pose no threat to pulpal tissue (Yap and Soh, 2003). No filters or fans are needed with LED LCUs because the blue light does not include light within the infrared or ultraviolet ranges (Gronberg *et al.*, 2006).

## HALOGEN VS. LED

Price *et al.* (2003) stated: “Ideally, new types of dental curing lights should perform as well as or better than a quartz-tungsten halogen light”. Indeed, halogen has been the benchmark for dental LCU performance. Despite the advantages of LED over halogen technology, the first generation of commercially available LED curing lights, consisting of an array of many small, low-powered LED elements (Bishara *et al.*, 2003), produced considerably lower intensities than halogen LCUs that measure between less than 100 up to 400 mW/cm<sup>2</sup> (Mavropoulos *et al.*, 2005). As these first LEDs became available to the field of dentistry, researchers evaluated their performance with inconsistent results with in vitro evaluations of restorative dental composites.

In evaluation of the hardness and depth of cure of restorative composites cured with first-generation LEDs, some studies found the performance of halogen to be significantly superior (Kurachi *et al.*, 2001; Dunn and Bush, 2002; Mills *et al.*, 2002; Uhl *et al.*, 2002; Uhl *et al.*, 2003). Kurachi *et al.* (2001), for example, evaluated LED



LCUs with irradiance values no more than 79mW/cm<sup>2</sup> compared to a 475mW/cm<sup>2</sup> halogen LCU. Every sample cured with LED was of inferior hardness. Dunn and Bush (2002), in a comparison of LED LCUs with irradiance at 150mW/cm<sup>2</sup> and halogen LCUs at 900 and 1030mW/cm<sup>2</sup>, found halogen to provide superior Knoop hardness and depth of cure.

Uhl *et al.* (2003) evaluated a commercially available first-generation LED LCU (270mW/cm<sup>2</sup>) and a prototype LED light composed of 63 LED elements totaling 638mW/cm<sup>2</sup>. The halogen LCU produced a significantly increased depth of cure than the LEDs on 4 proprietary composites. Two of the composites tested contained only CPQ as the photoinitiator and were not significantly different in top-hardness when cured with the halogen LCU or 63-element LED. The other two composites contained a co-initiator sensitive to light outside the spectral output of LED LCUs. These two co-initiator-containing composites obtained significantly increased top-hardness when cured with the halogen light. Similar results were found by Uhl *et al.* (2004) and Price *et al.* (2003). These studies all concluded that the co-initiator was *the* factor accountable for the superior performance of the halogen LCU because the co-initiator absorbed light outside the LED emission spectra yet well within the range of halogen light. Therefore, it is advised that clinicians and researchers should be aware of the photoinitiator systems used in composites (Uhl *et al.*, 2003).

Uhl *et al.* (2002) found similar interactions of co-initiators and light type, but also found that first-generation LED LCUs were no faster at curing composite than halogen

LCUs. The prototype LED obtained, despite its lower irradiance, higher compressive strength than halogen. The authors concluded that the superior performance of the LED LCU when no co-initiators were present was due the ideal overlap of CPQ absorbance and the spectral output of blue LEDs.

Other early research found first-generation LEDs to perform not below but *at* the level of quartz-tungsten halogen LCUs (Mills *et al.*, 1999; Santos *et al.*, 2004; Tsai *et al.*, 2004). Santos *et al.* (2004) achieved similar hardness of composite cured with an LED (320mW/cm<sup>2</sup>) and a quartz-tungsten halogen (550mW/cm<sup>2</sup>). Tsai *et al.* (2004) achieved similar top-hardness of composite cured with first-generation LEDs and halogen LCUs. However the LED LCUs were outperformed beyond 1mm into the composite.

Mills *et al.* (1999), in an effort to compare the quality of LED and halogen light, adjusted the irradiance of a halogen LCU down to 455mW/cm<sup>2</sup> to be more similar to the 25 LED LCU at 290mW/cm<sup>2</sup>. The depth of cure for each composite evaluated was greater when cured with the LED even though its irradiance was 64% of the halogen. This phenomenon was due to the fact that the LED LCU's irradiance was actually higher in the 468nm region, the peak light absorption of CPQ. Interestingly, less than a decade later, studies that wish to compare the quality of LED light compared to halogen now must attenuate the emission of the LED LCU in order to achieve equalized irradiance. Mills *et al.* (2002) concluded, based on the performance of high-output prototype LED LCUs and unlike the first generation of commercially available LED LCUs, that light-emitting diode technology can perform at the level of halogen.

The results of studies that compare the performance of halogen and LED LCUs on restorative composites are different than the results in orthodontic research. Restorative composites are activated and tested under very different conditions. In restorative material research, the light guide is aimed directly at a composite sample at varying distances from the surface while the light-cured orthodontic adhesive is cured under a bracket. The results of depth of cure and top/bottom surface hardness studies have limited applicability for orthodontics because the layer of adhesive under a bracket is less than an increment of restorative composite (Swanson *et al.*, 2004; Judy *et al.*, 2006). Unlike the conflicting research on restorative composites in depth-of-cure and hardness studies, orthodontic research generally agrees that the ability of first-generation LED LCUs to polymerize orthodontic adhesives is *not* significantly different than quartz-tungsten halogen (Dunn and Taloumis, 2002; Swanson *et al.*, 2004; Usumez *et al.*, 2004).

## SHEAR BOND STRENGTH

The major force in the oral environment that a bracket must reckon with is occlusal, or shear-like, loading (Reynolds, 1975). Therefore, as an attempt to simulate failure conditions, shear bond strength (SBS) testing is the most common *in vitro* method for the evaluation of orthodontic adhesive systems (Klocke and Kahl-Nieke, 2005). SBS, frequently reported in megapascals (MPa), is a measure of the maximum force required to debond a bracket per unit area of the its pad.

Dunn and Taloumis (2002) were likely the first to publish research comparing the SBS of the first commercially available LEDs to halogen. Two commercially available first-generation LED LCUs (both at 150mW/cm<sup>2</sup> and both with 7 LED elements) and two halogen LCUs (one quartz-tungsten halogen at 400mW/cm<sup>2</sup>, the other a 'high-power' halogen at 1030mW/cm<sup>2</sup>) were found to produce similar SBS with similar cure time. The LED LCUs performed as well as the halogen LCU of more than 6 times the irradiance. Usumez *et al.* (2004) found no difference between a halogen LCU with a 40 second cure time and a first-generation LED at 20 seconds, thereby showing equal results in half the cure time with an early LED light. The general conclusion among the early SBS studies is that LED LCUs perform as well as halogen LCUs with the same or even shorter cure time because LED light more closely matches the optimal absorbance of CPQ. Besides the effect of ideal emission/absorbance spectral overlap, Swanson *et al.* (2004) also concluded that the lower irradiance of the LED is able to adequately cure orthodontic adhesives because the composite layer under a bracket is so thin.

Other tests validated the use of the early LED LCUs. In diametrical tensile strength testing 7 days after cure of a restorative composite, Tolosa *et al.* (2005) found no difference between a halogen LCU (700mW/cm<sup>2</sup>) and a first-generation LED (150mW/cm<sup>2</sup>). Teshima *et al.* (2003) found that that a first-generation LED LCU generated 31-41% more radicals in a CPQ-amine photoinitiator system than a quartz-tungsten halogen LCU. The LED LCU's peak emission was measured at 465nm, halogen at 497nm, and the CPQ peak absorbance of 467nm. Again, the studies both

conclude that light emitted from LEDs is good at activating CPQ due to the fact that the spectral distribution of LED light matches the absorption of CPQ well.

## SECOND-GENERATION LIGHT-EMITTING DIODE

In conclusion of an evaluation of the first generation of LED LCUs, Bennett and Watts (2004) called for an improvement in LED technology. We now have available LED LCUs that boast intensities of about 1000mW/cm<sup>2</sup>, many times higher than those originally available. This 'second-generation' of LED LCUs, also known as high-powered LEDs, generate more power and have been shown to provide superior performance and shorter curing times over early LED LCUs (Price *et al.*, 2003; Wiggins *et al.*, 2004). Second-generation LED LCUs are defined by a very high intensity chip made of many small LEDs that are bonded to a heat sink which helps to dissipate heat (Bishara *et al.*, 2003). This 'cooling system' allows high power light production without heat-damaging the LED (Uhl *et al.*, 2004).

The amount of literature supporting the use of second-generation LED LCUs is deepening. Studies have found the quality of their activation of restorative composite polymerization, as measured by hardness and depth of cure, to be equal to or better than other LCUs (Uhl *et al.*, 2004; Wiggins *et al.*, 2004; Aravamudhan *et al.*, 2006). Uhl *et al.* (2004) produced greater depth of cure with all composites tested in a comparison of a second-generation LED LCU (901mW/cm<sup>2</sup>) and a quartz-tungsten halogen LCU (860mW/cm<sup>2</sup>) of similar irradiance. As in previously mentioned studies, it was found

that hardness testing *does* discriminate between the performance of LED and halogen LCUs in polymerizing restorative composites with co-initiators. Wiggins *et al.* (2004) found a second-generation LED (1000mW/cm<sup>2</sup>) to provide equal depth of cure and less increase in temperature compared to a high-intensity halogen LCU with a focusing light guide (1500mW/cm<sup>2</sup>).

The majority of the research on the effect of light source type on the SBS of orthodontic adhesive systems has concluded that second-generation LED LCUs are as good as or better than halogen LCUs at initiating polymerization and often with less cure time. Bishara *et al.* (2003), Loretto *et al.* (2004), Swanson *et al.* (2004), Mavropoulos *et al.* (2005), Turkkahraman and Kucukesmen (2005), and Thind *et al.* (2006) all showed that the second-generation LED LCUs provided adequate SBS.

In an attempt to ascertain minimum cure time of metal brackets with a second-generation LED LCU, Mavropoulos *et al.* (2005), found that 10 seconds of cure time with the Ortholux LED (3M Unitek, Monrovia, CA) achieved similar SBS compared to 40 seconds with a high-intensity halogen unit. However, the SBS reached with 5 seconds with the LED was significantly lower than 10 seconds with the LED and 40 seconds with the high-intensity halogen.

Gronberg *et al.* (2006) and Silta *et al.* (2005) obtained higher SBS measurements with halogen LCUs than with second-generation LED LCUs. Gronberg (2005) found that, at similar cure times, halogen produced superior SBS. Similar to research on the effects of

light source type on restorative composites, it was suggested that the superior performance of the halogen LCU was possibly due to the fact that halogen light, a broader spectrum of light, has more potential to initiate photoinitiators in the adhesive other than CPQ. The authors, however were not able to obtain proprietary information on the constituents of the composite adhesive used and, therefore, could not say with certainty that any co-initiators could have been a factor in the results.

Silta *et al.* (2005) obtained lower SBS with an Ortholux LED than with the Optilux 501 (Demetron, Danbury, CT) a high power halogen LCU. These results, however, are in direct opposition to those of Mavropoulos *et al.* (2005) who achieved statistically similar SBS values with the same LED and halogen LCU models used by Silta *et al.*

## TIME AFTER CURE

It has been well documented that the strength of the bond between a bracket and a tooth increases significantly with time after cure (Greenlaw *et al.*, 1989; Chamda and Stein, 1996; Rock and Abdullah, 1997; Bishara *et al.*, 1999; Bishara *et al.*, 2002; Evans *et al.*, 2002; Movahhed *et al.*, 2005; Silta *et al.*, 2005; Turk *et al.*, 2007). Studies that have evaluated the SBS of orthodontic adhesives over time have found the SBS to increase significantly from between 2 to 60 minutes on the low end up to 24 to 30 hours on the upper end. For instance, Evans *et al.* (2002) obtained 25% higher SBS at 24 hours compared to 5 minutes. Turk *et al.* (2007) showed nearly twice the strength at 24 hours compared to 5 minutes. Bishara *et al.* (1999) obtained 24-hour SBS values two times

the strength at 30 minutes. And lastly, Greenlaw *et al.* (1989) demonstrated SBS results at 30 hours nearly four times those at 1 hour.

Most SBS studies are done at 24 hours after cure. At 24 hours, polymerization is essentially complete and only minor changes in SBS will be expected (Klocke *et al.*, 2004). This time point, however, does not represent the typical clinical bonding situation, where an arch wire is placed just minutes after bracket placement and adhesive cure (Turk *et al.*, 2007). It has been hypothesized that the cause of this phenomenon of increased bond strength over time is the diffusion of free radicals produced at the completely illuminated periphery of the composite into the 'darker' areas under the bracket's center (Greenlaw *et al.*, 1989; Chamda and Stein, 1996).

## DEGREE OF CONVERSION

The measure of the extent to which the chemical process of composite polymerization proceeds is generally referred to as degree of conversion (DC%) of carbon-carbon double bonds. This is defined as the proportion of carbon-carbon double bonds that have reacted during polymerization to those that were initially present (Nomoto, 1997). With the high molecular-weight monomers, such as Bis-GMA, commonly used in today's composite systems, there is always a considerable amount of carbon-carbon double bonds left unreacted (Yoon *et al.*, 2002). Some factors that have been shown to affect the DC% are the total light energy density exposed to the composite (James *et al.*, 2003), the shade of composite (Aravamudhan *et al.*, 2006), the time after initial



cure (Bishara *et al.*, 2003), and the spectral distribution of the light source (Emami and Soderholm, 2005; Yoon *et al.*, 2002).

The highest possible DC% of a composite should be achieved for optimal physical and mechanical properties (Asmussen *et al.*, 1982). It has been shown that incomplete polymerization of composite can cause bond failures (Swanson *et al.*, 2004) and other consequences such as retention failures of restorative composites, higher solubility, and adverse pulpal response to unpolymerized monomer (Blankenau *et al.*, 1991). On the other hand, it has been shown that *adequate* degree of conversion brings about better mechanical properties of a composite: higher tensile and compressive strength, better solubility and degradation, and higher flexural modulus of elasticity (Eliades *et al.*, 1995).

DC% can be measured in various ways such as Fourier Transform Infrared (FTIR) spectroscopy, Near Infrared spectroscopy, High Performance Liquid Chromatography, and differential scanning calorimetry. Mills *et al.* (2003) stated that the best way to evaluate the performance of an LCU is to measure and compare the physical and mechanical properties of the composite. The “second best” method is to perform indirect tests, such as FTIR spectroscopy, to evaluate the degree of cure achieved with a certain LCU (Mills *et al.*, 2002). FTIR technology is able to quantify the relative amounts of compounds and molecules in a sample by measuring the absorption of infrared radiation in a range of wavelengths. This is done by quantifying the vibrations of the chemical bonds within the molecule as it absorbs energy (Yoon *et al.*, 2002).

The extent to which halogen and LED LCUs polymerize composite has been measured and compared by FTIR analysis. Studies have found that the surface of a sample is fully cured regardless of the light used (Ferracane and Greener, 1984; Rueggeberg *et al.*, 1994; Yoon *et al.*, 2002; Lohbauer *et al.*, 2005; Aravamudhan *et al.*, 2006). Yoon *et al.* (2002) found that a first-generation LED LCU, when cured with enough time to emit the same total light energy density as the halogen LCU, was able to achieve similar DC% as a quartz-tungsten halogen. Aravamudhan *et al.* (2006) evaluated the DC% of 7 LED LCUs compared to halogen and found no correlation between DC% at the surface and the power density of the LCU. In a comparison of LED and halogen LCUs with equalized total light energy density, Schneider *et al.* (2006) found that the DC% increased by a small amount (3-9%) from 24 hours to one month.

Kauppi and Combe (2003) stated that further research with FTIR in an orthodontic model would be of value in determining whether there is a correlation between the SBS and the DC% of an adhesive. To date, no study has been published that measures the DC% and SBS of an orthodontic light-cured adhesive activated with halogen and LED. Furthermore, the effect of time on SBS and DC% has not been evaluated. The ability of our adhesive systems to effectively bond brackets to teeth and to maintain the bond throughout treatment is a significance issue to both clinician and patient.

## OBJECTIVES

The overall goal of this project is to evaluate over time the shear bond strength (SBS) and degree of conversion (DC%) of an orthodontic resin-composite adhesive activated with different light sources and to determine whether there is a correlation between SBS and the DC%. Two different visible light-curing units will be tested, a quartz-tungsten halogen LCU and a high-intensity (second-generation) light-emitting diode (LED), in order to determine the effect of light-source type on the SBS and DC% of an orthodontic adhesive at various time intervals.

### SPECIFIC AIMS:

1. Determine the effect of two different light curing units, a quartz-tungsten halogen (Ortholux XT, 3M Unitek, Monrovia, CA) and a second-generation LED (Ortholux LED, 3M Unitek), on the shear bond strength (SBS) of an orthodontic adhesive used to bond orthodontic brackets to bovine incisors at 5 minutes, 60 minutes, and 24 hours after initial light exposure.
2. Determine the effect of two different light curing units, quartz-tungsten halogen and LED, on the degree of conversion (DC%) of carbon-carbon double bonds at 5 minutes, 60 minutes, and 24 hours after initial light exposure of an orthodontic adhesive used to bond orthodontic brackets to bovine incisors.
3. Determine the relationship between the DC% and the SBS of an orthodontic composite adhesive cured with two different light curing units.

4. Determine the effect of time after initial light exposure on shear bond strength and degree of conversion.

#### HYPOTHESES OF THE ABOVE OBJECTIVES

1. The second-generation LED LCU will provide a SBS that is significantly higher than the SBS of the quartz-tungsten halogen LCU at 5 minutes, 60 minutes, and 24 hours.
  - Rationale: The increased irradiance of the Ortholux LED and the ideal overlap of its emission spectra with the absorbance of CPQ will result in more photons with increased capacity to activate the composite adhesive compared to halogen.
2. The DC% of composite samples cured with LED will be significantly higher than the quartz-tungsten halogen DC%.
  - Rationale: The increased irradiance of the Ortholux LED and the ideal overlap of its emission spectra with the absorbance of CPQ will result in more photons with increased capacity to activate the composite adhesive compared to halogen.
3. There will be a positive correlation between SBS and DC%.
  - Rationale: As the DC% of a sample increases due to superior light activation, the SBS will increase accordingly.

4. Between time points for both curing units, there will be a statistically significant difference in SBS and DC%: with increased time after polymerization, SBS and DC% will increase.

- Rationale: With time, the physical and mechanical properties of the composite adhesive improve as polymerization continues after light exposure ceases.

## MANUSCRIPT FOR SUBMISSION

### Abstract

*Objective:* The purpose of this study is to evaluate over time the shear bond strength (SBS) and degree of conversion (DC%) of an orthodontic resin-composite adhesive activated with a quartz-tungsten halogen and a high-intensity (second-generation) light-emitting diode (LED) light-curing unit (LCU) in order to determine the effect of light-source type on the SBS and DC% of the adhesive at various time points after light exposure and to determine whether there is a correlation between SBS and DC%.

*Methods:* Brackets were bonded to permanent mandibular bovine incisors (N=138) with an orthodontic adhesive (Transbond XT, 3M Unitek, Monrovia, CA) and cured with either a second-generation LED light source (Ortholux LED, 3M Unitek) for 10 seconds or a quartz-tungsten halogen (Ortholux XT, 3M Unitek) for 20 seconds per manufacturer's instructions. SBS testing was done on a universal mechanical test instrument (Q Test, MTS Sintech, Research Triangle Park, NC) at 5 minutes, 60 minutes, and 24 hours after initial exposure. Immediately after bond failure, composite specimens were removed from the adhesive remaining on each tooth and were analyzed by Fourier Transform Infrared (FTIR) spectroscopy (DS-20/XAD, Analect Instruments, Irvine, CA) to determine the degree of conversion of carbon-carbon double bonds within the polymerized composite adhesive. Data were analyzed with two-way ANOVA and Bonferroni and Tukey post-hoc testing ( $p < 0.05$ ).

*Results:* There was no significant effect of light source type on the SBS or DC% at each time interval after photopolymerization ( $p \geq 0.05$ ). Time, however, was found to have a significant effect on the SBS of orthodontic brackets – SBS increased significantly with time up to 24 hours ( $p < 0.0001$ ). DC%, however, did not change significantly over time ( $p \geq 0.05$ ).

*Significance:* In addition to its practical and operational advantages the Ortholux LED LCU offered similar bond strength in half the cure time than the Ortholux XT quartz-tungsten halogen. Though it is not fully understood why SBS increases with time up to 24 hours while DC% remains constant, crosslinking and polymer entanglement may be factors. Within the limitations of this study, the use of second-generation LED technology is justified.

## Introduction

The polymerization of orthodontic resin adhesives with visible-light curing (VLC) allows time to accurately position a bracket without constraints normally imposed by chemically-initiated adhesive systems (Mavropoulos *et al.*, 2005). However, This extra working time that VLC provides comes at a cost to the clinician in the form of an additional monetary expense of the VLC units and their maintenance and the time needed to expose the adhesive (Bishara *et al.*, 2003). Manufacturers, therefore, have made efforts to provide light-curing units (LCUs) that emit light with qualities that reduce the time necessary to fully cure composite without compromising the composite's physical and mechanical properties.

The polymerization of a light-activated composite is a chemical process that combines monomers into a polymer network. The extent of this polymerization process is dependent upon the number of activated molecules of photoinitiator which is, in turn, determined by the number and wavelength of the photons delivered to the sample. The number of photons delivered to a sample of composite depends upon the total light energy density delivered – a function of light source and the irradiation time. The wavelength of the photons is also determined by the light source and when a light's wavelength more closely matches the maximum absorption of the photoinitiator, activation of polymerization is more efficient (Althoff and Hartung, 2000).



The photoinitiator CPQ, the most commonly used photoinitiator of dental VLC systems (Wiggins *et al.*, 2004) and the key player in the chemistry of photopolymerization, absorbs light in the blue region of the visible light spectrum (Blankenau *et al.*, 1991). Its absorption maximum, the wavelength of light that is absorbed more than any other, has been measured to be between 410 and 500nm (Cook, 1982) with a peak at 467nm (Nomoto, 1997; Teshima *et al.*, 2003). Activation of CPQ in dental composites is more likely when the photons have wavelengths that match the absorption maximum of CPQ (Mills *et al.*, 1999).

The most commonly used LCU is the quartz-tungsten halogen (Mills *et al.*, 1999). Halogen LCUs produce light by incandescence in which a small tungsten filament is heated to several thousand degrees Celsius as an electrical current is passed through it (Althoff and Hartung, 2000). The resistance of the filament causes excitation of atoms over a wide range of energy levels emitting light of a broad spectrum most of which is infrared (Mills *et al.*, 1999; Dunn and Bush, 2002). Only one percent of the energy used to power halogen LCUs is converted into light in the blue region necessary for photopolymerization (Althoff and Hartung, 2000). The mechanism of halogen LCUs and their inherent inefficiency, where most of the energy is given off as heat, are at the root of many of their shortcomings.

The filament of a halogen LCU has a lifetime of 50-100 hours of use (Rueggeberg *et al.*, 1996), is sensitive to shock (Silta *et al.*, 2005), and requires an internal fan to cool the unit. Associated with the fan are air slots that can make the handheld unit difficult

to properly disinfect (Wiggins *et al.*, 2004). It has been well documented that the many sensitive components of a halogen LCU, including the bulb, filter, and reflector, degrade over time due to the extreme heat generated by the element. This degradation causes decreased output that can result in a composite with decreased physical and mechanical properties (Barghi *et al.*, 1994; Miyazaki *et al.*, 1998).

Price *et al.* (2003) stated: “Ideally, new types of dental curing lights should perform as well as or better than a quartz-tungsten halogen light”. Indeed, halogen has been the benchmark for dental LCU performance. In 1995, Mills (1995) proposed the use of solid-state light-emitting diode (LED) technology for the polymerization of dental composite in order to overcome many of the inherent disadvantages of halogen LCUs. The relatively new blue LEDs showed considerable promise for use in the activation of composite because of their long lifetime and consistent output (Mills *et al.*, 1999).

The emission spectrum of LED light makes it ideal for the polymerization of dental composites because the absorption maximum of CPQ is very near the peak of LED light output (Nomoto, 1997). This may also increase the curing efficiency of the LED units, reduce the cure time for each bracket (Jandt *et al.*, 2000), and, thereby, greatly reduce the time required to bond several teeth compared to halogen LCUs.

LED LCUs, usually battery powered and cordless, have a lifetime of 10,000 hours and are, compared halogen LCUs, more resistant to vibration (Silta *et al.*, 2005). With their efficient energy conversion, LEDs produce little heat and, therefore, pose no threat to

pulpal tissue (Yap and Soh, 2003). Lastly, no filters or fans are needed with LED LCUs because the blue light produced does not include light from the infrared or ultraviolet ranges (Gronberg *et al.*, 2006).

The major force in the oral environment that a bracket withstand is occlusal, or shear-like, loading (Reynolds, 1975). Therefore, as an attempt to simulate failure conditions, shear bond strength (SBS) testing is the most common in vitro method for the evaluation of orthodontic adhesive systems (Klocke and Kahl-Nieke, 2005). Dunn and Taloumis (2002) were likely the first to publish research comparing the SBS of the first commercially available LEDs to halogen. Two commercially available first-generation LED LCUs (both at 150mW/cm<sup>2</sup> and both with 7 LED elements) and two halogen LCUs (one quartz-tungsten halogen at 400mW/cm<sup>2</sup>, the other a “high-power” halogen at 1030mW/cm<sup>2</sup>) were found to produce similar SBS with similar cure time. The LED LCUs performed as well as the halogen LCU of more than 6 times the irradiance. Usumeiz *et al.* (2004) found no difference between halogen at a 40 second cure time and a first-generation LED at 20 seconds, thereby showing equal results in half the cure time with an early LED light. The general conclusion among the early SBS studies is that LED LCUs perform as well as halogen LCUs with the same or even shorter cure time because LED light more closely matches the optimal absorbance of CPQ. In addition to the effect of ideal emission/absorbance spectral overlap, Swanson *et al.* (2004) concluded that the lower irradiance of the LED is able to adequately cure orthodontic adhesives because the composite layer under a bracket is so thin.

In the conclusion of an evaluation of the first generation of LED LCUs, Bennett and Watts (2004) called for an improvement in LED technology. We now have available LED LCUs that boast intensities of about 1000mW/cm<sup>2</sup>, many times higher than those originally available. This “second-generation” of LED LCUs, also known as high-powered LEDs, generate more power and have been shown to provide superior performance and shorter curing times over early LED LCUs (Price *et al.*, 2003; Wiggins *et al.*, 2004).

Much of the research on the effect of light source type on the SBS of orthodontic adhesive systems has concluded that second-generation LED LCUs are as good as halogen LCUs at initiating polymerization and often with less cure time. Bishara *et al.* (2003), Loretto *et al.* (2004), Swanson *et al.* (2004), Mavropoulos *et al.* (2005), Turkkahraman and Kucukesmen (2005), and Thind *et al.* (2006) all showed that second-generation LED LCUs provided adequate SBS compared to halogen.

It has been well documented that the strength of the bond between a bracket and a tooth increases significantly with time after cure (Greenlaw *et al.*, 1989; Chamda and Stein, 1996; Rock and Abdullah, 1997; Bishara *et al.*, 1999; Bishara *et al.*, 2002; Evans *et al.*, 2002; Movahhed *et al.*, 2005; Silta *et al.*, 2005; Turk *et al.*, 2007). Studies that have evaluated the SBS of orthodontic adhesives over time have found the SBS to increase significantly from between 2 to 60 minutes on the low end to 24 or 30 hours on the upper end. Evans *et al.* (2002), for instance, obtained 25% higher SBS at 24 hours compared to 5 minutes. Turk *et al.* (2007) showed nearly twice the strength at 24 hours

compared to 5 minutes. And Greenlaw *et al.* (1989) demonstrated SBS at 30 hours to be nearly four times the SBS at 1 hour.

The measure of the extent to which the chemical process of composite polymerization proceeds is generally referred to as degree of conversion (DC%) of carbon-carbon double bonds. This is defined as the proportion of carbon-carbon double bonds that have reacted during polymerization to those that were initially present (Nomoto, 1997). The highest possible DC% of a composite should be achieved for optimal physical and mechanical properties (Asmussen *et al.*, 1982) and it has been shown that incomplete polymerization of composite can cause bond failures (Swanson *et al.*, 2004)

The extent to which halogen and LED LCUs polymerize composite has been measured and compared to halogen by FTIR analysis. Studies have shown that the surface of a sample is fully cured regardless of the light used (Ferracane and Greener, 1984; Rueggeberg *et al.*, 1994; Yoon *et al.*, 2002; Lohbauer *et al.*, 2005; Aravamudhan *et al.*, 2006). Aravamudhan *et al.* (2006) evaluated the DC% of 7 LED LCUs compared to halogen and found no correlation between DC% at the surface and the power density of the LCU.

Kauppi and Combe (2003) stated that further research with FTIR in an orthodontic model would be of value in determining whether there is a correlation between SBS and the DC% of an adhesive. To date, no study has been published that measures the DC% and SBS of an orthodontic light-cured adhesive cured with halogen and LED.

Furthermore, the effect of time on SBS and DC% has not been evaluated. The ability of our adhesive systems to effectively bond brackets to teeth and to maintain the bond throughout treatment is a significance issue to both clinician and patient.

The overall goal of this investigation is to evaluate over time the shear bond strength (SBS) and degree of conversion (DC%) of an orthodontic resin-composite adhesive activated with different light sources and to determine whether there is a correlation between SBS and the DC% of an adhesive. Two different visible light-curing units will be tested, a quartz-tungsten halogen LCU and a high-intensity (second-generation) LED LCU in order to to determine the effect of light-source type on the SBS and DC% of an orthodontic adhesive at various time intervals.

## **Materials and Methods**

A pilot study was completed. to establish the workability and reproducibility of the proposed protocol.

### TEETH USED

Using extraction forceps engaged around the cervix of the teeth, the examiners extracted 138 fresh bovine permanent mandibular incisors from Holstein cattle (Walt's Meats, Woodland, WA) more than 24 months old. Teeth were randomly assigned to one of 6 groups of 23 teeth each: three groups cured with a quartz-tungsten halogen (Ortholux XT, 3M Unitek, Monrovia, CA) and tested at either 5 minutes, 60 minutes, or 24 hours after initiation of light exposure, and three cured with a battery powered second generation LED (Ortholux LED, 3M Unitek, Monrovia, CA) and tested at either 5 minutes, 60 minutes, or 24 hours after cure (Figure 1).

The bovine incisors were acquired in compliance with Title 9 Part 314 of the Electronic Code of Federal Regulations (e-CFR) use of animals and animal products (e-CFR, 2006). Mandibular bovine incisors were chosen because of their availability compared to human incisors and the ability to consistently obtain flaw-free bonding surfaces. Furthermore, the morphology of the bovine mandibular incisor is similar to that of human upper incisors (Mavropoulos *et al.*, 2005). It has been shown that bovine enamel

is similar to human enamel composition and adhesive strength to bovine enamel is equal to or slightly lower than human enamel (Oesterle *et al.*, 1998).

The teeth were stored in 0.5% chloramine-T solution at 37 degrees Celsius. The teeth were stored for no more than 8 weeks, during which time the solution was regularly changed to avoid the potential of excess bacterial growth (Usumez *et al.*, 2004). All teeth were examined with 20X stereomicroscopic magnification for any caries, cracks, or flaws which might effect bonding. Any teeth with these defects were omitted. Teeth remained in the chloramine-T solution at all times except for the brief mounting and bonding procedures.

## MOUNTING TEETH

Teeth were mounted vertically upright into clear square acrylic tubes (Thermo, Sterling, NJ) with a lumen of 1.0 square inch and 1.125 inches in length to provide standardization and stabilization during shear testing. The roots and cervical third of the crown were imbedded into light-cured acrylic (Triad TruTray, Densply, York, PA). A custom paralleling jig was used to ensure that the flattest area in the middle of the facial surface of the crown no closer than 3mm from the incisal edge was parallel to the direction of shear force to be applied. Care was taken not to touch the facial surface of the crown with the mounting material. The mounting material was cured in a light-cure unit (Triad Curing Unit, Densply, York, PA) for 4 minutes after which the acrylic tube was removed leaving a tooth mounted in a square block of acrylic (Figure 2).



## TOOTH CONDITIONING

Each step of enamel conditioning, bonding, and testing was performed by the same investigator to ensure consistency. Teeth were cleansed with non-fluoride pumice slurry for 20 sec with a rubber prophylactic cup on a slow speed handpiece, rinsed with water for 15 seconds, and then dried with oil-free air for 5 seconds. The teeth were etched with 37% Phosphoric acid etchant solution (3M Unitek, Monrovia, CA) for 15 seconds as per manufacturer's instructions and rinsed for 15 seconds with an air-water syringe. Teeth were dried for 10 seconds after which a chalky white appearance was consistently obtained. A thin film of Transbond XT adhesive primer (3M Unitek, Monrovia, CA) was placed as uniformly as possible over the entire etched area with an applicator brush and cured for 5 seconds with the same light intended to cure the adhesive.

## BONDING PROCEDURE

Zero degree tip, zero degree torque upper right central incisor brackets (Victory series, 3M Unitek, Monrovia, CA) with a pad area, reported by the manufacturer, of 10.84mm<sup>2</sup> received an adequate amount of orthodontic adhesive (Transbond XT, 3M Unitek, Monrovia, CA; Table 2) to cover the entire pad. The brackets were oriented on the tooth similar to the orientation of a human incisor and pressed onto the tooth with a force of one pound (454.5 grams) using weight on a vertically oriented piston centered

on the bracket between the tie-wings. Before curing, all excess composite was carefully removed with a sharp explorer.

Samples were cured in random order with either the Ortholux LED (3M Unitek, Monrovia, CA) or the quartz-tungsten halogen Ortholux XT (3M Unitek, Table 1). The irradiance of each LCU was measured with a lab-grade power meter (PowerMax 5200, Molecron Detector, Inc., Portland, OR; Table 1) Exposure time for the halogen groups was 20 seconds total (10 seconds mesial, 10 seconds distal) and the LED groups received 10 seconds total (5 seconds mesial, 5 seconds distal). Both curing times are those recommended by the manufacturer for curing Transbond XT on a metal bracket. The automatic shutoff time of each LCU was verified with stop watch. The battery-powered LED light was charged for a full 24 hours prior to testing and was kept on its charging base while not in use. To ensure consistent output throughout the investigation, the irradiance of the two LCUs was measured with a Demetron 100 (Demetron, Danbury, CT) and recorded before and after each session. During light exposure, the light guide tip was aimed at the proximal tooth-bracket interface at a 45 degree angle and held by hand as closely as possible without touching the bracket.

#### SHEAR BOND STRENGTH TESTING

The reviewers were appropriately masked as to LCU type for the 1 hour and 24 hour groups. However, masking the 5 minute groups was not feasible and, therefore, the examiners were aware of which light had been used on a particular tooth. Teeth that

were to be shear tested at 5 minutes after initiation of cure were immediately loaded into the universal mechanical test instrument (Q Test, MTS Sintech, Research Triangle Park, NC), whereas, in order to prevent desiccation of the teeth and to simulate the moist oral environment, teeth to be tested at 1 hour and 24 hours were placed in 37 degrees deionized water 15 minutes after cure. Delaying the placement of the 1 hour and 24 hour teeth into water by 15 minutes was done to simulate a clinical bonding procedure where a bracket is placed and cured onto a tooth and remains isolated and dry until all other brackets are placed and isolation removed.

Teeth in their acrylic blocks were mounted crown-up into a vise on the Q test machine with the edge of the chisel aligned parallel to the body of the bracket in the ligature groove between the pad and the incisal tie wings (Figure 3A, 3B). The chisel was set just away from the pad behind and the tie wings in front to avoid the effects of friction or binding (Figure 3C). One minute prior to initiating shear testing, the chisel was advanced at a crosshead speed of 0.5 mm/minute in an incisogingival direction until the edge of the chisel touched the bracket with a force of 0.25 pounds. At this point the chisel was automatically stopped. This preload procedure was used to bring the chisel to the bracket to remove any time variations of engaging the bracket thereby providing a uniform starting time point for all samples. At the exact indicated time after initiation of irradiation, the chisel was advanced at the same crosshead speed of 0.5 mm/min until bond failure. A crosshead speed of 0.5mm/minute was used because it has been shown that SBSs are greater and that the coefficient of variation is much less than at higher

crosshead speeds (Bishara *et al.*, 2005). The load at failure was recorded in pounds then converted to megapascals (MPa).

## DEGREE OF CONVERSION

Immediately after SBS testing, specimens of composite approximately 100  $\mu\text{m}$  long and 50  $\mu\text{m}$  thick were removed with a sharp blade from the center of the composite pad that consistently remained on the teeth after bracket failure. Taking specimens from the center of the area once covered by the bracket decreased the possibility of capturing a sample in the oxygen-inhibited regions likely to be found at the periphery of the adhesive. The composite samples were analyzed by Fourier transform infrared (FTIR) spectroscopy (DS-20/XAD, Analect Instruments, Irvine, CA, USA) with a 30-scan transmission technique on a KBr crystal with a nitrogen gas purge for a consistently stable background (Bang *et al.*, 2004; Ferracane and Greener, 1984). Seven uncured adhesive samples were analyzed with FTIR in order to obtain a mean uncured absorbance spectra for DC% calculation. The ratio of remaining carbon-carbon double bonds for each sample's spectrum was determined by the standard baseline method and by comparing the peak absorbance of the aliphatic carbon-carbon double bonds (C=C) at 1638  $\text{cm}^{-1}$  with the aromatic C=C at 1609  $\text{cm}^{-1}$  (Yoon *et al.*, 2002). DC% was calculated using the following formula (Ferracane and Greener, 1984):

$$\text{DC\%} = 100\% \times \left( \frac{[\text{Abs}(1638\text{cm}^{-1})/\text{Abs}(1609\text{cm}^{-1})]_{\text{cured}}}{[\text{Abs}(1638\text{cm}^{-1})/\text{Abs}(1609\text{cm}^{-1})]_{\text{uncured}}} \right)$$

## STATISTICAL ANALYSIS

The preliminary data of the pilot study were used to determine the number of samples in each group. The mean and standard deviation of SBS and DC% were calculated for each LCU at each time point . To analyze how SBS and DC% related to light source type and time after light exposure, the different groups were compared by two-way analysis of variance (ANOVA) with Bonferroni and Tukey post-hoc tests. Linear regression analysis was performed to evaluate any potential correlation between the DC% and the SBS of a sample. Statistical analyses were performed at the significance level of  $P, \alpha < 0.05$ .

## Results

Descriptive statistics, mean SBS and standard deviation, for each of the six groups are reported in Table 3 and in Figures 4 and 5. The effect of light source type on SBS was not statistically significant ( $p=0.13$ ; Table 4). Time after initial light exposure, on the other hand, was found to significantly affect SBS ( $p<0.001$ ; Tables 4 and 5). The interaction of light and time with respect to SBS was found not to be significant ( $p=0.4043$ ).

All combinations with respect to time were found by Bonferroni testing to be significant except for LED 5 minute vs. 1 hour. Therefore, with respect to LED, the 5 minute and 1 hour groups were no different while both had significantly lower SBS values than the 24 hour samples. For halogen, the 5 minute group had significantly lower SBS than the 1 hour group which had significantly lower SBS than the 24 hour group.

Descriptive statistics for the mean degree of conversion (DC%) of carbon-carbon double bonds and standard deviation for each of the six groups are reported in Table 6 and in Figures 6 and 7. Statistical analysis with 2-way ANOVA and Bonferroni post-hoc testing on the FTIR analysis shows no significant difference in DC% with respect to light source type or time (Table 7 and 8). Linear regression analysis and the coefficient of determination reveal that very little change in SBS can be attributed to varying DC% of a sample (Table 9).

## Discussion

Overall, the difference in SBS produced by the LED and halogen LCUs was not statistically significant at each time point. Therefore, light source type had no effect on the SBS of the orthodontic adhesive. On the other hand, time after cure had a significant effect on the SBS of composite adhesive regardless of LCU type – SBS increased from 5 minutes up to 24 hours. Neither light source type nor time after light exposure had an effect on the DC% of the adhesive. Furthermore, the DC% of a sample had no apparent correlation with the SBS of that sample.

Bond strength testing of orthodontic adhesive systems is most often evaluated by shear bond strength testing. However, the results of bond strength testing depend on the mode of testing; shear, tension, and torsion tests, in vitro methods for evaluating bond strength, produce different results (Katona and Long, 2006). Of these, SBS is the most frequently used method (Klocke and Kahl-Nieke, 2005) and, therefore, one is more likely to find results with which to compare. Furthermore, in vitro SBS testing is an attempt to simulate occlusal loading, the most significant in vivo conditions under which a bracket will be placed (Reynolds, 1975).

One limitation of this study is that only one adhesive was evaluated. Ideally, several adhesives would be tested to validate light source comparison. Transbond XT is an adhesive commonly used in clinical practice and is well documented in SBS studies. A PubMed search of the material results in 155 article or abstract hits. The use of more

than one LED LCU and more than one halogen LCU could also have provided more information on the effective differences of LED and halogen light in their ability to polymerize composite.

The results of SBS testing are affected by many variables and study parameters such as LCU type (Silta *et al.*, 2005), cure time (Mavropoulos *et al.*, 2005), time after cure (Turk *et al.*, 2007), light-tip distance (Cacciafesta *et al.*, 2005), type of teeth bonded (Oesterle *et al.*, 1998), type of adhesive (Sfondrini *et al.*, 2001), adhesive layer thickness (Neugebauer *et al.*, 2004), location of debonding force (Klocke and Kahl-Nieke, 2005), direction of force application (Klocke and Kahl-Nieke, 2006), and crosshead speed of the testing device (Bishara *et al.*, 2005). By equalizing all test conditions we attempted to evaluate only the effects of LCU type and time after light exposure on the SBS of an adhesive.

Two variables in our study were difficult to keep perfectly constant: the thickness of adhesive primer applied with an applicator brush and the shape and contour of the facial surface of the bovine incisors. Every other variable was controlled as much as possible by having a strict protocol and by having procedures done by the same examiner. Despite the care taken to keep all testing parameters constant, wide ranges of SBS were obtained. The coefficient of variation (ratio of standard deviation to mean) of our SBS results (.18 - .27) was similar to or less than other recent SBS studies (Bishara *et al.*, 2003; Mavropoulos *et al.*, 2005; Silta *et al.*, 2005; Thind *et al.*, 2006). As with most fracture-type studies, wide ranges and large standard error are expected.



In vitro testing allows the examiner to strictly control many variables and test conditions such as etch and rinse time, temperature and humidity, cure time, and debonding force and direction, and adhesive layer thickness while eliminating some of the potential clinical problems associated with bonding such as saliva contamination, access, and occlusion. However, variations in materials and methods of in vitro SBS testing make direct comparison between in vitro results “difficult if not impossible” (Fox *et al.*, 1994). We will, therefore, make only relative comparisons with other research.

The results of our investigation on the effects of light source type on SBS of orthodontic brackets are consistent with recent investigations: second-generation LED LCUs provide equal or better SBS values compared to halogen LCUs (Bishara *et al.*, 2003; Swanson *et al.*, 2004; Mavropoulos *et al.*, 2005; Thind *et al.*, 2006). In a study evaluating early LED LCUs, Bishara *et al.* (2003) obtained higher SBS with the LED compared to a halogen LCU even though the LED had the lower irradiance of the two. This difference, however, was not statistically significant, and, therefore, it was concluded that the lights perform equally well. The results of Swanson *et al.* (2004) also indicated that LED light provides superior SBS and were, however, statistically significant.

Thind *et al.* (2006) evaluated the same models of LED and halogen LCUs investigated in our study. It was found that Ortholux LED achieves similar 24 hour SBS (6.7 MPa

+/- 2.25) compared to the Ortholux XT halogen (6.83 MPa +/- 2.68). However, the cure time used in their investigation was 20 seconds for both units; not the manufacturer's recommended cure times as we used: 10 seconds for the Ortholux LED and 20 seconds for the halogen LCU. Overall comparison of our results to the study by Thind *et al.* does show us that there is no added benefit as measured by SBS to doubling the cure time of the Ortholux LED from 10 seconds to 20 seconds.

Mavropoulos *et al.* (2005) found that the Ortholux LED with a cure time of 10 seconds was able to produce similar in vitro 24 hour SBS to bovine incisors as a high-intensity halogen LCU. The cure time of 10 seconds per tooth, the manufacturer-recommended cure time for the Ortholux LED (1000mW/cm<sup>2</sup>), performed as well as the high-powered halogen LCU (900mW/cm<sup>2</sup>) in 40 seconds, one-quarter the time.

Silta *et al.* (2005) evaluated the same lights as Mavropoulos *et al.* (2005), but found that 20 seconds with the Ortholux LED produced significantly *lower* SBS than halogen. These results, however, are not consistent with the present study and most of the SBS research comparing LED to halogen. Differences in experimental setup may account for the results. Silta *et al.* used human molars, a self-etch primer, adhesive pre-coated (APC) brackets, and a 1mm/minute crosshead speed at 30 minutes while Mavropoulos *et al.* used bovine incisors, standard etch followed by adhesive primer, non-APC brackets, and a 0.5mm/minute crosshead speed at 24 hours.

In the first study evaluating the effect of the new LED LCUs, Dunn and Taloumis (2002) achieved similar SBS with first-generation LED lights compared to a high-output halogen LCU with nearly 7 times the irradiance. It was concluded that the LED LCUs performed as well because of their close spectral match of the absorption range of CPQ and because the composite under a bracket is much thinner than the maximum 2mm usually recommended for restorative purposes. Swanson *et al.* (2004), who also found the first-generation of LED LCUs to perform at the level of halogen LCUs, concluded that the close spectral match of LED light to CPQ makes the relatively new diode technology ideal for curing orthodontic composite adhesives.

Gronberg *et al.* (2006) performed an investigation on the cure times of a second-generation LED LCU also using Transbond XT as the adhesive. It was found that the halogen LCU with 20 seconds of cure time performed better than the LED LCU with the same cure time. It was hypothesized that this difference was due to the fact that the quartz-halogen LCU produces a broader spectrum of light compared to the LED. Extra coverage of the light spectrum may cause initiation of photoinitiators in Transbond XT that are activated at less than 410nm compared to CPQ's maximum absorbance of 467nm (Nomoto, 1997; Teshima *et al.*, 2003). At and below 410nm, LEDs are essentially powerless while halogen LEDs, with their wide spectrum, provide a significant amount of power (Mills *et al.*, 2002). Since the spectrum of LED light is so narrow compared to Halogen light, it will not activate some of the other photoinitiators that may be present in Transbond XT. We were informed by the manufacturer of Transbond XT that there are indeed "other initiators" within the adhesive. The

proprietary nature of the information related to composition of the adhesive precluded the authors of the present investigation from ascertaining the nature these “other initiators” and if the addition of photoinitiators other than CPQ may be the reason higher-irradiance narrow-spectrum LED LCU did not outperform the halogen unit.

Increased strength with time after cure is a common finding in SBS studies. Many have found SBS to increase significantly from between 2 to 60 minutes on the low end to 24 or 30 hours. Our study found a 56% increase (5.0MPa) from 5 minutes to 24 hours and 23% (2.67MPa) from 1 hour to 24 hours with the halogen LCU. SBS increased 58% (5.6MPa) from 5 minutes to 24 hours and 36% (4.1MPa) from 1 hour to 24 hours with the LED LCU.

From 5 minutes to 24 hours, Evans (2002) showed a mean increase of 25% (7.5MPa) in SBS of an adhesive cured with various halogen LCUs and light guides. Chamda (1996) showed an increase of 61% (4.4MPa) from immediately after cure to 24 hours. Bishara *et al.* (2002) showed a 100% increase (5.2MPa) in SBS from 30 minutes to 24 hours in a VLC adhesive system.

In light of the effect of time on the SBS of orthodontic brackets, researchers have advised clinicians to consider delaying the placement of the initial arch wire (Greenlaw *et al.*, 1989) or use caution and apply only light forces if an arch wire is to be placed soon after bracketing (Bishara *et al.*, 2002). It may also be advised to caution

orthodontic patients to avoid hard foods and exercise extra care while eating for the initial 24 hours in braces.

DC% values in our research, overall mean DC% = 60.6%, SD = 9.3, were similar to the results of studies that have quantified the extent of polymerization of composite samples exposed directly under the tip of an LCU: 50-70% 0.5mm into the sample (Lohbauer *et al.*, 2005), 40-65% at 1mm (Yoon *et al.*, 2002). Our similar results may be surprising considering the composite adhesive in our study was cured under stainless steel brackets that completely block light from directly reaching the composite. This similarity in DC% may be due to the fact that the layer of composite under a bracket is so thin (Dunn and Taloumis, 2002; Swanson *et al.*, 2004).

FTIR analysis was performed in our investigation to help us to further understand how DC% might be related to SBS. Time and light source type had no apparent effect on DC% obtained from composite samples previously under the center of the bracket. In our investigation, there was also no significant correlation between the DC% of a sample and its SBS. In an evaluation of the DC% of various composite resins, Uctasli *et al.* (2005) also found that LED and Halogen LCUs of similar irradiance provided the same DC% even though the total cure time of the LED was half that of the halogen LCU. It was also found, unlike our investigation, that DC% increased with time from 0 to 15 minutes after cure. In their study, most of the increase in DC% was seen in the first 5 minutes, therefore, we may not have seen an increase in DC% with time since we evaluated the cured adhesive no earlier than 5 minutes. Schneider *et al.* (2006) found no

difference in DC% at 24 hours between halogen and LED light when cured with equal total light energy density. Additionally, they found a significant yet very small difference in DC% over time from 24 hours to 1 month after cure. The increase in DC% taken from a sample of composite cured with a halogen LCU for 20 seconds had a mere 2.8% greater DC% from 24 hours to one month after cure.

It is not fully understood how the physical and mechanical properties of light cure composite such as strength and stiffness can increase over time while the DC% appears not to increase significantly. Greenlaw *et al.* (1989) and Chamda and Stein (1996) hypothesized that an increase in SBS with time is due to the diffusion of free radicals from the periphery of the adhesive layer where the composite is fully exposed by the LCU. These free radicals, according to the theory, diffuse into the area further under the bracket where they initiate polymerization of composite in these 'darker' areas, thereby increasing SBS. In order for this theory to be true, the initiating potential of free radicals in composite would have to be long-lived. Secondly, one would expect the degree of conversion of carbon-carbon double bonds to increase as these supposed diffusing free radicals initiate additional polymerization of unpolymerized monomers under the bracket. In opposition to this theory of free radical diffusion, a free-radical within composite is only active for 0.1 to 1.0 seconds and will initiate the polymerization of about 50 resin monomers. Furthermore, the deactivation mechanisms of a composite limit the amount of polymerization (Althoff and Hartung, 2000). The results of our FTIR measurements, taken from composite samples once under the center of the bracket, indicate that the DC% of carbon-carbon double bonds does not increase

from 5 minutes to 24 hours and, therefore, the increase in SBS over time is likely not due to free radical diffusion causing increased DC%.

DC% is not the only measure of the quality of cure in an adhesive system. As a composite is cured, monomers combine chemically to form growing linear polymer chains. DC%, a relative measure of how many monomers react to join one of many polymer chains, does not account for crosslinking, chemical bonds that form between chains, nor does it account for the physical entanglement of the many polymer chains in a composite network. Two different composite samples may have similar DC% and exhibit different physical and mechanical properties due to different amounts of crosslinking and entanglement (Soh and Yap, 2004). While we see similar DC% between 5 minutes, 60 minutes, and 24 hours, the SBS increase is possibly due to the development of polymer crosslinking and entanglement.

Though there are obvious differences between in vitro and in vivo SBS investigations and direct comparison of the results has been discouraged (Pickett *et al.*, 2001), it is of some value to understand how LED and halogen LCUs have been shown to compare clinically. Krug and Conley (2005) performed a split-mouth evaluation that compared the clinical performance of halogen and LED LCUs on 554 bonded brackets. There was a mean failure rate of 0.6 brackets per patient with no difference between light source types. Layman and Koyama (2004) found a second-generation LED LCU to perform, in half the cure time, as well as a quartz-tungsten halogen LCU as measured by bond failures over a three month period.

A bond strength of 5.9-7.8MPa was estimated by Reynolds (1975) to be the minimum threshold of an ideal adhesive system. Our mean SBS values for both LCU types at all three time intervals achieved this often cited benchmark. It has been shown that the SBS of metal brackets to permanent bovine incisors is 40 per cent lower than to human teeth (Oesterle *et al.*, 1998). Therefore, had human teeth been used in the present investigation, we would expect about 66% higher mean SBS values for all groups.

Proper maintenance of an LCU is of great clinical importance and if halogen is used, replacement of lamps, filters, and light guides greatly increased performance (Miyazaki *et al.* 1998). Interestingly, at the time of this investigation, production of the Ortholux XT halogen LCU was stopped as the demand for LED LCUs has dramatically increased among orthodontists. Only replacement parts for the Ortholux XT halogen units are now listed in the manufacturer's online catalog.

The manufacturer-recommended cure time for the Ortholux XT halogen LCU is 20 seconds per tooth while 10 seconds is recommended for the Ortholux LED. In a full-mouth bonding of 20 teeth, this 10 second difference per tooth would represent a time savings of 3 minutes 20 seconds. In a practice where 200 patients are fully bonded in one year, this difference equates to over 11 hours of chair time per year. The reduction in cure time may also help to reduce the risk of saliva contamination, which in itself can greatly compromise bond strength (Thind *et al.*, 2006). Other factors in the oral environment such as salivary degradation of composite adhesives, acid from food and



beverages, masticatory activity, patient abuse, and orthodontic mechanics can all negatively affect in vivo SBS (Pickett *et al.*, 2001). The bond strength of an adhesive system is, therefore, a significant factor for the clinician to consider as the oral environment presents with many dangers to the longevity of a bracket's adhesion to a tooth.

## Conclusions

This in vitro study evaluated the effect of time and light source type, quartz-tungsten halogen and LED, on the shear bond strength and degree of conversion of carbon-carbon double bonds of an orthodontic adhesive. Six groups of 23 bovine teeth showed no significant effect of light source type on the SBS or DC% at 5 minutes, 60 minutes, and 24 hours after photopolymerization. Time, on the other hand, was found to be a significant factor in the SBS of orthodontic brackets. SBS increased 56-58% from 5 minutes to 24 hours. DC%, however, did not change significantly over time.

Bond strengths achieved with high-intensity LED light-curing units such as the Ortholux LED (3M Unitek) provide adequate SBS compared to quartz-tungsten halogen technology. In addition to their practical and operational advantages, LED LCUs offer similar bond strength with significantly less cure time. The spectral output of LED is theoretically ideal for CPQ activation and may be the reason for their ability to show equal results in less time.

Regardless of LCU type, The composite resin under a bracket is adequately cured by both LCUs as measured by DC%, possibly because the adhesive layer is so thin. Though it is not fully understood why SBS increases with time up to 24 hours while DC% remains constant, crosslinking and polymer entanglement may be significant factors. It is not known if other photoinitiators within Transbond XT and their interaction with the two LCU types are accountable for the results.

The use of second-generation LED technology is now adequately justified by the literature and supported by this study.

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**Tables:**

**Table 1.** Light-curing units tested

<b>Curing Light</b>	<b>Irradiance</b> (mW/cm <sup>2</sup> )	<b>Irradiance*</b> (mW/cm <sup>2</sup> )	<b>Cure Time</b> (S)
Reported by the Manufacturer			
<b>Halogen</b> Ortholux XT (3M Unitek, Monrovia, CA)	550	986	20
<b>LED</b> Ortholux LED (3M Unitek, Monrovia, CA)	1000	1488	10

\*Irradiance measured with lab-grade power meter (PowerMax 5200, Molelectron Detector, Inc., Portland, OR)

**Table 2.** Transbond XT Composition  
(% by Volume)

<b>Bis GMA</b>	<b>14%</b>
<b>Bis EMA</b>	9%
<b>Fillers</b>	77%
Silane treated quartz	
Silane treated silica	
<b>Curatives</b>	<1%
Camphorquinone	
<b>Others</b>	<1%

**Table 3.** Descriptive statistics for SBS (MPa)  
Halogen (Hal), Light-emitting diode (LED)

	<i>5min Hal</i>	<i>1hr Hal</i>	<i>24hr Hal</i>	<i>5min LED</i>	<i>1hr LED</i>	<i>24hr LED</i>
<b>Mean</b>	8.93	11.30	13.97	9.64	11.20	15.27
<b>S.D.</b>	2.46	2.26	2.49	2.12	2.35	3.06

**Table 4.** Two -Way ANOVA and Bonferroni analysis of SBS

<b>2Way ANOVA</b>	<b>p-value</b>
Interaction	0.4043 (NS)
Light	0.1328 (NS)
Time	P < 0.0001
<b>Bonferroni</b>	
<b>(5min v. 1hr)</b>	<b>p-value</b>
Halogen	P < 0.01
LED	P > 0.05 (NS)
<b>Bonferroni</b>	
<b>(5min v. 24hr)</b>	<b>p-value</b>
Halogen	P < 0.001
LED	P < 0.001
<b>Bonferroni</b>	
<b>(1hr v. 24hr)</b>	<b>p-value</b>
Halogen	P < 0.001
LED	P < 0.001

**Table 5.** One -Way ANOVA and Tukey analysis of SBS

<b>1Way ANOVA (Time as predictor)</b>	
	<b>p-value</b>
Halogen	P < 0.0001
LED	P < 0.0001
<b>Tukey post-test (Halogen)</b>	
	<b>p-value</b>
5min v. 1hr	P < 0.01
5min v. 24hr	P < 0.001
1hr v. 24hr	P < 0.01
<b>Tukey post-test (LED)</b>	
	<b>p-value</b>
5min v. 1hr	P > 0.05 (NS)
5min v. 24hr	P < 0.001
1hr v. 24hr	P < 0.001

**Table 6.** Descriptive Statistics for % Degree of Conversion of C=C Halogen (Hal), Light-emitting diode (LED)

	<b>5min Hal</b>	<b>5min LED</b>	<b>1hr Hal</b>	<b>1hr LED</b>	<b>24hr Hal</b>	<b>24hr LED</b>
<b>Mean</b>	59.58%	59.97%	61.16%	61.59%	59.03%	62.33%
<b>S.D.</b>	8.44%	10.00%	8.86%	9.09%	10.92%	8.52%

**Table 7.** Two -Way ANOVA and Bonferroni analysis of DC%

<b>2Way ANOVA</b>		<b>p-value</b>
Interaction		0.6956 (NS)
Light		0.3960 (NS)
Time		0.7218 (NS)
<b>Bonferroni</b>		
<b>(5min v. 1hr)</b>		<b>p-value</b>
Halogen		P > 0.05 (NS)
LED		P > 0.05 (NS)
<b>Bonferroni</b>		
<b>(5min v. 24hr)</b>		<b>p-value</b>
Halogen		P > 0.05 (NS)
LED		P > 0.05 (NS)
<b>Bonferroni</b>		
<b>(1hr v. 24hr)</b>		<b>p-value</b>
Halogen		P > 0.05 (NS)
LED		P > 0.05 (NS)

**Table 8.** One -Way ANOVA and Tukey analysis of DC%

<b>1Way ANOVA (Time as predictor)</b>		<b>p-value</b>
Halogen		0.7328 (NS)
LED		0.6883 (NS)
<b>Tukey post-test</b>		
<b>(Halogen)</b>		<b>p-value</b>
5min v. 1hr		P > 0.05 (NS)
5min v. 24hr		P > 0.05 (NS)
1hr v. 24hr		P > 0.05 (NS)
<b>Tukey post-test</b>		
<b>(LED)</b>		<b>p-value</b>
5min v. 1hr		P > 0.05 (NS)
5min v. 24hr		P > 0.05 (NS)
1hr v. 24hr		P > 0.05 (NS)



**Table 9.** Linear regression analysis of SBS and DC%

<b>Linear Regression</b>	<b>r<sup>2</sup></b>	<b>Pearson's r</b>
Halogen 5min	0.04787	-0.2188
Halogen 1hr	0.1473	-0.3838
Halogen 24hr	0.07885	-0.2808
LED 5min	0.000604	-0.0246
LED 1hr	0.003371	-0.0581
LED 24hr	0.000642	0.0253

## Figure Legend:

- Figure 1. Assignment of teeth into 6 groups. With each curing-light source, SBS and DC% was tested after one of three time intervals.
- Figure 2. Permanent mandibular bovine incisor mounted in square acrylic block.
- Figure 3. (A) Tooth secured by a vise in the Q Test universal testing machine. (B) Chisel edge parallel to body of bracket. (C) Chisel directed parallel to bonding site.
- Figure 4. The effect of irradiation condition on shear bond strength(SBS) over time. Mean SBS values shown with SD bars.
- Figure 5. Scatter diagram showing the effect of irradiation condition on shear bond strength(SBS) over time. Bars show mean values.
- Figure 6. The effect of irradiation condition on per cent degree of conversion (DC%) over time. Mean DC% values shown with SD bars.
- Figure 7. Scatter diagram showing effect of irradiation condition on per cent degree of conversion (DC%) over time. Bars show mean values.

**Figures:**

Figure 1.

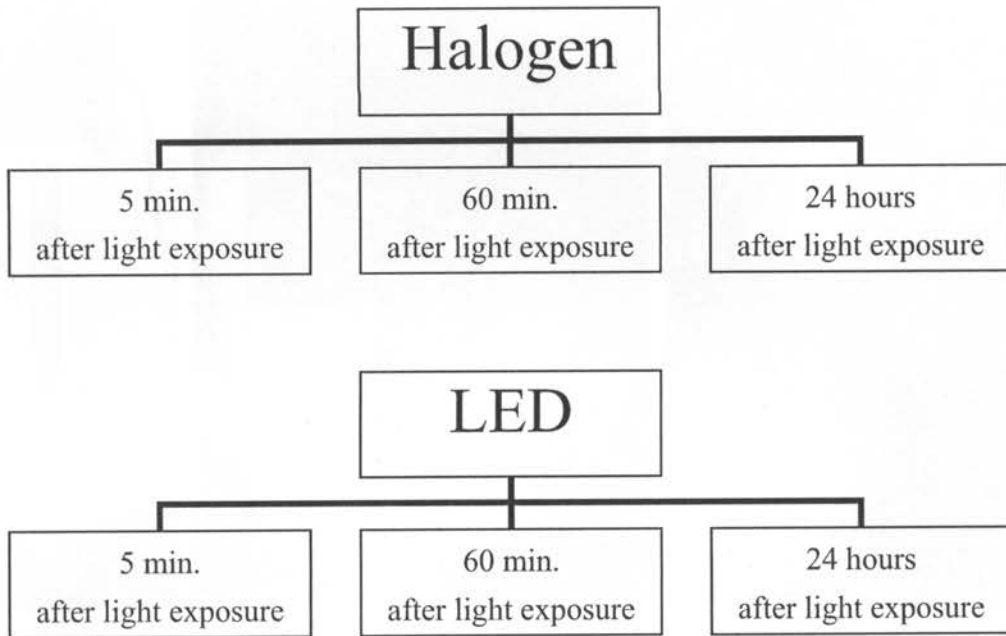


Figure 2.

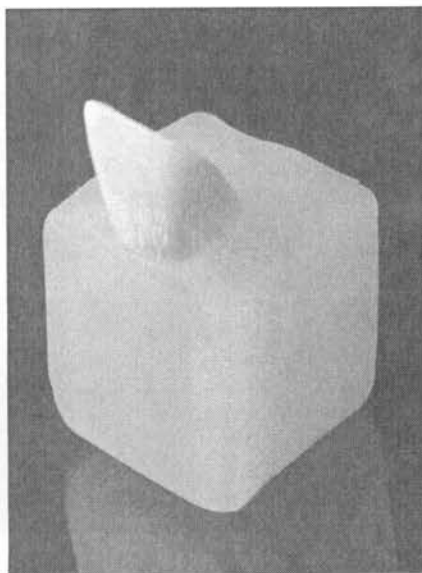


Figure 3

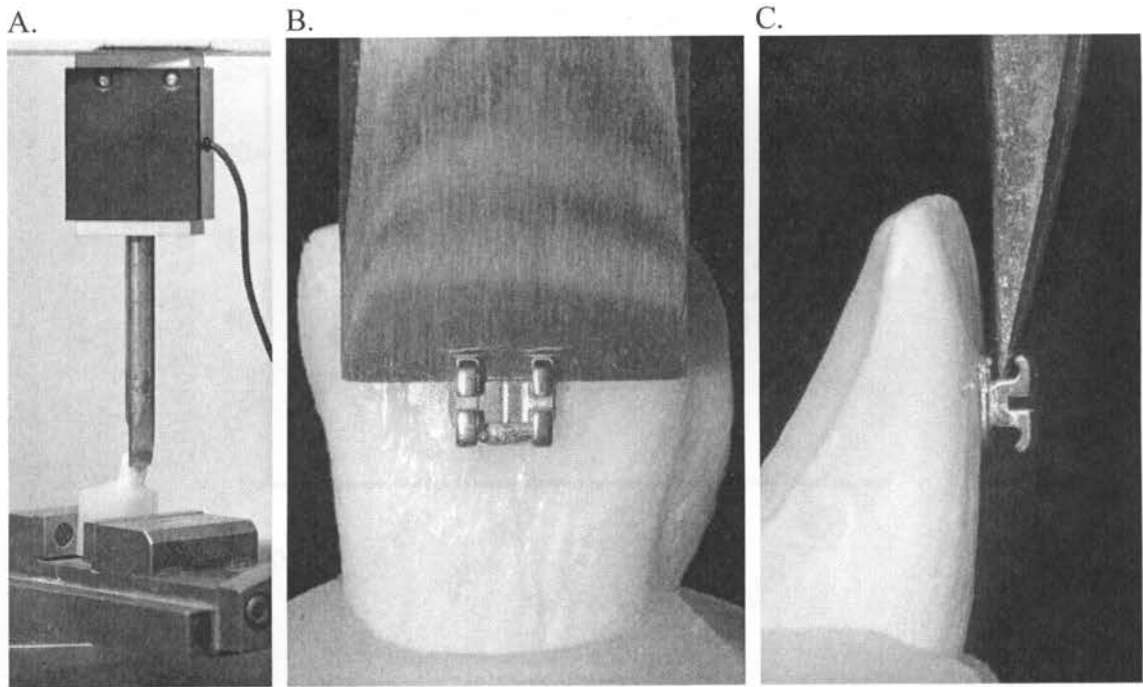


Figure 4.

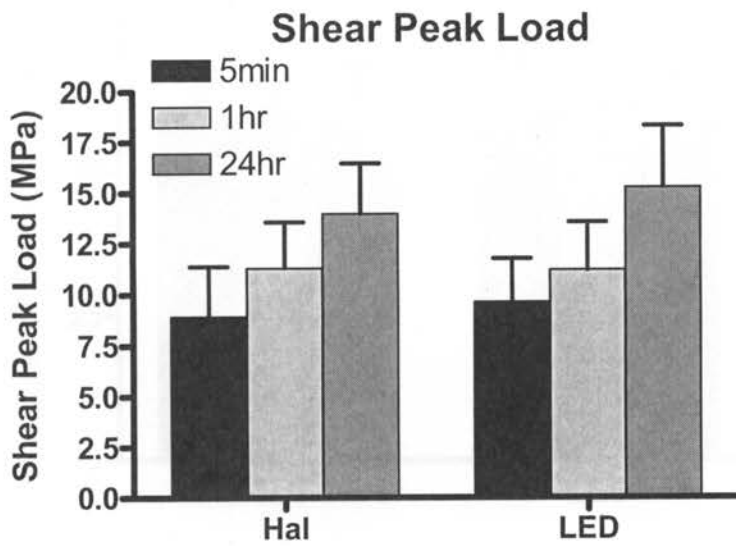


Figure 5.

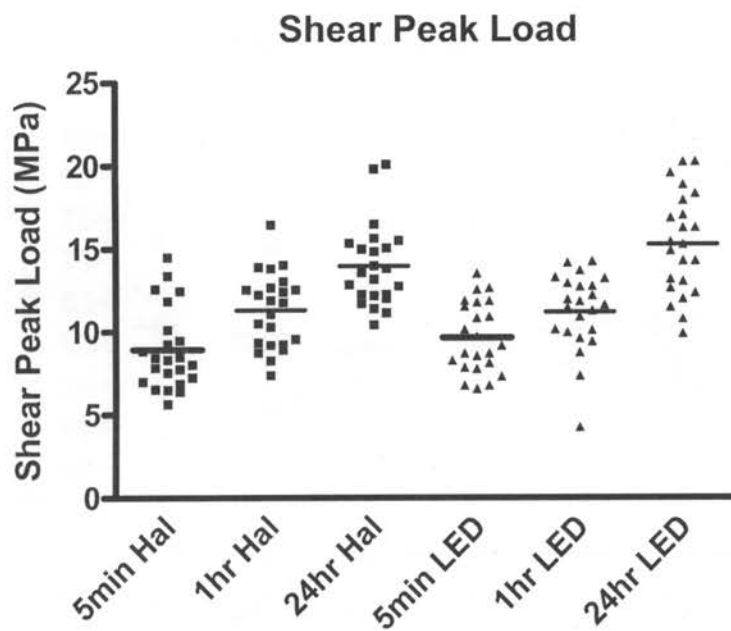


Figure 6.

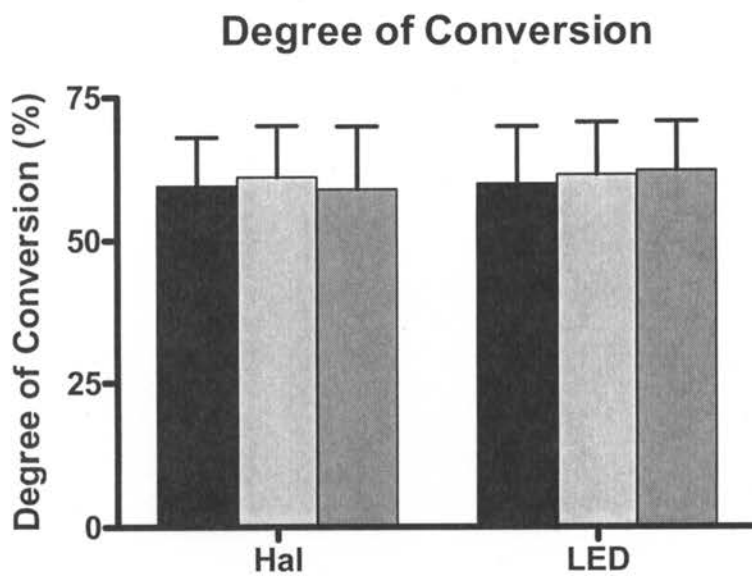


Figure 7.

