ORTHODONTIC BRACKET BOND STRENGTH AND RESIN COMPOSITE ADHESIVE

DEGREE OF CONVERSION ASSOCIATED WITH TYPE OF CURING UNIT AND

TOTAL ENERGY

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ORTHODONTIC BRACKET BOND STRENGTH AND RESIN COMPOSITE ADHESIVE DEGREE OF CONVERSION ASSOCIATED WITH TYPE OF CURING UNIT AND TOTAL ENERGY

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ABSTRACT

This study examined the effect of light curing sources on shear bond strength and degree of conversion of orthodontic resin composite adhesive as function of type of curing unit and total energy, as well as evaluated the correlation between shear bond strength and degree of conversion. Curing units included in the study were plasma arc (PA), light emitting diode (LED) and quartz tungsten halogen (QTH) with 5k, 10k, or 15k mJ/cm^2 of total energy delivered to the orthodontic adhesive by each unit type.

Based on an analysis of variance (α=.05), there was no significant effect of type of curing source on shear bond strength; however, there was a significant effect of total energy, with shear bond strength increasing as energy increased across curing units. For degree of conversion there was a significant effect of curing unit with PA producing higher degree of conversion than LED or QTH, which were not significantly different from each other. There was also a significant effect of total energy on degree of conversion with a significant increase between 5k to 10k mJ/cm^2. There was a positive moderate overall correlation between shear bond strength and degree of conversion.

Results of this study suggest that the PA curing unit is more efficient at polymerization (degree of conversion) of orthodontic resin composite adhesive. However, there is no difference in shear bond strength produced when using PA, LED or QTH curing sources for orthodontic bracket bonding procedures when equal total energy is delivered to the adhesive.

APPROVAL PAGE

The faculty listed below, appointed by the Dean of the School of Dentistry, have

examined a thesis titled "Orthodontic Bracket Bond Strength and Resin Composite Adhesive

Degree of Conversion Associated with Type of Curing Unit and Exposure Time and Total

Energy: Pilot Study "presented by Natalia Henbest, candidate for the Master of Science

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CHAPTER 1

INTRODUCTION

History of Bracket Bonding

Orthodontic treatment is based on application of appropriate forces to move teeth

through the alveolar bone without causing permanent damage to either the teeth or their

attachment to the bone. Several types of tooth movement can occur during the process of

orthodontic treatment, they include: tipping, rotational, bodily, torque and vertical

movements. Orthodontic appliances deliver controlled force to produce desired tooth

movement and they divide into two broad categories: removable and fixed. Fixed appliances

act through attachments fitted directly to the teeth. Advantages of fixed appliances over

removable are: appliances do not get dislodged in the mouth and therefore, reduce treatment

time; less skill is required from the patient to manage the appliance; more tooth movements

are possible (Foster 1990).

The success of a fixed dental appliance depends on the attachments: bands and

brackets being securely attached to the teeth so that they do not become loose during

orthodontic treatment (Millett et al. 2007). Tooth movement is achieved when arch wires

apply force to a tooth via fixed attachments: bands or brackets. Until the 1980s, the only

practical way to place a fixed attachment to a tooth was to adhere it to a band that could be

cemented to a tooth. In the 1980s, a fixed attachment that could be bonded to a tooth using of

the acid-etch bonding systems was developed and it eliminated banding as a single

attachment entity. Bracket bonding is based on the mechanical locking of an adhesive to

irregularities in the enamel surface of the tooth and to mechanical locks formed in the base of the orthodontic bracket (Proffit and Sarver 2007).

Bracket Adhesives

The success of a fixed orthodontic appliance depends on attachments having adequate bond strengths and a low failure rate. Orthodontic attachments are subjected to a large number of forces in the mouth, resulting in a complex distribution of stresses within the adhesive and at its junctions with the enamel and the bracket base (Sunna and Rock 1998). Bond failures between the bracket and the tooth during the treatment slow down the progress of the treatment, and it also could be costly in terms of clinical time, materials and time loss for the patient. Ideally, the adhesive should be: strong enough so the brackets would stay bonded to the teeth for the length of the treatment; the bond between the tooth and the appliance should be not overly strong so upon the removal of the appliance the tooth surface would be damaged; clinically easy to use for the operator; protect against dental caries; be available at a reasonable cost (Millett et al. 2007).

There have been two main types of orthodontic adhesives: acrylic and dyacrylate resins. Since dyacrylate resins offer increased bond strength, they became more popular than acrylic adhesives (Read 1984). Most commonly used dyacrylate resins are based on the bisphenol A glycidyl dimethacrylate (Bis-GMA) monomer (Read 1984; Wilson 1988). The essential parts of the Bis-GMA monomer are the C=C, double bonds at the terminal end of each monomer chain (Wilson 1988). These 'vinyl 'groups are involved in addition polymerization of the monomer into polymer chains as well their cross-linking, which improves rigidity of the final polymer molecule. Bis-GMA monomer is a viscous liquid and

to make it more useable in dentistry, a more fluid monomer triethylene glycol dimethacrylate (TEGDMA) was formulated into composite resin (Garg and Garg 2010). A typical ratio is about 70-75% of Bis-GMA to 25-30% of TEGDMA; however, the higher ratio of TEGDMA increases the chances of polymerization shrinkage (Watts 2001; Garg and Garg 2010). Inorganic fillers are added to the Bis-GMA/TEGDMA resin matrix to produce a composite material, which exhibits improved physical properties (Robertson at el. 2006; Garg and Garg 2010) such as higher strength and modulus and also reduces polymerization shrinkage . The fillers used are some form of glass or ground quartz, which are pretreated with a silane coupling agent to produce a bond between the inorganic hydrophilic filler and the hydrophobic resin matrix (Wilson 1988; Robertson et al. 2006; Garg and Garg 2010). Resin composite adhesives used for orthodontic bracket bonding are an adaptation of composite restorative materials (Watts 2001; Powers and Sakaguchi 2006). Orthodontic composite adhesives differ from restorative bulk composites in the increased proportion of co-monomer in the formulation, which reduces viscosity of the adhesive composites (Eliades and Eliades 2001b). Lower viscosity of orthodontic adhesives provides superior diffusion into the enamel rods and results in improved interfacial adaptation between enamel and the bracket base (Eliades and Eliades 2001b).

Chemically-Cured Adhesives

The chemically-cured composite adhesives were the first systems developed for bracket bonding (Newman et al. 1968; Rachala and Yelampalli 2010). The polymerization of self-cured resin with the two-paste system starts immediately upon mixing; thus the operator is unable to manipulate the setting time, which affects bracketing accuracy and

positioning on the tooth surface (Rachala and Yelampalli 2010). The air bubbles might be incorporated in resins, which are mixed by hand and produce porosity, inhibit polymerization and ultimately weaken the bond strength in the two-paste system (Wilson 1988; Mitchell 1994; Nomoto 1997; Eliades 2006). Two-paste systems were also time consuming due to the mixing time and because several mixes were often required to bond brackets to teeth in both arches (Sunna and Rock 1999).

Light-Cured Adhesives

Introduction of ultra-violet light and later the visible light activated systems solved many deficiencies of chemically cured composite bonding. In both systems the composite resin was polymerized only when the light was applied, thus the operator had virtually unlimited working time and 'command set' of the material, and ability to remove excess adhesive before it has set (Read 1984; Eliades and Eliades 2001b; Cunningham et al. 2002). Ultra violet light systems were time consuming (90 sec per bracket) and since the UV light is poorly transmitted by the tooth structure, perforated or plastic brackets had to be used (Read 1984; Rachala and Yelampalli 2010). Also, there were safety concerns with long-term use of UV systems; therefore, UV systems were quickly replaced with visible light activated systems around 1980 (Sfondrini et al. 2001). Visible light-cured resins are single paste products (Garg and Garg 2010). The drawbacks of light-cured composite adhesives include: time required to cure the adhesive under each bracket, and a possibility of incomplete polymerization of the resin under the bracket due to insufficient exposure to the curing light (Smith and Shivapuja 1993; Sunna and Rock 1999; Rachala and Yelampalli 2010).

Dually-Cured Adhesives

In the 1980s, resins that were both light activated and chemically cured were introduced into dentistry (Smith and Shivapuja 1993). The term 'dual-curable luting composite' refers to an adhesive agent that contains chemical compounds behaving as accelerators and initiators for both chemical- and light-cure and that can benefit from both polymerization systems (Tanoue et al. 2003; Arrais et al. 2008). The main clinical disadvantage of dual-cured composites is that there is a limited working time due to their chemically cured properties. Once initiated polymerization cannot be stopped, and if an operator placed a bracket with a half-cured adhesive or attempted to remove access adhesive, the bond strength would be drastically affected (Smith and Shivapuja 1993).

Over time, light-cured composite adhesives became the most popular adhesive systems used for bonding orthodontic brackets to the teeth (Sfondrini et al. 2002). The popularity is based on the following characteristic that light-cured composite adhesives provide: a reduced risk of contamination, similar material consistency, and a virtually unlimited working time to place bracket on the tooth accurately and remove adhesive flash before initiation of polymerization (Cacciafesta et al. 2004).

Light-curing Sources

Because the majority of orthodontic bracket adhesives are light cured, the light-curing process is an important factor. Light-curing units that are available to dental practitioners have different light intensities, light sources and energy levels ranging from 300 to more than 2000 Mw/cm^2 (Santini 2010). The efficacy of a light-curing unit depends on its ability to produce photo-radiation of appropriate wavelength and intensity to produce optimal number

of free-radicals, which will increase the likelihood of achieving the best possible polymerization (Lynch 2008). The intensity of light radiation is related to the power of the polymerization device, the surface area and the time: the power, which is measured in watts, is defined as work that can be produced over a certain time; the surface area, measured in cm^2, is area over which the light is applied; and the time, measured in seconds, during which the light source is operated (Abate et al. 2001). Curing units that are available today include: halogen curing lights, light-emitting diodes, and plasma arc units.

Quartz Tungsten Halogen

Traditionally, quartz tungsten halogen (QTH) curing lights traditionally have been the most widely used photopolymerization sources by dental practitioners (Nomoto 1997; Rueggeberg 1999; Powers 2002; Nomoto et al. 2004; Yazici et al. 2007; Garg and Garg 2010; Rachala and Yelampalli 2010). QTH curing units have been very popular because they have sufficient light intensity, emit a broad spectrum of usable light and are relatively inexpensive (Burgess et al. 2002). In halogen curing units the light is produced when a tungsten filament, which is housed in a quartz bulb filled with halogen gas, emits electromagnetic radiation (Rueggeberg 1999; Burgess et al. 2002; Robertson et al. 2006; Sherwood 2010). Electric current flows through a thin tungsten filament, which acts as a resistor and generates heat. To generate blue light, the filaments require to be heated to high temperatures emitting a wavelength of a wide spectrum. Therefore, to produce a light beam of a specific waveband in the region of 470 nm, unwanted portions of the spectrum must be filtered out (Rueggeberg 1999; Althoff and Hartung 2000; Burgess et al. 2002; Oberholpez et al. 2005; Santini 2010; Sherwood 2010). The light contains the following filtration

mechanism: a paraboloid dichroic filter (a"cold-mirror"), which removes infrared light; a glass filter removes ultraviolet light; and a blue filter narrows the spectra of visible white light wavelengths to the region of blue wavelength of 470 nm. The light then passes through fiber optic bundle and is emitted from the unit via a light-guide to concentrate the light and to deliver photo-irradiation to the desired location (Burgess et al. 2002; Robertson et al. 2006; Lynch 2008; Sherwood 2010). However, this system is inefficient, because the largest part of the radiative power of halogen light units is wasted and it is the main disadvantage of this type of light source (Lynch 2008; Santini 2010). The light power output is less than 1% of the consumed electrical power (Althoff and Hartung 2000; Oyama et al. 2004; Rachala and Yelampalli 2010). Due to the associated generation of heat, a QTH also requires a cooling system, a fan-generated air current that must pass through slots in the frame, which can make disinfection of the unit problematic (Santini 2010). Another disadvantage of halogen light curing units is that the bulb, reflector and filter can degrade over time, which reduces light output levels and compromises the photopolymerization process (Sakaguchi et al. 1992; Rueggeberg 1999; Burgess et al. 2002; Nomoto et al. 2004; Oberholpez et al. 2005; Yazici et al. 2007; Lynch 2008; Santini 2010). A typical bulb has about 100 hours of life (Oyama et al. 2004; Oberholpez et al. 2005; Robertson et al.2006; Garg and Garg 2010; Sherwood 2010). In restorative dentistry, for the majority of restorative composite resins, a QTH unit with power density in the region of 400 mW/cm^2 requires a 40 second exposure for adequate polymerization of that composite (Strydom 2002; Oberholpez et al. 2005). Typically, the power density of QTH ranges from 400 to 800 mW/cm^2 (Powers 2002; Robertson et al. 2006).

Light Emitting Diode

A light-emitting diode (LED) uses diode technology, which incorporates chips containing "doped cells" (Powers 2002; Rachala and Yelampalli 2010). Electron movement within these cells produces blue light (Lynch 2008). Blue light is generated not by a thermal process, but by a well-defined relaxation of excited electrons (Althoff and Hartung 2000). LED curing units generate blue light of selected wavelength between 400 and 500 nm without requirement of filters by using a semiconductor material system (gallium nitride) (Burgess et al. 2002; Powers 2002; Oyama et al. 2004; Lohbauer et al. 2005; Oberholpez et al. 2005; Sherwood 2010). The chemical composition of the semiconductor can be manipulated to obtain a specific wavelength range with a narrow spectrum distribution (Althoff and Hartung 2000; Mills et al. 2002; Yazici et al. 2007; Santini 2010). LED curing units when compared to the halogen curing units, have much longer lifetimes and undergo little degradation of light output. Also, LEDs are much more efficient in conversion of electric current into a light beam and are very shock and vibration resistant in contrast to their halogen counterparts (Mills et al. 1999; Burgess et al. 2002; Oyama et al. 2004; Lynch 2008). LED curing units available to dentists are lightweight, portable, without need of a cooling system (Burgess et al. 2002; Powers 2002; Oyama et al. 2004; Yazici et al. 2007; Rachala and Yelampalli 2010; Sherwood 2010). Recent advances in LED technology allowed development of high power units comparable to plasma arc curing sources (Oyama et al. 2004; Santini 2010). LEDs have long life approximately 10,000 hours (Oyama et al. 2004; Rachala and Yelampalli 2010). The main disadvantage of LED curing lights is that they could only be used for camphoroquinone-based composite resins, because these units have a

limited wavelength spectrum (Rueggeberg 2002; Oberholpez et al. 2005; Robertson et al. 2006; Garg and Garg 2010). However, it has been speculated that LED units will cure resin composites containing camphoroquinone photoinitiator more efficiently than QTH or PA units, because LED produce peak spectral output that is very close to the maximum absorption spectrum of 468 nm of camphoroquinone, when QTH and PA units have initial broadband emissions that have to be filtered (Mills et al. 1999; Burgess et al. 2002; Rueggeberg 2002 ; Nomoto et al. 2004).

Plasma Arc Unit

In the late 1990's, the plasma arc curing unit was introduced as a means of rapid light curing (Oesterle et al. 2001; Oyama et al. 2004; Garg and Garg 2010). The term 'plasma' refers to the gas which has most of its atoms ionized, in case of plasma arc curing light the gas used is xenon (Rueggeberg 1999; Oesterle et al. 2001; Santini 2010; Sherwood 2010). Light beam in plasma arc (PA) units is generated by passing a high voltage current across two tungsten electrodes within a xenon-filled fluorescent bulb (Rueggeberg 1999; Althoff and Hartung 2000; Oesterle et al. 2001; Burgess et al. 2002; Powers 2002; Oberholpez et al. 2005; Garg and Garg 2010; Santini 2010).When electric current passes, the gas between the electrodes becomes ionized and positively and negatively charged particles are created. As a result, the plasma is heated to several thousand degrees Celsius and produces ultraviolet radiation (Althoff and Hartung 2000). After the UV radiation collides with the wall of the bulb, it is converted to light and heat (Santini 2010). There are two filters through which the light passes: band pass filter and infrared filter. The infrared filter reduces the infrared spectra and the band pass filter narrows the spectra of white light to the blue light wavelength

(Rueggeberg 1999; Burgess et al. 2002; Robertson et al. 2006; Sherwood 2010). This process

results in the emission of light with intensity in the region from 1,800 Mw/cm ^{2} to 2,500

Mw/cm ^{2} and a wavelength of 470 nm (Rueggeberg 1999; Oesterle et al. 2001; Oberholpez et

al. 2005; Lynch 2008; Garg and Garg 2010). Despite the initial broadband emission, because

of the filtering process with some PA units, the final photoradiation produced by a plasma arc

curing unit can be a somewhat narrow wavelength spectrum, with little emission of light with

a wavelength that falls outside the activation range of camphoroquinone (Rasetto et al. 2001).

However, a concern with PA units is their low efficiency because only 1% of the energy is

given off as light and the remainder is converted to heat (Althoff and Hartung 2000). Besides

low efficiency, there are other disadvantages of PA curing units including: 1) high power

consumption, higher than QTH curing lights; 2) high operating temperatures that require a

cooling system; 3) bulky units that lack of portability due to a tabletop base housing the bulb;

4) wide-spectrum light that must be filtered; and 5) the units are expensive (Burgess et al.

2002; Garg and Garg 2010; Santini 2010). However, in terms of advantages, the expected life

of PAs is much greater than that of QTH curing units (Rueggeberg 1999). Moreover, plasma

arc units were intended to reduce curing times to as little as 3 sec (Danesh et al. 2004;

Nomoto et al. 2004; Robertson et al. 2006; Lynch 2008). Thus, plasma arc units cure

composite resin more quickly than any other curing light source (Burgess et al. 2002;

Nomoto et al. 2004).

Polymerization Process of Light-Cured Adhesives

Light-cured adhesives contain a two-component initiator system: a ketone and an

amine. The ketone, which is a photo-absorbing molecule, serves as an activator for

polymerization (photoinitiator) and the amine is an accelerator. In most composite resins, the photoinitiator is usually camphoroquinone (Yearn 1985; Althoff and Hartung 2000; Burgess et al. 2002; Robertson et al. 2006; Garg and Garg 2010). It absorbs energy at the wavelength peak at approximately 465 – 470 nm within the blue region of the visible light spectrum (Yearn 1985; Fan et al. 1987; Rueggeberg 1999; Althoff and Hartung 2000; Abate et al. 2001; Burgess et al. 2002; Lynch 2008; Sherwood 2010). It was reported that the effective range of light emission spectrum that can initiate polymerization process is relatively narrow (Santini 2010). When camphoroquinone is irradiated with a light of a specific wavelength and appropriate intensity for a required period of time, it becomes raised to the excited state and when camphoroquinone collides with the amine it results in formation of free-radical molecules. These free radicals initiate the polymerization process which results in monomer molecules joining together to form a polymer chain (Rueggeberg 1999; Althoff and Hartung 2000; Oesterle et al. 2001; Rasetto et al. 2001; Lynch 2008). Therefore, the greater the light intensity, the greater number of photons will reach the resin composite material and produce the greater number of excited camphoroquinone molecules, resulting in more free radicals. It was concluded that intensity of the light radiation is an essential component in terms of the rate and extent of the polymerization process (Rueggeberg 1999; Abate et al. 2001). Inappropriate wavelength and intensities of the light from a curing unit are associated with inadequate polymerization (Abate et al. 2001). When the light intensity is less than optimal, a proportional increase in curing time can be applied to achieve optimal polymerization and physical properties of the polymer (Sakaguchi et al. 1992; Miyazaki et al. 1996; Strydom 2002). However, if the light source is inadequate to activate the polymerization reaction, no

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compensatory mechanisms can produce an optimally cured resin composite (Sakaguchi et al. 1992). A minimum of 400 mW/cm^2 is recommended for routine polymerization of light-activated resin composites (Rueggeberg et al. 1994). Moreover, it was reported that other factors affecting polymerization include: composite filler type, size and loading; thickness and shade of the composite resin; effectiveness of light transmission through the light tip; light intensity; exposure time and distance of the light source from the composite resin (Lynch 2008; Santini 2010).

Total Energy Concept

Currently, there is a theory that the polymerization of composite resin is based on the concept of the total energy delivered to the material (Oesterle et al. 2001; Burgess et al. 2002). The reason for using total energy concept is that the light initiator used in a light-curable resin composite needs certain light energy to begin polymerization process. Thus, a fixed energy level produces certain number of free radicals and achieves the same degree of conversion (Emami et al. 2003). Moreover, a given total energy can be delivered with different combinations of light intensity and exposure duration (Miyazaki et al. 1996; Peutzfeldt and Asmussen 2005). The total energy is measured in mJ/cm^2 and can be calculated if the output of the photopolymerization source in mW/cm^2 and the duration of exposure in seconds are known (Emami et al. 2003). All light curing units have their energy output levels and each composite resin has required energy in order to polymerize, both are labeled on the products. Since some manufacturers use different initiators other than camphoroquinone in their composite resins, not all light sources will polymerize all composite resins (Rueggeberg 1999; Abate et al. 2001).

Curing Time and Total Energy

Inadequate curing times are associated with inadequate polymerization of composite resins including orthodontic adhesives that results in reduced orthodontic bracket bond strengths (Sargison et al. 1995; Abate et al. 2001). It is important for composite resin increment to be irradiated for an appropriate period of time, usually 20 to 30 seconds, which is the time required for the photoinitiator to be activated. If the curing time is reduced below the sufficient period, it tends to result in the early termination of polymerized chains – "short-chain termination." Short curing times increase polymerization stresses and reduce mechanical properties of the cured composite (Lynch 2008).

Higher total energy delivered to composite adhesive produces greater polymerization and degree of conversion resulting in improved mechanical properties. However, the kinetics of polymerization has been found to be very complex and a simple reciprocal relationship between light intensity and the exposure duration does not exist (Peutzfeldt and Asmussen 2005; Robertson et al. 2006; Feng et al. 2009). For a given total energy, longer exposure durations at low light intensity produced a more efficient polymerization than short curing times at high light intensity (Peutzfeldt and Asmussen 2005; Feng et al. 2009). The polymer network formed by high-intensity curing is greatly different (Rueggeberg 1999; Robertson et al. 2006; Sherwood 2010). For example, when using high-intensity curing units with shorter exposure times, an average polymer chain length is shorter, with lower molecular weight and less cross-linking. Since many physical properties depend on the molecular weight and the extent of polymer network cross-linking, there is potential for poorer physical properties with faster polymerization processes (Rueggeberg 1999; Millar and Nicholson 2001; Robertson et

al. 2006; Lynch 2008).When compared to conventional QTH units, plasma arc units resulted in more short-chain termination in polymerized composite resin (Lynch 2008). Typically, an exposure of 10 sec from a PA unit is equivalent to 40 sec of irradiation from QTH (Powers 2002). Furthermore, since the increase in intensity produces a stronger influence than reduction of time on the total energy level, high levels of intensity curing with shorter exposure periods produce more internal stress in the final polymer (Althoff and Hartung 2000; Lohbauer et al. 2005). However, composite resin polymerization shrinkage in orthodontic adhesives is not a concern due to the following factors: 1) the adhesive layer is very thin; 2) the excess of resin at the edges of the adhesive area absorbs some of the shrinkage; 3) since the bracket is a free floating object, the shrinkage will pull the bracket closer to enamel, which is most likely an advantage rather than a disadvantage (Oesterle et al. 2001; Klocke et al. 2002; Eliades 2006).

Degree of Conversion of Resin Composite Materials

To evaluate the efficiency of the polymerization process, the degree of conversion of the monomer can be measured. Degree of conversion is the measure of percentage of carbon-carbon double bonds that have been converted to single bonds to form a polymeric resin. Composite resins based on Bis-GMA generally are converted approximately 65% (Robertson et al. 2006; Sherwood 2010), but the degree of conversion typically can range from 55 – 75% (Watts 2001; Kauppi and Combe 2003). A conversion degree of 60% means that 60% of the methacrylate groups have been polymerized. With dimethacrylate monomers, this means that with the remaining 40% one of the two methacrylate groups on a monomer may be unreacted or both methacrylate groups could be unreacted (Robertson et al.

2006; Sherwood 2010). While the majority of unconverted monomer is usually unreacted at only one end of the bifunctional molecule to form the end of a polymer chain, any totally unreacted monomer will act as a plasticizing agent and result in a polymer network with less than ideal mechanical properties (Watts 2001) (Rueggeberg 2002; Sherwood 2010). Therefore, it is generally desirable to increase the degree of conversion in order to produce stiffer and more durable resins (Watts 2001; Bang et al. 2004; Sherwood 2010). Just as with polymerization, the degree of conversion is related to the intensity of the curing light and the duration of exposure (Nomoto 1997; Robertson et al. 2006).

Measurement of Degree of Conversion

Degree of conversion can be measured by various spectroscopic methods including Fourier transform infrared (FTIR) spectroscopy and Raman microspectroscopy (Pianelli et al. 1999; Soh et al. 2004). Raman microspectroscopy can be a powerful tool for characterization of polymers, especially for components that are present in a small concentration, only 5-10% of the sample. When the radiation from a monochromatic source (a laser) is focused on a sample microregion, the fraction of radiation scattered by the sample consists of a component of the exciting radiation known as the Rayleigh line and other weak lines called Raman lines. The frequency shifts of the Raman lines from the Rayleigh line correspond to the molecular vibrational frequencies within the sample molecules. The plot of the Raman frequencies as a function of their intensities yields the Raman spectrum, which provides important information on the structure, orientation, and chemical state of a sample (Eliades and Brantley 2001a).

Raman microspectroscopy offers several advantages over FTIR spectroscopy. The entire vibrational spectrum is probed with one instrument at increased sensitivity and at high spatial resolution (~1 µm) (Eliades and Brantley 2001a). Moreover, sampling procedure is easily performed and is non-destructive for most applications (Eliades and Brantley 2001a; Soh et al. 2004). Raman spectra of solids and crystals contain contributions from lattice vibrations at low frequencies that provide important information on crystal structure (Eliades and Brantley 2001a). In addition, when degree of conversion is measured using the Raman technique, performed without any mechanical or chemical pretreatment, which reduces the potential of influencing the results. Thus, Raman microspectroscopy may be a more convenient, accurate technique than FTIR for determining the degree of conversion (Pianelli et al. 1999; Soh et al. 2004; Gilchrist et al. 2007; Miletic and Santini 2008).

Curing Unit Effect on Orthodontic Adhesive Degree of Conversion

Two previous studies compared QTH and LED units (Carvalho et al. 2010; Cerveira et al. 2010), and one reported there was no significant difference in degree conversion of orthodontic adhesives (Cerveira et al. 2010), while the other indicated that LED was more efficient (Carvalho et al. 2010). Another study compared QTH and PA units and reported lower DC with the PA unit (Bang et al. 2004). Finally, only one study has compared all three types of curing units, QTH, LED and PA, and in that study there were significant differences reported between the units, but those differences varied depending on the curing times (Niepraschk et al. 2007).

After analysis of the current literature on degree of conversion of orthodontic composite resin adhesives as a function of type of curing unit, it was concluded that all

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present articles contain one or more of the following limitations. Most studies did not compare all three light-curing units. Because the concept of total energy was not used in the studies, the results obtained from different energy intensities and different curing times were difficult to interpret. Moreover, none of the studies that measured DC included bonding orthodontic brackets to human teeth and simulating clinical orthodontic bracket bonding is an important factor related to DC of the orthodontic adhesive resin.

Curing Unit Effect on Orthodontic Bracket Shear Bond Strength

Orthodontic bracket bond failure results in added treatment time and frustration for the clinician and patient. Thus, adequate orthodontic bracket bond strength is an important factor in reducing premature bracket debonding. It has been speculated that an adhesive-bracket system should be able to withstand a stress of at least 6-8 MPa (Reynolds 1975; Powers 2001). However, these values may underestimate the probability of bond breakage during mastication, a factor which may explain the disagreement between clinical failure rates and in vitro bond strength data (Eliades et al. 2004).

There have been numerous articles evaluating shear bracket bond strength, the most prevalent mode of in vitro testing (Bayne 2002). Shear bracket bond strength equals the force of shear load required to remove or debond the bracket divided by the area of the bonded interface (area of the bracket base) and is commonly reported in megapascals (MPa). Because the curing unit is an important factor in orthodontic adhesive bracket bond strength, numerous articles on the comparison of shear bond strength produced by different light-curing sources have been published. Some investigations comparing QTH and LED reported no significant difference in shear bond strength (Evans et al. 2002; Bishara et al. 2003;

Gronberg et al. 2006; Palomares et al. 2008; Di Nicolo et al. 2010; Retamoso et al. 2010), while others reported differences between QTH and LED (Usumez et al. 2004; Gronberg et al. 2006; Rachala and Yelampalli 2010). Other studies comparing shear bond strength with QTH and PA units reported no difference in shear bond strength (Oesterle et al. 2001; Pettemerides et al. 2001; Sfondrini et al. 2001; Signorelli et al. 2006; Toodehzaeim et al. 2012), and again other studies found QTH and PA to produce significantly different shear bond strength (Klocke et al. 2002; Sfondrini et al. 2002; Signorelli et al. 2006). One study that compared LED and PA curing units reported similar shear bond values (Dall'Igna et al. 2011) and finally, two studies that compared all three curing sources and reported no significant difference in shear bond strength (Thind et al. 2006; Ulusoy et al. 2008).

Similar to the evaluation of the literature related to the degree of conversion of orthodontic adhesives as a function of curing unit, there was an important limitation with the shear bond strength studies that compared curing units. Because the concept of total energy was not used, the shear bond strength results obtained using different energy intensities and different curing times were difficult to compare and interpret.

Correlation of Degree of Conversion and Shear Bond Strength

Numerous studies have evaluated degree of conversion of orthodontic adhesives as a function of curing unit and other studies have evaluated shear bond. However, to date, no study has correlated the degree of conversion and shear bond strength based on a comparison of curing units and associated exposure time taking into consideration the total energy. Such a comparison is valuable in terms of trying to explain any potential differences of bond strength that might occur with different units and curing times that translate into total energy.

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Problem Statement

The purpose of this study is to evaluate how QTH, LED, and PA light curing sources and associated exposure time (total energy) affect degree of conversion and shear bond strength of light-cured orthodontic adhesives, as well as to determine if there is a correlation between degree of conversion and shear bond strength.

Hypotheses

1. There will be a significant difference in bracket shear bond strength of light-cured orthodontic adhesives as a function of different light curing sources and total energy.

2. There will be a significant difference in degree conversion of light-cured orthodontic adhesives as a function of different light curing sources and total energy.

3. There will be a correlation between bracket shear bond strength and degree of conversion of light-cured orthodontic adhesives.

CHAPTER 2

MATERIALS AND METHODS

Specimen Preparation

Information regarding this project was submitted to the University of Missouri-Kansas City (UMKC) Adult Health Science Institutional Review Board (AHSIRB) and it was determined that the project did not qualify as human subject research (12-11 – NHSR).

Ninety previously extracted third maxillary molars were collected from the Oral Surgery Clinic at the UMKC School of Dentistry. No patient identifiers were associated with the teeth. Following extraction, the teeth were stored in 0.9% phosphate buffered saline (PBS) with 0.002% sodium azide included to prevent microbial growth. Teeth were inspected for an intact buccal surface with no evidence of carious lesions, demineralization, fluorosis, abfraction lesions, restorations or anomalous morphology. Specimen teeth were excluded if any enamel damage was present including enamel craze lines or trauma from extraction forceps. Each tooth was randomly assigned to one of the experimental groups.

Teeth were embedded in self-cured acrylic resin[1] with anatomical crown exposed up to, but not covering cemento-enamel junction (Figure 1). In a mounting ring[2] each tooth was oriented so that the least curved portion of the labial surface of tooth crown was perpendicular to the surface of acrylic resin. Acrylic resin was allowed to cure for at least one hour, and after that time the embedded specimens were removed from the mounting ring and

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[1] Biocryl #040-016, Great Lakes, 200 Cooper Ave., Tonawanda, NY 14150
[2] Item #20-8180, Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044

subjected to bracket bond testing.

Figure 1. Maxillary third molar embedded in self-cure acrylic resin.

Bracket Bonding

The orthodontic brackets[3] used in this research study were standard edgewise maxillary premolar steel brackets with 0.022-inch slot MBT prescription and concave bracket base. The premolar bracket was chosen for its universality: it could be used on right and left maxillary first and second premolars, and also, it adapts well to the mesio-labial surface of the right and left maxillary third molars.

The orthodontic resin composite adhesive[4] used in this study was composed of 70-80% silane-treated quartz, 10-20% BisGMA, 5-10% Bisphenol A Bis (2-hydroxyethyl ether) dimethacrylate, less than 2% silane-treated silica, and less than 0.2 % diphenyliodonium hexafluorophosphate.

Bracket bonding procedures were performed in an environmental chamber at 33ºC (+/- 2º) and 75% (+/- 5%) humidity simulating clinical conditions (Plasmans et al. 1994). Per manufacturer's instructions, the buccal surface of each tooth was etched with 34% phosphoric acid[5] for 15 sec, rinsed with distilled water for 15 sec and air dried using oil and moisture-free air source for 5 sec to insure the appearance of a frosty enamel surface. A uniform coat of primer[6] which had the following composition: Bisphenol A Diglycidyl Ether Demethacrylate 45-55%, Triethylene Glycol Dimethacrylate 45-55%, 4-(Dimethylamino)-Benzeneethanol < 0.5%, DL- Camphorquinone < 0.3%, Hydroquinone < 0.03% was applied

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[3] Victory Series/MBT, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016
[4] Transbond XT[TM], 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016
[5] Caulk Dentsply Inc., 221 W. Philadelphia Street. P.O. Box 872 York, PA 17405-0872
[6] Transbond XT Primer, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

to the etched tooth surface and then followed by a blast of air for 1s to thin the primer.

Orthodontic resin composite adhesive was placed to completely cover the mesh surface of the bracket base via pre-loaded syringe. After completion the syringe tip was wiped clean and the cap was replaced. Each bracket was placed at the mesial-buccal line angle of maxillary third molar with the vertical scribe line aligned perpendicular to the upper surface of the embedding resin. After the bracket was placed and aligned, it was compressed against the tooth surface until all excess adhesive was expressed and then it was removed with a scaler[7]. Within 60s of dispensing the orthodontic resin adhesive, a curing unit corresponding to the testing group was used to polymerize the resin adhesive for specified time period. During bonding procedure the light tip of each curing unit was held about 1 mm away from the bracket / tooth interface for one half of the proposed curing time from the distal and the mesial sides.

Measuring Light Intensity of the Curing Units

Before performing each bonding procedure, light intensity of the curing unit was checked with a radiometer[8] to confirm its attributed light intensity. These intensity measurements were used to calculate the total energy delivered to the adhesive resin under each bracket.

Experimental Design and Sample Size

With the exception of using different curing units and different curing times, all

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[7] CVHK ½, Hu-Friedy, 3232 N Rockwell St., Chicago, IL 60618
[8] OrthoLux LED radiometer, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

processes, materials, and equipment for bonding of all experimental groups were performed the same way as described above. The study utilized a two-factor design with independent variables: curing light source and total energy output. Curing light sources included: QTH[9], LED[10] and PA[11]. By varying the curing time, there were three levels of total energy used with each curing unit: 5k, 10k, and 15k mJ/cm^2. As already indicated, total energy was calculated based upon output light intensity multiplied by curing time. The dependent variables were bracket shear bond strength and degree of conversion of the orthodontic adhesive resin. Using this design, there were 9 experimental groups with a convenience sample of 10 teeth used with each group. The experimental design is presented in Table 1.

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[9] Spectrum 800 Quansten Halogen Light, Dentsply, 221 W. Philadelphia St., York, PA 17405
[10] OrthoLux LED, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016
[11] Sapphire Plasma Arc Light, Den-Mat Holdings LLC, Skyway Dr., Santa Maria CA 93455

TABLE 1

EXPERIMENTAL DESIGN

* Curing Unit and Power Output	Curing Time (sec)	*Total Energy (mJ/cm^2)	**Shear Bond Strength (MPa)	**Degree of Conversion (%)
PA ($\sim$ 2000 mW/cm^2)	3	5,000		
	6	10,000		
	9	15,000		
	Total			
LED($\sim$ 1600 mW/cm^2)	4	5,000		
	8	10,000		
	12	15,000		
	Total			
QTH($\sim$ 424 mW/cm^2)	12	5,000		
	24	10,000		
	36	15,000		
	Total			

* Independent variables

** Dependent variables

Instrumentation and Measurement

Shear Bond Testing

The bond strength testing was carried out with a universal testing machine[12] as soon as possible after bracket bonding. The testing was performed at ambient temperature and relative humidity conditions. The tooth embedded in acrylic was placed on the specimen holder and stabilized with four locking screws on the universal testing machine platform. Attached to the tester crosshead, the knife-edge stainless steel rod was oriented at the occlusal edge of the bonded bracket base in a way so the load was applied in the occlusogingival direction paralleling the buccal surface of the tooth (Figure 2). The brackets were sheared off the tooth using a crosshead speed of 1mm/min and the maximum load in Newtons (N) was recorded at debonding. Shear bond strength was calculated using the following equation: Shear Strength (MPa) = Debonding Force (N) / Bracket base surface area (mm^2), where Bracket base surface area (mm^2) = width of the bracket base (mm) * height of the bracket base (mm) = 3.55 *3.05 = 10.83 mm^2. A representative shear bond load-extension graph is presented in Figure 3.

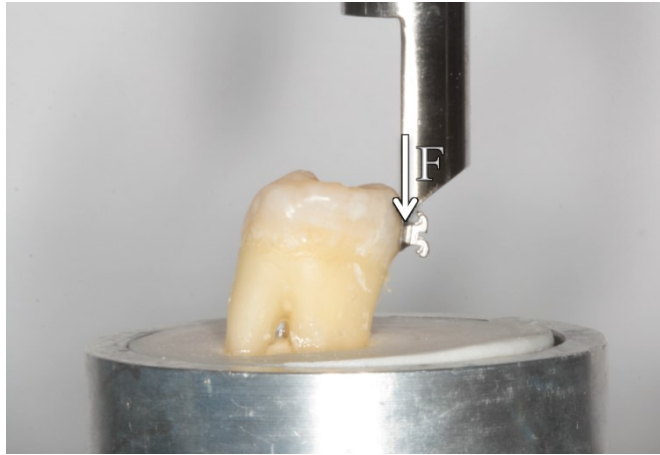[12] Model 1125/1150R, Instron Industrial Products, 100 Royal St., Canton MA 02021

Figure 2. Bonded, mounted tooth secured for shear bond strength testing. Shear load (F) was applied by the stainless steel rod of universal testing machine.
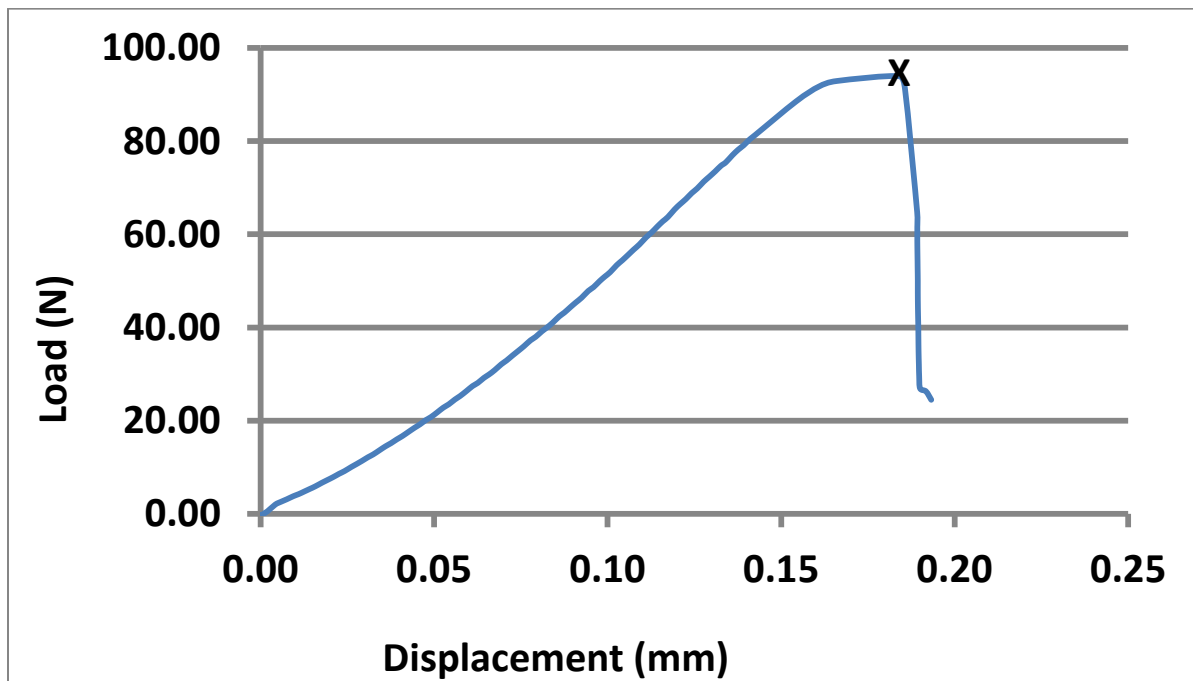

Figure 3. Representative of shear bond load-displacement graph. Maximum load (X) was used to calculate shear bond strength.

Degree of Conversion Measurements

Micro-Raman[13] spectra collection was performed immediately after shear bond

testing of each bracket and was completed within 50 min after initial light-cure bracket

bonding. This protocol for collecting micro-Raman spectra was developed in order to prevent

measuring any potential dark cure effects, which is polymerization of the adhesive resin that

can occur subsequent to light activation. Three point measurements were taken at three

separate locations on the residual resin adhesive on the bracket base, and if there was not

enough resin adhesive remaining on the bracket base, the three point measurements were

taken from residual adhesive on the tooth. The micro-Raman analysis was performed with a

red argon-ion laser at 632.8 nm with spectra collected in the region of 800 - 1800 cm^{-1}.

Spatial resolution was 1.5 μm and spectral resolution was 2.5 cm^{-1}. Prior to each series of

measurements of residual adhesive, the spectrometer was calibrated internally to zero and

then, using a silicon sample, calibrated for coefficient values. Spectral acquisition time was

30 s with two accumulations for a total of 60 s per site. The laser beam was focused through

a 50x objective lens. Spectral measurements of polymerized orthodontic resin adhesive were

made using the same instrumentation parameters.

"Band fitting" was completed with software[14] that allowed accurate calculation of

peaks and band positions, the elimination of extraneous peaks, and the subsequent calculation

of peak amplitude, band width, and integrated areas. The auto fit function of the software was

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[13] HR800, Horiba Jobin, Yvon, 231 Rue deVille, Villeneuve, France 59650
[14] GRAMS/AI v7.02, Galactic Industries Corp., 395 Main St., Salam, NH 03079

applied to establish a baseline for the entire range of collected spectra. Peaks were measured

at 1640 cm^{-1} and 1610 cm^{-1} to calculate the degree of conversion using the following

equation: DC(%) = 100*[1-($R_{polymerized}$ / $R_{unpolymerized}$)], where R = band height at 1640 cm^{-1}

/band height at 1610 cm^{-1} (Pianelli et al. 1999). Figure 4 illustrates an example of a micro-

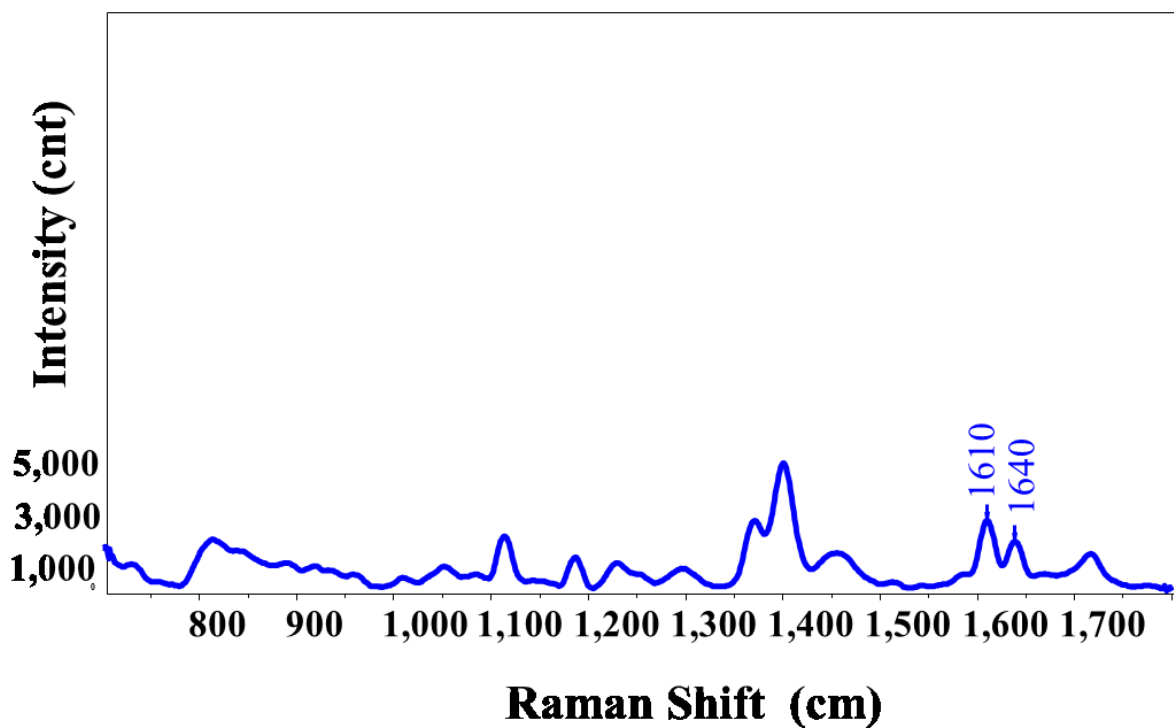Raman spectrum collected for one of the specimens.

Figure 4. Representative micro-Raman spectrum. Identified peaks at 1640 cm^{-1} and
1610cm^{-1} were used to calculate the degree of conversion.

Data Analysis

Collected data was analyzed using statistical software[15]. The design of the study was a two-factor (curing unit and total energy), randomized design with measurement of the dependent variables (bracket shear bond strength, degree of conversion) at the interval/ratio level. A multivariate analysis of variance (MANOVA), $\alpha = 0.05$, was utilized. Tukey's post-hoc comparison tests were used to determine where differences exist. To determine any association between degree of conversion and bracket bond strength, a Pearson correlation was also used.

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[15] SPSS version 16, 233 S. Wacker Dr., Chicago IL 60606

CHAPTER 3

RESULTS

Shear Bond Strength Measurements

Ninety maxillary third molars were randomly assigned to one of the nine experimental groups. Bonding and shear bond strength testing was performed according to the protocol described in Chapter 2. Means and standard deviations (SD) of shear bond strength for 9 testing groups are presented in Figure 5.

Based on a 2-factor multivariate analysis of variance (MANOVA), there were no significant differences ($p < 0.05$) identified between overall shear bond strength when brackets were bonded with different type of curing units. However, there were significant differences ($p \leq 0.05$) in bracket bond strength as a function of total energy across curing units. Based on the Tukey's post-hoc analysis, as total energy increased, there was a significant increase in bond strength across all curing units. Based on these results, hypothesis one was partially supported.

Degree of Conversion Measurements

Degree of conversion measurements were performed according to the protocol described in Chapter 2. Means and standard deviations (SD) of degree of conversion for 9 testing groups are presented in Figure 6. Based on the 2-factor MANOVA, there was a significant difference ($p < 0.05$) in overall degree of conversion as a function of curing unit and total energy. The Tukey's post-hoc analysis indicated that overall DC produced by PA was significantly higher than LED and QTH, which were not significantly different from each other. Post-hoc comparisons of total energy indicated that DC was significantly lower at

5k mJ/cm^2 than 10 and 15 k mJ/cm^2, which were not different from one another. Based on these results, hypothesis two was supported.

Correlation Between Shear Bond Strength and Degree of Conversion

Based on Pearson correlation analysis, the overall correlation between shear bond strength and degree of conversion across curing units was 0.437. Specifically for each unit, there was moderate positive correlation between shear bond strength and degree conversion, respectively 0.561 and 0.518 for the LED and QTH units, while there was a weak positive correlation, 0.372, for the PA unit. Collectively, these results substantiate hypothesis three.
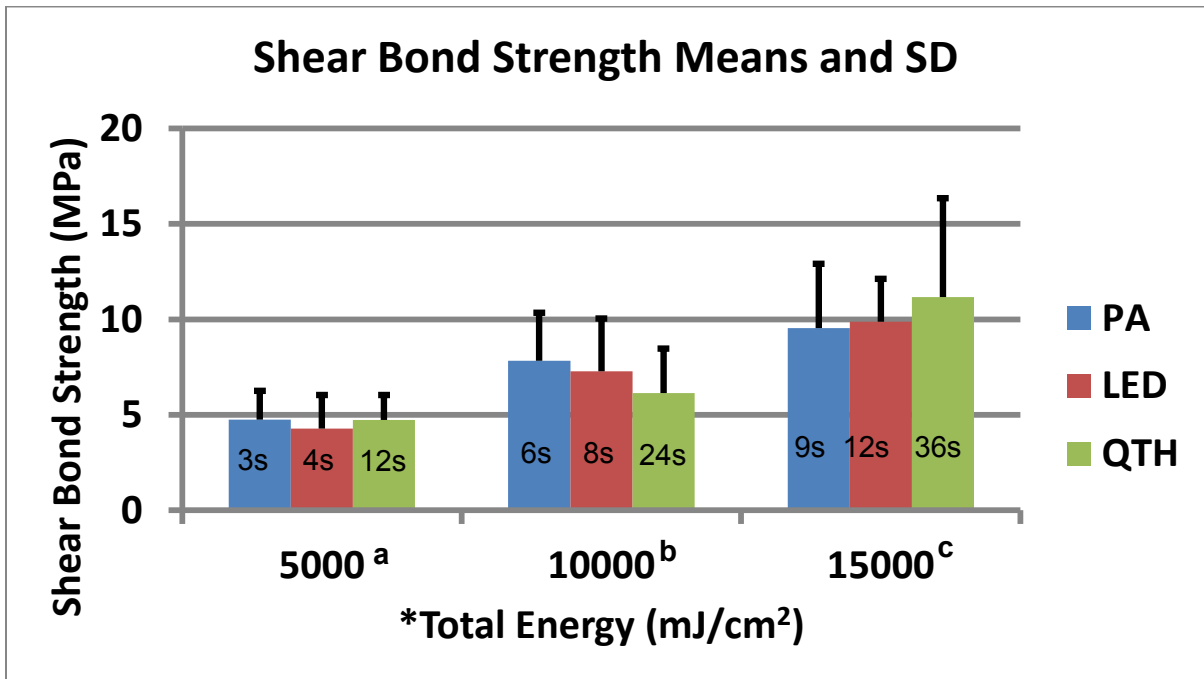
Figure 5. Mean and SD shear bond bracket strength values. N=10/type of unit and energy level.

*There was no significant difference between curing units at each energy level.

**Across curing units, there was a significant increase in shear bond strength with increasing total energy. Subsets are indicated by a, b, or c.
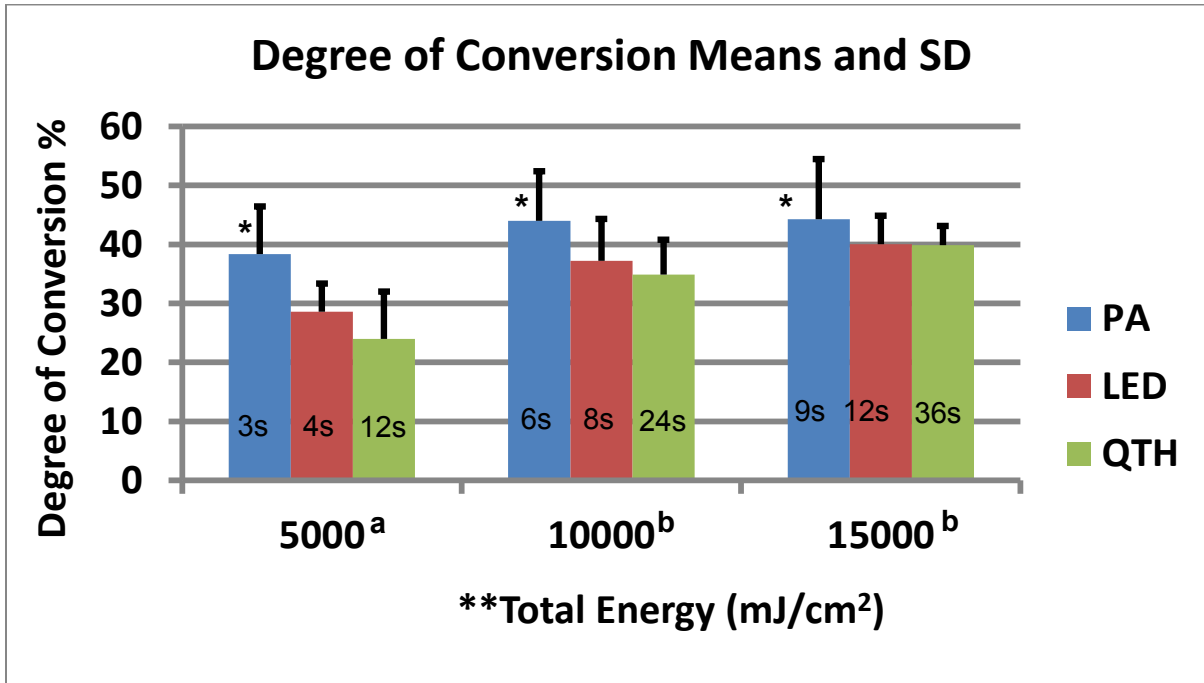
Figure 6. Mean and SD degree of conversion values. N= 10/unit type and energy level.

*Across energies, there was a significant effect of curing unit with PA demonstrating significantly higher degree of conversion than LED or QTH.

**Across units, there was also a significant effect of total energy on degree of conversion with a significant increase between 5,000 to 10,000 mJ/cm^2 and no further significant change at 15,000 mJ/cm^2. Subsets are indicated by a or b.

CHAPTER 4

DISCUSSION

One of the most widely used orthodontic adhesives is light-cured resin composite and therefore, there is a need for an efficient light-curing unit to perform orthodontic bonding. Currently, there are a variety of types of curing units available on the market and among them there are: quartz tungsten halogen (QTH), light-emitting diode (LED), and plasma arc (PA) curing units. Quartz tungsten halogen curing lights traditionally have been the most widely used by dental practitioners (Nomoto 1997; Rueggeberg 1999; Powers 2002; Nomoto et al. 2004; Yazici et al. 2007; Garg and Garg 2010; Rachala and Yelampalli 2010). LEDs are gaining popularity over QTH units owing to greater efficiency, lightweight, shock resistance (Mills et al. 1999; Burgess et al. 2002; Powers 2002; Oyama et al. 2004; Yazici et al. 2007; Lynch 2008; Rachala and Yelampalli 2010; Sherwood 2010). Plasma arc units allowed shorter curing times than any other curing sources (Burgess et al. 2002; Nomoto et al. 2004). There are several ways to evaluate an efficiency of a light-curing unit and some of them are: shear bond strength, degree of conversion and curing times. While previous studies compared shear bond strength (Oesterle et al. 2001; Pettemerides et al. 2001; Sfondrini et al. 2001; Evans et al. 2002; Klocke et al. 2002; Sfondrini et al. 2002; Bishara et al. 2003; Usumez et al. 2004; Gronberg et al. 2006; Signorelli et al. 2006; Palomares et al. 2008; Di Nicolo et al. 2010; Rachala and Yelampalli 2010; Retamoso et al. 2010; Dall'Igna et al. 2011) (Thind et al. 2006; Ulusoy et al. 2008) or degree of conversion (Bang et al. 2004; Niepraschk et al. 2007; Carvalho et al. 2010; Cerveira et al. 2010) based on curing times, this

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study compared shear bond strength and degree of conversion produced by QTH, LED, and PA based on the concept of total energy (Oesterle et al. 2001; Burgess et al. 2002). Total energy concept simplified the process of evaluation and comparison between the curing units. Moreover, this study was first to investigate the correlation between shear bond strength and degree of conversion of resin composite in orthodontic bonding.

Shear Bond Strength Testing

Based on results of this study, there were no significant differences identified in orthodontic shear bond strength between the groups bonded with PA, LED or QTH curing units at each of the total energy levels. This finding demonstrated that when equal total energy was delivered to resin composite adhesive, there was no difference in bond strength when different types of curing units with varying light intensities were used. The outcomes of this project agree with one earlier study, which compared the same three curing units and found no significant difference in SBS using a protocol similar to the current study (Thind et al. 2006). However, in another study comparing the three types of curing by Ulusoy et al. (2008), the results differed from ours in that they reported significantly higher SBS with QTH as compared to LED. These differences could be related to the fact that the study did not account for total energy differences across units and also did not test multiple levels of energy per unit.

In this study, although there were no differences between curing units at each total energy level, there was a significant difference in bond strength across units as total energy increased from 5k to 10k and from 10k to 15k. This trend is supported in a study by

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Youshida et al. (2012), which stated that an increase of SBS was associated with increase in exposure time. Collectively, based on these findings it could be speculated that by increasing curing time, shear bond strength would increase regardless of the type of curing unit used given that total energy delivered to orthodontic adhesive is matched for QTH, LED and PA units.

Degree of Conversion Measurements

Based on the results of this study, across all energy levels, PA produced significantly higher DC than LED or QTH. While our results suggest that PA is more efficient than LED or QTH in polymerizing orthodontic adhesive, these results are not in agreement with some previous studies. In terms of PA and LED comparison studies, Niepraschk et al. (2007) reported no significant difference in DC when the same curing times were used for PA and LED units, therefore the difference between the studies could be attributed to the fact the total energies of PA and LED were not matched.

Bang et al. (2004) compared PA and QTH units and reported significantly higher degree of conversion produced by QTH than by PA. In that study Bang and colleagues used the concept of total energy to identify curing times for PA and QTH units; however, to evaluate degree of conversion, orthodontic adhesive was cured through a glass slide without brackets.

In this study, the degree of conversion produced by LED and QTH were not statistically different which agreed with a study by Cerveira et al. (2010), where specimen disks, not human teeth were used to test DC. It also was supported in the study by

Niepraschk et al. (2007), which found similar DC produced by QTH and LED; however, total energy was not matched between the testing groups in this study. Opposite findings were reported by Calvalho et al. (2010) where QTH and LED units were compared using bovine teeth as specimens and found LED light more efficient in polymerization of orthodontic resin composite adhesive.

When three groups of total energy were compared, there were significant differences in DC found between 5k and 10k groups and 5k and 15k, and no significant differences in DC were discovered between 10k and 15k total energy groups. Only one study by Bang et al. (2004) calibrated total energy when evaluating DC: 4k and 8k where PA and QTH units were compared. It was reported no significant difference in DC between 4k and 8k groups were found, which contradicts the present study where DC in 5k groups was found significantly lower than in 10k group.

Based on the findings of this study, it could be speculated that degree of conversion of orthodontic composite resin adhesive increases significantly with increased total energy applied up to a limit, and after that limit an increase in total energy would not produce an increase in degree of conversion in the orthodontic adhesive.

Correlation Between Shear Bond Strength and Degree of Conversion

Overall correlation between shear bond strength and degree of conversion for types of curing units was moderate (0.437). Previous papers stated that by increasing degree of conversion of resin composite, the mechanical properties of the material would improve (Watts 2001; Bang et al. 2004; Sherwood 2010). However, to date, no previous

investigations evaluated the relationship between degree of conversion and shear bond strength in resin composite used for orthodontic bracket bonding. The results of the current study suggest that there is a moderate positive correlation in degree of conversion and shear bond strength when QTH, LED and PA units were evaluated together.

However, when each unit was examined separately, the correlation between DC and SBS varied from unit to unit. The strongest correlation between DC and SBS was found in LED group (0.561), which had curing intensity of approximately 1600 mW/cm^2. QTH group had comparable correlation to the LED group, which was 0.518 with light intensity of about 424 mW/cm^2. The lowest correlation between DC and SBS was found in PA group (0.372) with curing light intensity of approximately 2000 mW/cm^2.

As stated previously, this study found that PA unit produced higher degree of conversion than QTH or LED within each energy level; however, there were no significant differences identified in shear bond strength between the three units within each energy level. Moreover, PA group demonstrated the lowest correlation between DC and SBS. Therefore, it could be concluded that the degree of conversion increases significantly with curing light intensity of about 2000 mW/cm^2; nevertheless, the shear bond strength remains comparable to about 424 mW/cm^2 when equal total energy is applied to composite adhesive.

LED and QTH curing units had comparable correlation between DC and SBS of 0.561 and 0.518 accordingly. Moreover, with light intensity in LED of 1600 mW/cm^2 and of 424 mW/cm^2 in OTH, the two units had no significant differences in produced degree of conversion or shear bond strength.

As reported in the previous studies, the higher intensity curing lights produce the final polymer with more internal stress and potentially poorer physical properties of the composite due to faster polymerization process (Rueggeberg 1999; Millar and Nicholson 2001; Robertson et al. 2006; Lynch 2008). Nonetheless, it was found in this study the speed of polymerization process did not appear to negatively affect shear bond strength of orthodontic resin composite adhesive. However, this is likely related to differences in the configuration factor (C-factor), ratio of bonded to unbounded surfaces. As the C-factor increases, there is an associated increase in residual stress development in the polymerized composite. (Feilzer et al. 1987). The C-factor associated with bonding a bracket to the external surface of a tooth would be lower as compared to bonding a restorative composite into an intra-coronal preparation.

Study Limitations

In this study, while three types of curing sources were evaluated (QTH, LED and PA) potential differences between the same types of units from different manufacturers were not assessed. Due to the limitations of collecting adequate numbers of extracted teeth, maxillary third molars were used in this research project rather than maxillary premolars. Since it is known that maxillary third molars have more variable anatomy between each other than maxillary premolars, care was taken to select only well-formed teeth for bonding and testing. Furthermore, shear bond testing was performed with crosshead speed at 1mm/min in a room environment, which is very different from oral cavity conditions, where clinically brackets are sheared off at a much faster speed. The speed of the crosshead was chosen to match the

shearing speed used most frequently in the literature, thus allowing better comparison to SBS results of previous studies.

It is also interesting to note that when compared to some previous investigations, this study demonstrated lower mean shear bond strength values across all experimental groups. In this study, means ranged from 4.27 to 11.18 MPa, while across other studies, overall means ranged from 10.35 to 14.06 MPa (Palomares et al. 2008; Retamoso et al. 2010). The current lower SBS values could possibly be explained by fact that SBS testing was performed immediately after the bonding procedure of each specimen, whereas in the majority of the studies, SBS testing was performed 24-hour post initial bonding procedure. Since a part of this study was to evaluate if there was any correlation between shear bond strength and degree of conversion, the testing was performed as soon as possible to minimize any dark cure effects on bond strength and degree of conversion measurements.

Clinical Significance

The main focus of this study was to compare PA, LED and QTH curing units in terms of produced shear bond strength and degree of conversion, and to identify if there was a correlation between those properties. The concept of total energy was used to simplify the process and make assessment more straightforward in comparison to the existing research studies. Furthermore, this study was the first to evaluate if there is a correlation between degree of conversion and shear bond strength in orthodontic composite adhesive produced by PA, LED and QTH light curing sources.

In terms of shear bond strength, the results of this study suggest there are no differences between PA, LED or QTH curing units as long as they are activated for appropriate curing times to deliver approximately the same total energy to the adhesive. Moreover, it was found that an increase in curing time produced higher shear bond strength for the three curing units: PA from 4.74 MPa in 3 sec group to 9.54 MPa in 9 sec group, LED from 4.27 MPa in 4 sec group to 9.88 MPa in 12 sec group, QTH from 4.74 MPa in 12 sec group to 11.18 MPa in 36 sec group. Except for the shortest curing times for all units, the SBS values in this study are within limits or above often cited in the literature minimal clinical bond strength values of 6-8 MPa suggested by Reynolds (1975). However, these suggested values are not evidence based, and merely are a speculation.

The results of this study do not support the existing findings that light curing units with high light intensities worsen physical properties of polymerized resin composite when compared with traditional curing sources with much lesser light intensity. As already noted, to produce maximum SBS, curing times were 9, 12, and 36 sec for PA, LED, and QTH, respectively. Thus, QTH requires approximately three to four times longer than PA and LED units to produce similar SBS. Although more expensive, PA and LED units are much more efficient for orthodontic bracket bonding and allow a clinician to perform bonding in less time without affecting bond strength. The most efficient and also the most expensive curing unit was PA, and the least efficient and the least expensive was QTH.

Future Investigations

Curing units offered by many manufacturers are available to an orthodontic clinician today. It might be beneficial to compare curing efficiency of the same type of unit made by different manufacturers using the total energy concept. For an orthodontist, it would be advantageous to know if curing units of the same type from different companies have similar efficiency in orthodontic bonding or not.

Usually manufacturers of curing units suggest curing times for orthodontic bonding procedures. It would be interesting to find out how these curing times affect shear bond strength and degree of conversion using the total energy concept.

Due to the current widespread use of self-etching primers during orthodontic bonding, it could also be beneficial to include an experimental group with self-etching primer in a future study. That would allow the comparison of efficiency of curing units when traditional bonding protocol and newer technique including self-etching primer are utilized.

CHAPTER 5

CONCLUSIONS

1. There were no significant differences in shear bond strength between PA, LED and QTH curing units when equal total energy was delivered to orthodontic adhesive. However, there was a significant increase in shear bond strength as total energy increased across all curing units.

2. There was significantly higher degree of conversion produced by PA than by LED or QTH units at each total energy level. Furthermore, there was a significant increase in degree of conversion when total energy increased from 5k to 10k across all curing units.

3. There was a positive correlation between degree of conversion and shear bond strength: the strongest with the LED and QTH group (0.561 and 0.518, respectively), while a weaker correlation was demonstrated in the PA group (0.372).

LITERATURE CITED

Abate PF, Zahra VN, Macchi RL. Effect of photopolymerization variables on composite hardness. J Prosthet Dent 2001;86:632-5.

Althoff O, Hartung M. Advances in light curing. Am J Dent 2000;13:77D-81D.

Arrais CA, Rueggeberg FA, Waller JL, de Goes MF, Giannini M. Effect of curing mode on the polymerization characteristics of dual-cured resin cement systems. J Dent 2008;36:418-26.

Bang HC, Lim BS, Yoon TH, Lee YK, Kim CW. Effect of plasma arc curing on polymerization shrinkage of orthodontic adhesive resins. J Oral Rehabil 2004;31:803-10.

Bayne S. Bonding to dental substrates. In: Restorative dental materials. E Forest editor. 11 ed. St. Louis, USA: Mosby. A Harcourt Health Sciences Company; 2002, pp. 259-86.

Bishara SE, Ajlouni R, Oonsombat C. Evaluation of a new curing light on the shear bond strength of orthodontic brackets. Angle Orthod 2003;73:431-5.

Burgess JO, Walker RS, Porche CJ, Rappold AJ. Light curing--an update. Compend Contin Educ Dent 2002;23:889-92, 94, 96 passim; quiz 908.

Cacciafesta V, Sfondrini MF, Scribante A. Plasma arc versus halogen light-curing of adhesive-precoated orthodontic brackets: A 12-month clinical study of bond failures. Am J Orthod Dentofacial Orthop 2004;126:194-9.

Carvalho FA, Almeida RC, Almeida MA, Cevidanes LH, Leite MC. Efficiency of light-emitting diode and halogen units in reducing residual monomers. Am J Orthod Dentofacial Orthop 2010;138:617-22.

Cerveira GP, Berthold TB, Souto AA, Spohr AM, Marchioro EM. Degree of conversion and hardness of an orthodontic resin cured with a light-emitting diode and a quartz-tungsten-halogen light. Eur J Orthod 2010;32:83-6.

Cunningham SJ, Jones SP, Hodges SJ, Horrocks EN, Hunt NP, Moseley HC, Noar JH. Advances in orthodontics. Prim Dent Care 2002;9:5-8.

Dall'Igna CM, Marchioro EM, Spohr AM, Mota EG. Effect of curing time on the bond strength of a bracket-bonding system cured with a light-emitting diode or plasma arc light. Eur J Orthod 2011;33:55-9.

Danesh G, Davids H, Reinhardt KJ, Ott K, Schafer E. Polymerisation characteristics of resin composites polymerised with different curing units. J Dent 2004;32:479-88.

Di Nicolo R, Araujo MA, Alves LA, Assuncao e Souza RO, Rocha DM. Shear bond strength of orthodontic brackets bonded using halogen light and light-emitting diode at different debond times. Braz Oral Res 2010;24:64-9.

Eliades G, Brantley WA. Instrumental techniques for study of orthodontic materials. In: Orthodontic materials. Scientific and clinical aspects New York, USA: Thieme Stuttgart; 2001a, pp. 49-76.

Eliades T, Katsavrias E, Zinelis S, Eliades G. Effect of loading rate on bond strength. J Orofac Orthop 2004;65:336-42.

Eliades T. Orthodontic materials research and applications: Part 1. Current status and projected future developments in bonding and adhesives. Am J Orthod Dentofacial Orthop 2006;130:445-51.

Eliades T, Eliades G. Orthodontic adhesive resins. In: Orthodontic materials. Scientific and clinical aspects. New York, USA: Thieme Stuttgart; 2001b, pp. 201-20.

Emami N, Soderholm KJ, Berglund LA. Effect of light power density variations on bulk curing properties of dental composites. J Dent 2003;31:189-96.

Evans LJ, Peters C, Flickinger C, Taloumis L, Dunn W. A comparison of shear bond strengths of orthodontic brackets using various light sources, light guides, and cure times. Am J Orthod Dentofacial Orthop 2002;121:510-5.

Fan PL, Wozniak WT, Reyes WD, Stanford JW. Irradiance of visible light-curing units and voltage variation effects. J Am Dent Assoc 1987;115:442-5.

Feilzer AJ, De Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. Journal of Dental Research 1987;66:1636-9.

Feng L, Carvalho R, Suh BI. Insufficient cure under the condition of high irradiance and short irradiation time. Dent Mater 2009;25:283-9.

Foster TD. Orthodontic bonding. In: Textbook of orthodonticsOxford, Great Britain: Blackwell Scientific Pulblications; 1990, pp. 260-74.

Garg N, Garg A. Composite restorations. In: Textbook of operative dentistry. N Garg and A Garg editors New Dehli, India: Jaypee Brothers Medical Publishers Ltd; 2010, pp. 255-80.

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Gilchrist F, Santini A, Harley K, Deery C. The use of micro-raman spectroscopy to differentiate between sound and eroded primary enamel. Int J Paediatr Dent 2007;17:274-80.

Gronberg K, Rossouw PE, Miller BH, Buschang P. Distance and time effect on shear bond strength of brackets cured with a second-generation light-emitting diode unit. Angle Orthod 2006;76:682-8.

Kauppi MR, Combe EC. Polymerization of orthodontic adhesives using modern high-intensity visible curing lights. Am J Orthod Dentofacial Orthop 2003;124:316-22.

Klocke A, Korbmacher HM, Huck LG, Kahl-Nieke B. Plasma arc curing lights for orthodontic bonding. Am J Orthod Dentofacial Orthop 2002;122:643-8.

Lohbauer U, Rahiotis C, Kramer N, Petschelt A, Eliades G. The effect of different light-curing units on fatigue behavior and degree of conversion of a resin composite. Dent Mater 2005;21:608-15.

Lynch CD. Shedding light on placement techniques for posterior composites. In: Successful posterior composites. NHF Wilson editorLondon, GB: Quintessence Publishing Co. Ltd.; 2008, pp. 63-80.

Miletic VJ, Santini A. Remaining unreacted methacrylate groups in resin-based composite with respect to sample preparation and storing conditions using micro-raman spectroscopy. J Biomed Mater Res B Appl Biomater 2008;87:468-74.

Millar BJ, Nicholson JW. Effect of curing with a plasma light on the properties of polymerizable dental restorative materials. J Oral Rehabil 2001;28:549-52.

Millett DT, Glenny AM, Mattick CR, Hickman J, Mandall NA. Adhesives for fixed orthodontic bands. Cochrane Database Syst Rev 2007:CD004485.

Mills RW, Jandt KD, Ashworth SH. Dental composite depth of cure with halogen and blue light emitting diode technology. Br Dent J 1999;186:388-91.

Mills RW, Uhl A, Blackwell GB, Jandt KD. High power light emitting diode (led) arrays versus halogen light polymerization of oral biomaterials: Barcol hardness, compressive strength and radiometric properties. Biomaterials 2002;23:2955-63.

Mitchell L. Orthodontic bonding adhesives. Br J Orthod 1994;21:79-82.

Miyazaki M, Oshida Y, Moore BK, Onose H. Effect of light exposure on fracture toughness and flexural strength of light-cured composites. Dent Mater 1996;12:328-32.

Newman GV, Snyder WH, Wilson CE, Jr. Acrylic adhesives for bonding attachments to tooth surfaces. Angle Orthod 1968;38:12-8.

Niepraschk M, Rahiotis C, Bradley TG, Eliades T, Eliades G. Effect of various curing lights on the degree of cure of orthodontic adhesives. Am J Orthod Dentofacial Orthop 2007;132:382-4.

Nomoto R. Effect of light wavelength on polymerization of light-cured resins. Dent Mater J 1997;16:60-73.

Nomoto R, McCabe JF, Hirano S. Comparison of halogen, plasma and led curing units. Oper Dent 2004;29:287-94.

Oberholpez G, Botha CT, du Preez IC. Advances in light curing units and curing techniques: A literature review. SADJ 2005;60:451-4.

Oesterle LJ, Newman SM, Shellhart WC. Rapid curing of bonding composite with a xenon plasma arc light. Am J Orthod Dentofacial Orthop 2001;119:610-6.

Oyama N, Komori A, Nakahara R. Evaluation of light curing units used for polymerization of orthodontic bonding agents. Angle Orthod 2004;74:810-5.

Palomares NB, Cal-Neto JP, Sampaio-Filho H, Almeida MA, Miguel JA. Effect of high-intensity led units at reduced curing time on in vitro bond strength of orthodontic brackets. World J Orthod 2008;9:203-8.

Pettemerides AP, Ireland AJ, Sherriff M. An ex vivo investigation into the use of a plasma arc lamp when using a visible light-cured composite and a resin-modified glass poly(alkenoate) cement in orthodontic bonding. J Orthod 2001;28:237-44.

Peutzfeldt A, Asmussen E. Resin composite properties and energy density of light cure. J Dent Res 2005;84:659-62.

Pianelli C, Devaux J, Bebelman S, Leloup G. The micro-raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins. J Biomed Mater Res 1999;48:675-81.

Plasmans PJ, Creugers NH, Hermsen RJ, Vrijhoef MM. Intraoral humidity during operative procedures. J Dent 1994;22:89-91.

Powers JM. Composite restorative materials. In: Restorative dental materials. E Forest editor. 11 ed. St. Louis, USA: Mosby. A Harcourt Health Sciences Company; 2002, pp. 231-58.

Powers JM, Sakaguchi RL. Resin composite restorative materials. In: Restorative dental materials. P Rudolph editor. 12 ed. St. Louis: Mosby; 2006, pp. 189-212.

Powers JMaM, M.L. Enamel etching and bond strength. In: Orthodontic materials. Scientific and clinical aspectsNew York, USA: Thieme Stuttgart; 2001, pp. 105-22.

Proffit WRaF, H.W. and Sarver, D.M. Contemporary orhodontic appliances. In: Contemporary orthodontics. J Dolan editor. 4 ed. St. Louis, USA: Mosby Elsevier; 2007, pp. 395-430.

Rachala MR, Yelampalli MR. Comparison of shear bond strength of orthodontic brackets bonded with light emitting diode (led). Int J Orthod Milwaukee 2010;21:31-5.

Rasetto FH, Driscoll CF, von Fraunhofer JA. Effect of light source and time on the polymerization of resin cement through ceramic veneers. J Prosthodont 2001;10:133-9.

Read MJ. The bonding of orthodontic attachments using a visible light cured adhesive. Br J Orthod 1984;11:16-20.

Retamoso LB, Onofre NM, Hann L, Marchioro EM. Effect of light-curing units in shear bond strength of metallic brackets: An in vitro study. J Appl Oral Sci 2010;18:68-74.

Reynolds IR. A review of direct orthodontic bonding. British journal of orthodontics 1975;2:171.

Robertson TM, Heymann HO, Swift TJJr. Biomaterials. In: Art and science of operative dentistry. Robertson TM editor. 4 ed. St. Louis, USA: Mosby Elsevier; 2006, pp. 135-242.

Rueggeberg F. Contemporary issues in photocuring. Compend Contin Educ Dent Suppl 1999:S4-15; quiz S73.

Rueggeberg FA, Caughman WF, Curtis JW, Jr. Effect of light intensity and exposure duration on cure of resin composite. Oper Dent 1994;19:26-32.

Rueggeberg FA. From vulcanite to vinyl, a history of resins in restorative dentistry. J Prosthet Dent 2002;87:364-79.

Sakaguchi RL, Douglas WH, Peters MC. Curing light performance and polymerization of composite restorative materials. J Dent 1992;20:183-8.

Santini A. Current status of visible light activation units and the curing of light-activated resin-based composite materials. Dent Update 2010;37:214-6, 8-20, 23-7.

Sargison AE, McCabe JF, Gordon PH. An ex vivo study of self-, light-, and dual-cured composites for orthodontic bonding. Br J Orthod 1995;22:319-23.

Sfondrini MF, Cacciafesta V, Pistorio A, Sfondrini G. Effects of conventional and high-intensity light-curing on enamel shear bond strength of composite resin and resin-modified glass-ionomer. Am J Orthod Dentofacial Orthop 2001;119:30-5.

Sfondrini MF, Cacciafesta V, Klersy C. Halogen versus high-intensity light-curing of uncoated and pre-coated brackets: A shear bond strength study. J Orthod 2002;29:45-50.

Sherwood IA. Resin composite restorative material. In: Essentials of operative dentistry. 1 ed. New Delhi, India: Jaypee Brothers Medical Publishers Ltd; 2010, pp. 327-41.

Signorelli MD, Kao E, Ngan PW, Gladwin MA. Comparison of bond strength between orthodontic brackets bonded with halogen and plasma arc curing lights: An in-vitro and in-vivo study. Am J Orthod Dentofacial Orthop 2006;129:277-82.

Smith RT, Shivapuja PK. The evaluation of dual cement resins in orthodontic bonding. Am J Orthod Dentofacial Orthop 1993;103:448-51.

Soh MS, Yap AU, Yu T, Shen ZX. Analysis of the degree of conversion of led and halogen lights using micro-raman spectroscopy. Oper Dent 2004;29:571-7.

Strydom C. Curing lights--the effects of clinical factors on intensity and polymerisation. SADJ 2002;57:181-6.

Sunna S, Rock WP. Clinical performance of orthodontic brackets and adhesive systems: A randomized clinical trial. Br J Orthod 1998;25:283-7.

Sunna S, Rock WP. An ex vivo investigation into the bond strength of orthodontic brackets and adhesive systems. Br J Orthod 1999;26:47-50.

Tanoue N, Koishi Y, Atsuta M, Matsumura H. Properties of dual-curable luting composites polymerized with single and dual curing modes. J Oral Rehabil 2003;30:1015-21.

Thind BS, Stirrups DR, Lloyd CH. A comparison of tungsten-quartz-halogen, plasma arc and light-emitting diode light sources for the polymerization of an orthodontic adhesive. Eur J Orthod 2006;28:78-82.

Toodehzaeim MH, Kazemi AD, Aghili HA, Barzegar K, Fallahtafti T. Comparison of shear bond strength of orthodontic brackets bonded with halogen and plasma arc light curing. Dent Res J (Isfahan) 2012;9:321-7.

Ulusoy C, Irmak O, Bagis YH, Ulusoy OI. Temperature rise and shear bond strength of bondable buccal tubes bonded by various light sources. Eur J Orthod 2008;30:413-7.

Usumez S, Buyukyilmaz T, Karaman AI. Effect of light-emitting diode on bond strength of orthodontic brackets. Angle Orthod 2004;74:259-63.

Watts DC. Orthodontic adhesive resins and composites: Principle of adhesion. In: Orthodontic materials. Scientific and clinical aspectsNew York, USA: Thieme Stuttgart; 2001, pp. 190-200.

Wilson HJ. Resin-based restoratives. Br Dent J 1988;164:326-30.

Yazici AR, Kugel G, Gul G. The knoop hardness of a composite resin polymerized with different curing lights and different modes. J Contemp Dent Pract 2007;8:52-9.

Yearn JA. Factors affecting cure of visible light activated composites. Int Dent J 1985;35:218-25.

Yoshida S, Namura Y, Matsuda M, Saito A, Shimizu N. Influence of light dose on bond strength of orthodontic light-cured adhesives. Eur J Orthod 2012;34:493-7.

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