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To my boys xxx

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ABSTRACT

In orthodontic bonding a bracket is placed on the tooth enamel and forces are applied to move the teeth. Orthodontic bracket should therefore be resistant to both applied and masticatory forces. Polymerization of the adhesive can be activated chemically or by light. Light curing of the adhesive under stainless steel brackets is a matter of interest among clinicians and researchers. It is known, that proper polymerization of the adhesive resin influences to the mechanical properties of a bonded bracket. Unpolymerized monomers decrease bond strength but are also shown to have harmful effects when leaching to the oral cavity. The aim of this thesis was to investigate polymerization of the adhesive and bond strength of orthodontic brackets when bonded with light-curing resin adhesive with different light curing techniques. Furthermore, light transmission through human teeth is studied to evaluate transillumination as a curing method.

To study polymerization, the degree of conversion (DC%) was measured after light curing the adhesive under bracket of various compositions. It was found, that the DC % of the adhesive was significantly higher under transparent brackets varying from 48% to 52% with direct curing. However, the study confirmed that the DC% under metal brackets can be improved by 17–21% by adding a glass-fiber weave under the bracket.

According to the results of this study, the light attenuation through human teeth follows the Beer-Lambert law where the transmitting light decreases as the specimen thickness increases. The present study showed a significant difference in light attenuation, where 1 mm thick layer of enamel attenuated 73% of incoming light and dentin 79%, respectively. 4 mm thick tooth barrier was found to obstruct all light transmission and the light transmission through extracted incisors were found the be 38 mW/cm^2 and 6 mW/cm^2 for premolars, respectively. Light curing by transillumination through the extracted incisors however resulted comparable DC% and bracket bond strength to conventional curing where the light is administrated from the sides of the bracket. In case of extracted premolars, light curing by transillumination showed poor DC% and bracket strength values compared to conventional curing.

To conclude, polymerization of the orthodontic adhesive under the bracket can be achieved with multiple combinations of curing time, direct of light curing and bracket material.

KEYWORDS: Transillumination, Light-Curing of Dental Adhesives, Bracket, Bonding

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TIIVISTELMÄ

Kiinteissä oikomiskojeissa käytetään hampaita siirtävien voimien välittämiseen oikomiskiinnikkeitä eli braketteja, jotka kiinnitetään hampaan kiilteeseen tavallisimmin valokovetteisen sidosmuovin avulla. Kiinnityksessä käytetty kovetin tuottaa valoa, joka saa kiinnitysmuovissa aikaan polymerisaatioreaktion, jonka vaikutuksesta kiinnityksestä tulee riittävän luja. Polymerisoitunut sidosmuovi kestää siihen kohdistuvan rasituksen murtumatta ja on samalla turvallinen suun olosuhteissa. Oikomishoidossa tavallisimmin käytetyt metalliset kiinnikkeet estävät sidosmuovin suoraa valottamista ja vaativat erityisen tekniikan, jotta kovetus on riittävä. Kovettumattomasta sidosmuovista saattaa ajan kuluessa vapautua haitallisia monomeereja, jotka ovat haitallista päästessään suun limakalvojen kanssa kosketuksiin. Tässä väitöskirjassa tutkittiin valokovetteisten sidosmuovien polymerisoitumista erityyppisten kiinnikkeiden alla käyttäen erilaisia valokovetuksen menetelmiä. Lisäksi tutkimuksen kohteena oli hampaan eri kovakudosten erot valonläpäisevyydessä.

Tutkimuksessa todettiin, että kovettamisaste metallisten kiinnikkeiden alla on merkittävästi matalampi kuin läpikuultavien. Polymerisoitumisaste läpinäkyvien kiinnikkeiden alla vaihteli 48–52 %:n välillä, kun taas metallisten kiinnikkeiden alla polymerisoitumisaste jäi alle 40 %. Tutkimuksessa kuitenkin vahvistettiin aiemmin tutkittu tieto, että lasikuituverkon avulla voidaan polymerisoitumisastetta parantaa jopa 17–21 %.

Hampaan läpi kulkeva valo noudattaa tutkimuksen mukaan Beer-Lambertin lakia, jossa näytteen läpi tulevan valon intensiteetti laskee näytepaksuuden kasvaessa. Tutkimuksessa todettiin myös merkittävä ero hampaan kiilteen ja dentiinin valonläpäisevyydessä. 1 mm paksuinen kiillenäyte läpäisi 27 % valokovettimen valosta, kun taas saman paksuinen dentiinileike vain 21 %. 4 mm paksun dentiinileikkeen todettiin vaimentavan sen läpi osoitetun valon. Tutkimuksessa todettiin, että inkisiivin läpi kulkeutuvan valon intensiteetti on keskimäärin 38 mW/cm^2, kun taas premolaareilla vastaava arvo on 6 mW/cm^2. Valokovetus inkisiivin läpi osoitti kliinisesti hyväksyttäviä arvoja sekä sidosmuovin polymerisoitumisen että irroitusvoiman suhteen. Kiinnikkeen sidostamisessa premolaareihin sen sijaan havaittiin, että valokovettamalla sidosmuovi kiinnikkeen sivuilta saavutetaan parempi polymerisoitumisaste sekä kiinnitysvoima kuin valokovettamalla koko hampaan läpi. Yhteenvetona voidaankin todeta, että valokovetteisen kiinnikemuovin riittävä kovettumisaste voidaan saavuttaa erilaisilla yhdistelmillä muutellen kovettamisaikaa, valon suuntaa sekä kiinnikemateriaaleja.

AVAINSANAT: Valokovetus, Oikomishoito, Sidostus

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Abbreviations

ANOVA	Analysis of variance
ARI	Adhesive remnant index
ATR	Attenuated total reflectance
Bis-GMA	Bisphenol-A-glycidyl ether dimethacrylate
BPA	Bisphenol-A
BS	Bond strength
CQ	Camphorquinoine
DC%	Degree of conversion
DF	Debonding force
EDTA	Ethylenediaminetetraacetic
FT-IR	Fourier transformed infrared, spectrometer
FRC	Fiber reinforced composites
HAP	Hydroxyapatite crystals
LCU	Light curing unit
LED	Light emitting diode
mm	Millimeter
MPa	Megapascal
nm	Nanometer
PFC	Particulate filler resin composite
PFR	Particulate filler resin
PMMA	Polymethylmethacrylate
POB	Pre-coated orthodontic
SBS	Shear bond strength
SD	Standard deviation
TEGDMA	Triethylene glycol methacrylate
µm	Micrometer
∅	Diameter

List of Original Publications

This dissertation is based on the following original publications, which are referred to in the text by their Roman numerals:

I L. Kilponen, **E. Uusitalo**, M. Tolvanen, J. Varrela, and P. K. Vallittu. "Photopolymerization of Light Curing Adhesives Used with Metal Orthodontic Brackets and Matrices". Journal of Biomaterial Tissue Engineering 2016;6:659–664.

II **E. Uusitalo**, J. Varrela, L. Lassila, and P. K. Vallittu. "Transmission of Curing Light through Moist, Air-Dried, and EDTA Treated Dentine and Enamel". BioMed Research International 2016, Article ID 5713962, 6 pp.

III **E. Mäkinen**, L. Lassila, J. Varrela and P.K. Vallittu. "Light-curing of Orthodontic Bracket Adhesive by Transillumination through Dentine and Enamel". Biomaterial Investigations in Dentistry 2019;6:6–12.

IV **E. Mäkinen**, A. Suominen, J. Varrela and P.K. Vallittu, "Monomer Conversion of Orthodontic Resin Adhesive Under Brackets of Various Kinds", manuscript.

E. Uusitalo and E. Mäkinen represent the same author. The original papers are reproduced with permission of the copyright holders.

1 Introduction

Orthodontic brackets are usually bonded to tooth enamel with light-activated resin based adhesives. This has become popular because light curing provides significant advantages compared to chemically curing adhesives, such as longer application time and reduced chair-time. Stainless steel brackets are most commonly used but because metal prevents transmission of light, a direct curing of the adhesive is not possible. To cure the adhesive under metal bracket, the light must be transmitted sideways to the center of the bracket, or the light curing can be attempted from the palatal or lingual side of the tooth. In case of tooth-colored brackets, light curing can be performed through the bracket.

With metal brackets, there is a risk that the adhesive at the center of the bracket remains incompletely polymerized, due to the convexity of the labial tooth surface and attenuation of light. Incompletely polymerized adhesive can leach acrylate components such as bis-GMA (bisphenol A diglycidyl ether dimethacrylate) which is a common monomer in light-curing adhesives. The leaching of residual monomers is potentially harmful, and if the degree of conversion (DC%) under orthodontic brackets remains low, significant leaching of residual monomer and other substances may occur. Thus, it is important to obtain a sufficient level of DC% throughout the bracket area.

It has been shown that glass fillers can be used to enhance the scattering of light to the center of the bracket and thereby increase the DC% of the adhesive. In addition to the use of glass fiber reinforcement, curing with transillumination through the tooth has been suggested to improve the DC% under the bracket, but only few studies have yet been conducted. Dental hard tissues and resin matrix itself presumably attenuate light transmission by decreasing the light energy that reaches the adhesive under the bracket. By prolonging the curing time, it is possible to increase the number of free radicals and consequently the DC% if the curing light is able to reach the unpolymerized monomers and the amount of initiator present is sufficient. The level of the DC% is known to correlate with the mechanical properties of the polymerized adhesive; therefore improving the DC% will contribute to the clinical performance of the bonded brackets.

2 Review of the Literature

2.1 Bonding in orthodontics

The first fixed orthodontic brackets were welded to stainless steel or gold bands which then were cemented around the teeth. However, this technique required tooth separation to create interproximal space between teeth that was carried out with wires and more recently with elastomers. This was time consuming for the clinician and uncomfortable for the patient. (Gange 2015)

In 1960s, researchers began to test adhesives that could be used to bond plastic brackets directly to enamel surface of teeth. In 1970s, Miura et al. (1971) developed a technique to bond polycarbonate brackets to the tooth enamel by using a restorative composite with acid etching technique. However, they found that humidity in oral cavity resulted as weak bond strength and the use of metal archwires and ligatures caused multiple fractures to the polycarbonate bracket tie wings. Despite the weaknesses of the early polycarbonate brackets, these findings started a new era in the development of fixed appliances.

After introduction of the first direct bonding techniques, several new studies were conducted and new materials tested. The first adhesives to bond metal brackets were based on the research of Bowen et al. (1956) and were introduced to the market by Retief et al. (1970). The benefits of using epoxy resin based on new monomer were the low shrinkage during polymerization, thermal coefficient of expansion similar to enamel and low water absorption due to cross-linked structure. These characteristics provided better mechanical attachment of metal brackets to resist masticatory and orthodontic forces.

Soon after the release of chemically cured orthodontic adhesives, UV light curing composites were introduced by Dentsply in 1974 (Gange 2015). UV-curing however had disadvantages such as eye and soft tissue irritation. In 1980's adhesives that were cured by visible light were introduced and became popular among orthodontist.

In contemporary fixed orthodontic treatment, appliances are attached to the enamel surface by means of direct bonding with light curing adhesive. Bonding of orthodontic brackets straight to the tooth enamel is currently the most popular technique among clinicians. Not only were the metal bands unpractical for clinical

use, they also presented a risk for gingival trauma. (Mitchell 2007) Nevertheless, traditional bands are still frequently used around molars for better stability against greater masticatory- and orthodontic forces.

2.1.1 Dental tissues as bonding substrates

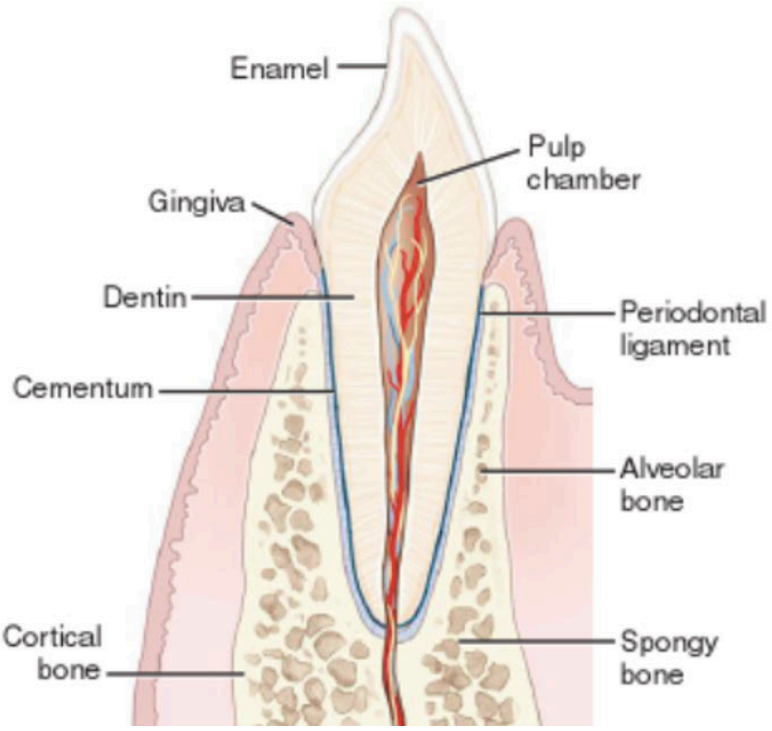

Figure 1. Schematic presentation of vertical cross-section of incisor. Picture from the book *Phillips' Science of Dental Materials.* Anusavice KJ., Shen C., Rawls HR. 2012. 12th Edition.

2.1.1.1 Dental enamel

Enamel is a very complex hierarchical structure of mineralized material, that is mainly composed of aligned rods of 6–8 µm diameter. Rods are arranged nearly perpendicular to the tooth surface creating an anisotropic structure. (Cheng et al. 2010, Cui and Ge 2007) Mature enamel shows a mineralization gradient which decreases from outer to inner enamel. The outermost layer of enamel is usually harder due to the salivary calcium ions that can mineralize into the enamel and the fluorine ions that transform the hydroxyapatite to fluoroapatite. Also the composition of enamel varies according to patient's age and eating habits; in young tooth the outer layer of enamel is condensed and aprismatic. (Gwinnet 1967, Gwinnet 1992, Lopes 2007)

Enamel rods consists of fibril-like hydroxyapatite crystals (HAP) with a diameter of 30 nm. HAP crystals are positioned parallel and aligned lengthways to the rod axis and hence the complex structure of tooth enamel the mechanical properties vary in different areas of tooth. (Habelitz et al. 2001) Schematic drawing of a vertically cut incisor is presented in Figure 1.

When a resin based adhesive is bonded to tooth enamel the bonding is primary thought to be based on micromechanical interlocking of the resin to the etch pits which are formed during acid etching or by using self-etching adhesive (Buonocore et al. 1968). Hannig et al. (2002) found that the non-decalcified ultrathin sections, taken at the interface between enamel and self-etching adhesive, showed a 1.5–3.2 μm deep enamel layer, characterized by a less-dense arrangement of enamel crystallites separated from each other by nanometer-sized spaces. In addition, they observed a 1.5–3.2 μm wide netlike resinous structure in corresponding decalcified specimens. It was concluded that self-etching priming agents dissolve both peripheral and central part of the enamel crystallites and promote inter- and intra-crystallite monomer infiltration. A similar phenomenon was detected with a 3-step adhesive systems causing even deeper enamel surface hybridization (6.9 μm) (Hannig et al. 2002).

2.1.1.2 Dentin

Dentin has a tubular structure, the density and diameter of the tubules increasing from the outer to inner layers of dentin. Dentin is composed of a mineral phase (40–45% in volume), organic matrix (30% in volume) and water (20–25% in volume). Intertubular and peritubular dentin can be found from the lumen of the tubules. In the intertubular dentin, collagen I is the major protein, whereas no collagen fibrils are found in the peritubular dentin. (Goldberg et al. 2011)

Unlike enamel bonding, dentin bonding relies on formation of a hybrid layer that is composed of reinforced demineralized collagen fibrils in the resin matrix. (Nakabayashi et al. 1991). Because the dentin bonding is based on the presence of a hybrid layer, the collagen fibrils must be revealed. Acid etchant is used to remove the mineral crystals and to expose the collagen fibrils. After etching and careful air drying, applying the hydrophilic primer will allow the infiltration of resinous monomers into the hydrophobic collagen matrix of demineralized dentin (Swift et al. 1995, Breschi et al. 2018).

2.1.1.3 Curing light transmission through dental hard tissues

When light propagates through scattering media, it becomes a mixture of absorbed, transmitted and reflected light. (Gobrecht et al. 2015) These features of light

determine the opacity, translucency and transparency of the tooth. Scattering is a phenomenon where the light is reflected from the rough, uneven surface, such as etched enamel. (Anusavice et al. 2012) When the light first reaches the enamel surface, it is transmitted through the enamel via rods and HAP crystals, scattered from the incisal and approximal edges of the tooth (edge-loss effect) or absorbed (Yu et al. 2009). Human enamel is highly translucent, the translucency representing the relative amount of light transmission or the diffuse reflectance from the substrate surface when light is traveling through turbid media. The translucency of enamel depends on the wavelength of incoming light, as the translucency increasing with increasing wavelength of light. (Brodbelt et al. 1981) However, translucency describes the optical properties of a media, such as enamel, whereas light transmission is a physical term representing the ability of turbid media to pass light through (Xiong et al. 2008, Lee 2015).

Vaarkamp et al. (1995) suggested that dentin tubules are in the key role of light scattering in dentin, based on the finding that light transmission was greater when light was directed parallel to the dentin tubules. Zijp and Bosch (1993) concluded that most of the transmitted light is scattered from the dentin tubules and scattering from the collagen fibrils and mineral crystals is of minor importance.

When light is transmitted through the tooth, it first scatters from the enamel and then from the dentinal tubules. Reflections of light at the material interphases, e.g. at the junction of dentin and enamel, scatter the light. The amount of scattered light is a function of relative refractive indices of two different phases. (Southan 1987) The refractive index for enamel is 1.63 and that for dentin 1.54 (Meng et al. 2009). In case of vital, young teeth light may also propagate through pulp tissue. This, however, is a complex issue to study because it can be affected by trauma, caries and individual variations in tertiary dentin formation.

There are only few studies that have investigated the light transmittance through human and/or bovine teeth. Oesterle et al. (2001) studied the light transmission through extracted human lateral and central maxillary incisors with average thickness of 4.2 mm. The authors found out that the light irradiance was significantly decreased when transmitted through the tooth. Similar results were obtained by Kumar et al. (2013) who studied light attenuation through the tooth.

2.1.2 Orthodontic brackets

Orthodontic brackets act as a medium between the tooth and the applied orthodontic forces that are executed on tooth via archwires and ligatures with intention to move the tooth to the desired place and position in the dental arch. Brackets should be able to resist masticatory forces, mediate orthodontic forces and be comfortable for the

patient. Orthodontic brackets can be metallic (stainless steel, gold, titanium) or tooth-colored (ceramic, plastic) (Subramani et al. (2013).

2.1.2.1 Metal brackets

Metal brackets are commonly manufactured from stainless steel that contains also chromium and nickel. (Fonseca et al. 2014). The basic structure of metal brackets is presented in Figure 2.

Figure 2. The structure of metal bracket includes a slot for the archwire (usually of width of 0.018 or 0.022 inch), wings for ligating, and a base structure that is designed to lock the adhesive (photo: Nielson2000 / CC-BY-SA-3.0).

A mesh pattern of the bracket base, introduced by the Ormco Company (Orange, Calif) in 1979, allows the adhesive to penetrate the mesh before polymerization (Gange 2015, Knox et al. 2000).

When bonding metal brackets to enamel with resin based adhesive, the bond between the bracket and adhesive is based on mechanical retention. Retention of metal brackets is commonly achieved by mesh pattern of the base (Smith and Maijer 1983). Retention can also be achieved by grooving, sandblasting, chemical etching or sintering with porous metal powder (Wang et al. 2004). The size of the bracket base influences to the bond strength, the bond strength increasing as the diameter of the bracket base becomes larger (Wang et al. 2004). The bond strength of stainless steel brackets is not affected by a prior application of adhesive resin, but it is influenced by variables such as curing time, bracket base area and force location (Altmann et al. 2016). In addition, results of laboratory testing of bond strength depend on the crosshead speed of the test machine.

2.1.2.2 Ceramic and plastic brackets

Plastic brackets were first made of acryl, and subsequently, of polycarbonate. Problems encountered with plastic brackets included discoloration, breakage of the ligature tie wings, bracket deformation and poor bond strength. (Matsu et al. 2015) To overcome weaknesses of polycarbonate brackets, glass fibers and ceramic fillers were blended with polycarbonate, and metal reinforced slots were placed in the brackets. However, Nishio et al. (2009) showed that adding ceramic fillers in the plastic bracket did not increase its strength but a metal slot did enhance its resistance against deformation.

Ceramic brackets were introduced as an alternative to plastic brackets because they are resistant to abrasion, have a high rigidity and are free from discoloration. Furthermore, ceramic brackets have similar resistance against moment loads as stainless steel brackets. (Matsui et al. 2015) Nevertheless, regardless of several favorable features, ceramic brackets are brittle and they can cause tooth wear to the opposing tooth. In contrast to the stainless steel brackets, ceramic brackets tend to have problems with debonding because of the strong chemical bond between ceramic bracket, adhesive and tooth enamel. Therefore, enamel damage is more likely to occur when debonding ceramic brackets than metal brackets (Joseph and Rossouw 1990). Enamel cracks have commonly been reported, up to 20% of teeth, after debonding ceramic brackets with pliers (Mundstock et al. 1999, Liu et al. 2005, Bishara et al. 1995). It has been shown that chemical bonding (silanized bases) of the ceramic brackets results in higher bond strengths than mechanical bonding, bond strength being almost as high as that between adhesive and enamel, which increases the risk of damage (Gittner et al. 2012, Falkensammer et al. 2012). Klocke et al. (2003) did not observe enamel fractures in their study with mechanically retained ceramic brackets but multiple studies have reported enamel defects associated with debonding of ceramic bracket with silanized bases (Viazis et al. 1990, Winchester 1991, Gittner et al. 2012). Thus, mechanic retention of the bracket base should be favored when bonding ceramic brackets.

Ceramic and polycarbonate brackets are tooth-colored, but come in different shades and sizes. Tooth-colored brackets aim to be as invisible as possible for better esthetics and patient comfort (Fonseca et al. 2014). Ceramic and polycarbonate brackets are designed to be translucent like human enamel (Southan 1987) making it superior from esthetic point of view (Yu et al. 2009). Factors that affect translucency include ceramic thickness, crystalline structure, ceramic grain size, shade, pigment and porosity of the ceramic (Heffernan et al. 2002). The translucency of dental ceramics further depends on light scattering: if the major amount of light transmission is scattered and diffusely reflected, ceramic will appear opaque. (Brodbelt et al. 1980)

2.1.3 Principles of bracket bonding

Bond strength between bracket and enamel depends on three factors: retentive properties of the bracket base, chemical properties of the adhesive resin and preparation of the tooth surface (Urabe et al. 1999). In addition to these factors, light-curing technique and degree of conversion of the adhesive play a crucial role in the survival of brackets during treatment.

2.1.3.1 Surface conditioning

In orthodontics, surface conditioning focuses mostly on enamel. Acid etching of the enamel with 35% phosphoric acid removes few micrometers from the top layer of enamel and forms a better base for bonding by increasing the area, energy and microporosity of the enamel surface where the adhesive can flow in and form microtags. (Buonocore et al. 1968) Microtags have been reported to reach a length up to 50 μm (Fitzpatrick and Way 1977) but the bond strength does not seem to increase with increasing the length of the microtags (Shinchi et al. 2000). In addition to the microtags, retention is enhanced by chemical adhesion between the calcium ions of the enamel and the chelating functional groups of the monomers (Anusavice et al. 2012).

In clinical work phosphoric acid with 35-40% concentration is conventionally used for etching. Acid etching dissolves selectively the hydroxyapatite crystals within prismatic and interprismatic enamel resulting in different etching patterns depending on the direction of enamel rods. Three types of etching patterns have been recognized: the center of the prisms is dissolved, the periphery of the prisms is dissolved, or no prism structure is visible (Lopes et al. 2007). The formation of the etching pattern depends also on the composition of enamel, which is affected by patient's age, daily fluoride intake and composition of the saliva (Gwinnet 1992). In enamel bonding, etching and rinsing technique, also known as the "total-etch" technique, remains as a golden standard (Van Meerbeek et al. 2008).

2.1.3.2 Adhesive selection

During the recent decades, direct bonding of the orthodontic fixed appliances has markedly been developed. In 1955 Buonocore et al. first described a method to bond acrylic resin to tooth enamel after acid etching. In 1965, Newman suggested that direct bonding could be used in orthodontics.

Conventional adhesive system contains three agents: enamel conditioner, primer solution and adhesive resin (4^{th} and 5^{th} generation techniques, see Figure 3 showing a classification of techniques). After etching the enamel, a thin layer of primer is applied to the enamel surface in order to enhance the bond strength between enamel and adhesive resin. Primer increases resistance for marginal microleakages (Lüscher

et al. 1978), enamel damping (Rider et al. 1977) and retention to etched enamel (Smith et al. 1987, Baharava et al. 1988). However, a primer does not affect bracket bond strength (O'Brien et al. 1991, Arash et al. 2017). After etching and priming of the enamel, a thin layer of adhesive is applied onto the bracket base, and the bracket is placed firmly against the enamel surface in the desired position.

To cut the chair-time, a "universal" adhesive for a 1-step self-etching technique has been developed to be used, either with conventional phosphoric acid etching or without a separate etching (7th generation) (Hanabusa et al. 2012). It seems, however that a preparatory etching of the enamel significantly increases the bond strength for both 1-step "multi-mode" self-etching adhesives (7th generation) and for 2-step self-etching adhesives (6th generation) (Hanabusa et al. 2012, de Goes et al. 2014). Despite weaknesses of the 7th generation adhesive systems, they have many advantages that are tempting for clinicians: short, user friendly bonding protocol, lower risk for post-operative sensitivity as over etching can be avoided and lower risk for user errors (Bishara et al. 2001).

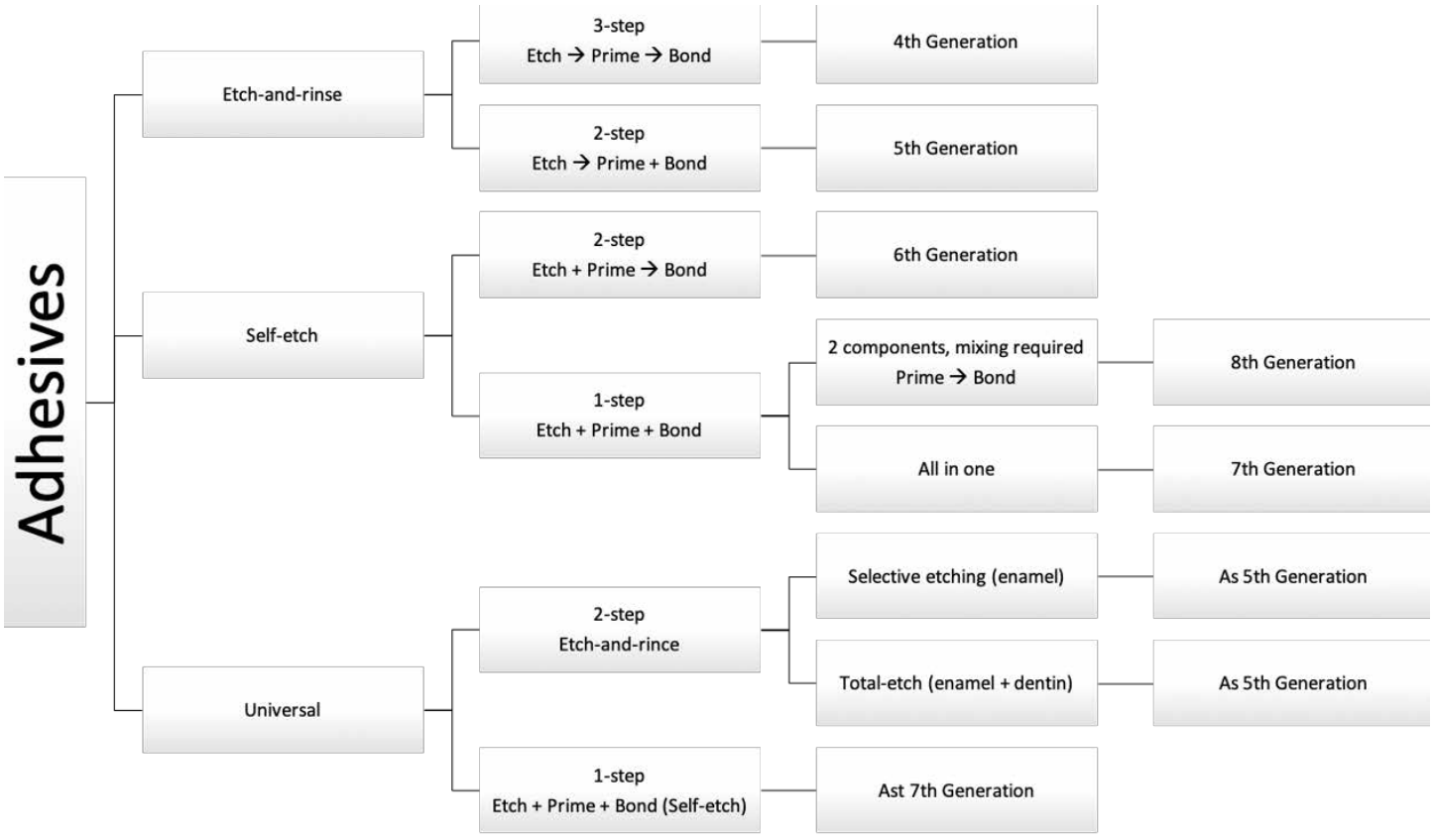

Figure 3. Classification of adhesive generations (Van Meerbeek et al. 2003, Sofan et al. 2017)

2.1.3.3 Light curing

Bonding is based on curing the adhesive resin which forms the mechanical attachment between bracket material and enamel. Resin composite and adhesive are

interlocked mainly by penetration of the adhesive into the microirregularities of the enamel surface and mesh-based bracket. (Ferracane et al. 2003)

Manufacturers' instructions vary depending on the bracket material and adhesive resin system. For metal brackets, light curing is recommended from the sides of the bracket, from 3 to 10 seconds per side. Tooth-colored ceramic or polycarbonate brackets are commonly cured through the bracket with similar curing times as with metal brackets. However, translucency of the bracket material must be taken into consideration, different ceramic/polycarbonate brackets absorbing different amount of light (Lee 2015, Mohamed et al. 2016).

When the adhesive is light cured from the sides of a metal bracket, convexity of labial/buccal tooth surface curing light may arrest the penetration of light to the center of the bracket resulting in low degree of conversion of the monomer and leaching of unpolymerized monomers (Eliades et al. 1995 (1), Eliades et al. 1995 (2)). To solve this problem, light curing through the tooth has been suggested. Tavas and Watts (1979) first introduced transillumination technique with light curing at 45 degrees' angle to the lingual and occlusal surfaces and concluded that transillumination could enhance the clinical performance of brackets. King et al. (1987) found that that by increasing the transillumination time, a bond strength of 6–8 MPa was achieved which was considered adequate to withstand masticatory and orthodontic forces (Reynolds 1975). However, if the bond strength is measured as shear bond strength (SBS), a strength at the level of 20 MPa is commonly regarded as sufficient to withstand masticatory forces in the premolar and molar area (Barkmeier et al. 1986, Gilpatrick et al. 1991). Cheng et al. (1989) studied light curing *in vivo* directing the light from the incisal edge and shielding the tooth with a light inhibiting mold. They concluded that illuminating along the vertical axis of the tooth for 20 or 40 seconds resulted softer and therefore less polymerized adhesive compared to direct illumination. However, they did not test direct light transmission through the tooth. Oesterle and Shellhart (2001) showed that transillumination through extracted maxillary incisors resulted in sufficiently high bond strengths, particularly when the curing time was increased to 50 seconds. Kumar et al. (2013) studied effectiveness of transillumination through maxillary incisors and premolars and concluded that light transmission depended on the thickness of the teeth. Heravi et al. (2013), on the other hand, found that light curing through premolars resulted in bond strengths below clinically acceptable values even if curing time was increased to 80 seconds. Heravi et al. (2013) suggested that bond strength of incisor brackets can be enhanced by increasing light intensity and the exposure time. Dobrin et al. (2018) studied bond strength of incisor and premolar brackets by directing light at different angles from the lingual or labial side of the tooth, or from both. The authors found that in the maxillary central incisors, curing from the lingual side through the tooth resulted in nearly equal bond strength compared to curing from the labial side.

On the lateral incisors, the brackets showed higher bond strengths when cured from the lingual side at mesial-distal direction compared to curing from the labial side.

2.1.4 Clinical aspects in bracket bonding

There has been a trend in orthodontics to reduce steps of the bonding protocols without compromising bond strength. Treatment with fixed appliances requires sufficient bond strength to minimize bracket failures during the treatment, and at the same time, safe and easy removal of the brackets after the treatment. Bond strengths between 5.6 to 7.8 MPa are considered sufficient to withstand masticatory and orthodontic forces (Reynolds 1975). On the other hand, the risk for enamel fracture during debonding is increased when the bond strength exceeds 9.7 MPa (Ostertag et al. 1991). Therefore, moderate bond strengths are favored in clinical use, since they are less likely to cause enamel fractures during debonding (Kilponen et al. 2019).

The failure rates for metal, ceramic and plastic brackets are reported to vary between 3.9–11.2% depending on the bonding protocol (Hitmi et al. 2001, Murfitt et al. 2006, Romano et al. 2012). Resin based orthodontic adhesives produce higher bond strengths compared to glass-ionomer cements, but resin-modified glass-ionomer cements can provide clinically sufficient bond strengths (Cook and Youngson 1988, Klockowski et al. 1989, Hitmi et al. 2001, Yassaei et al. 2014). Brackets bonded with the conventional etch and rinse method had 60% lower risk for bracket failure over an observing period of 12 months compared to brackets bonded with self-etching adhesive (Murfitt et al. 2006).

In addition to the bonding protocol, patients' age and gender impact on the bracket survival rate. Males and younger patients (12 years or younger) have increased risk for bracket failure compared to females and patients of the age of 16 years or more (Millet and Gordon 1994, Murfitt et al. 2006). Furthermore, the place of the bracket in the dental arch affects the survival rate, the lowest failure rate being in maxillary incisors and the highest in premolars (O'Brien et al. 1989, Millet and Gordon 1994). Because of the sensitivity of the acid-etching technique to moisture, bonding *in vivo* in oral conditions in challenging, especially in the posterior area (Hormati et al. 1980, O'Brien et al. 1987, Silverstone et al. 1985).

2.2 Resin based orthodontic adhesives and composites

Dental applications of resin composites were introduced in the mid-60's, first for restorations of the anterior teeth (Peutzfeldt 1997). Since 1980's, light-cured composites have become popular in dentistry, including orthodontics. Adhesives used in orthodontics are modified from restorative adhesives to have lower viscosity

and to allow a better penetration of the adhesive into the mesh base of the bracket and etched enamel surface (Eliades et al. 2001, Sakaguchi and Powers 2012).

2.2.1 Adhesives

Dental resin adhesives have three main components: resin matrix, monomer system and initiator system for free radical polymerization (Rueggeberg et al. 2017). Monomers used in orthodontic resin adhesives are commonly Bis-GMA and TEGDMA (Figure 4). Bis-GMA, also known as Bowen's resin, contain aromatic ring and hydroxyl groups forming hydrogen bonds that impair mobility. Due to two –OH groups forming hydrogen bonds, bis-GMA is highly viscous. Advantages of bis-GMA is relatively less shrinkage, reduced tissue toxicity and higher modulus compared to smaller methyl methacrylates (Sideridou et al. 2002). Rigid central core of two aromatic rings reduce the ability to rotate during polymerization and thereby participate more efficiently to the polymerization process which is better known as degree of conversion. Disadvantages of the bis-GMA are high molecular weight, stiffness and rigidity which shows as hindrance of curing light during polymerization (Söderholm and Mariotti 1999).

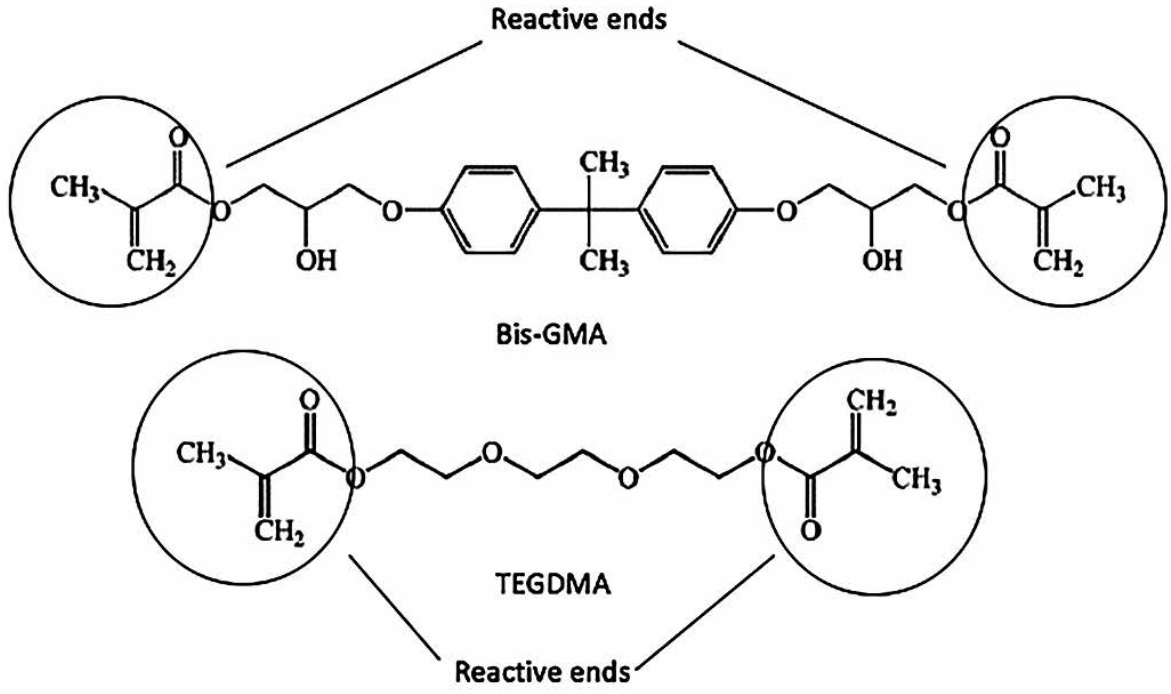

Figure 4. Monomer structures of bis-GMA and TEGDMA. Reactive ends are the C=C double bonds that link monomers into growing polymer chain.

Bis- GMA is often combined with more flexible monomers such as TEGDMA to reduce viscosity and to increase to degree of conversion. TEGDMA has a flexible

aliphatic structure, which increases reactivity and monomer mobility. (Pfeifer et al. 2011) Initiator system in light-curing resin adhesives is usually composed of camphorquinone and tertiary amine system. (Moszner et al. 2008).

2.2.2 Particulate filler resin composites (PFCs)

Particulate filler resin composites, known as PFC's, consist on resin based matrix that have fillers incorporated within the resin matrix (Thomaidis et al. 2013). Composites are usually filled with silica, barium oxide, strontium, zinc, aluminium or zirconium (Khan et al. 1992). Silanes with functional methacrylate groups are necessary to provide a strong bond between inorganic fillers and organic monomers (Faltermeier et al. 2007). Filler particles give strength and wear resistance to otherwise soft polymer matrix and so form the composite material. Like resin adhesives, also PFC's have light activated initiator system, mainly composed of camphorquinone and tertiary amine (Moszner et al. 2008).

It is known that a high filler ratio increases the viscosity of the composite and reduces its ability to flow to the bracket base mesh pattern or grooves, and to etch pits of the etched enamel surface. Low-viscosity adhesives have been developed, which retain the small filler particle size of the conventional PFC's while the filler content is reduced (Tecco et al. 2005). Some studies have reported lower bond strengths of the brackets bonded with low-viscosity adhesives compared to normal-viscosity adhesives (Faltermeier et al. 2007, Ryou et al. 2008) whereas others did not find significant differences between the two adhesive types (Tecco et al. 2005, Albaladejo et al. 2011). From the clinical point of view, a good adhesive has a balance in stiffness and flowability, i.e. enough stiffness to prevent sliding of the bracket but flowable enough to penetrate the mechanical attachment in the bracket base and enamel. Low-viscosity adhesive resins are commonly used in orthodontics (Tecco et al. 2005, Ryou et al. 2008, Albaladejo et al. 2011).

2.2.3 Fiber-reinforced composites (FRCs)

Glass-fibers are used in many dental applications such as prosthetic restorations, root canal posts, oral implant abutments and composite fillings (Bell-Rönnlöf 2007, Abdulmajeed 2013, Mosquera 2015, Omran 2019). FRC contains a polymer matrix and reinforcing fibers. Fibers can be either continuous unidirectional (rovings), continuous bidirectional (weave) or continuous random oriented (mat), or short, random oriented fibers (Vallittu 2008). Matrix is composed of bis-GMA, TEGDMA and polymethylmethacrylate (PMMA). Thus, two types of resins are present: cross-linked and linear which together form a semi-interpenetrating network (semi-IPN) (Vallittu 2008, Vallittu 2015). With proper pre-impregnation, glass-fibers enhance

the flexural properties of dental composites and provide better resistance against fatigue and fracture, especially in posterior fillings, and improved bond strength due to micromechanical attachment (Vallittu 1999, Lastumäki et al. 2002, Garoushi et al. 2006).

In orthodontics, FRCs are used for bonded retainers, space maintainers, and for anchorage and active tooth movement. Pre-impregnation allows the clinician to form long FRCs before polymerization and provides good coupling and esthetics. (Freudenthaler et al. 2001, Karaman et al. 2002) Shinya et al. (2009) showed that after adding a glass-fiber weave under the bracket, the DC% of the adhesive was significantly higher. In addition, more consistent DC% values were obtained by using the weave. Higher DC% was explained with the fibers' ability to conduct light under the metal bracket and enhance scattering of light. It is known that during the polymerization of bis-GMA-TEGDMA monomer system its refractive index increases while the refractive index of glass fibers remains the same. This improves scattering of light from the glass fibers which enhance curing of the resin phase (Lehtinen et al. 2008).

2.2.4 Polymerization of resin dental systems

Monomers can be linked together by either addition or condensation reactions. In addition polymerization, monomers are activated one after another and the monomers are added sequentially to the end of the growing chain, whereas in condensation polymerization all monomers are activated simultaneously. In condensation polymerization, the chains grow by stepwise linking of bifunctional monomers that produce by-product, such as water, that is to be 'condensed out'. Most dental resin systems are polymerized by addition polymerization. Addition polymerization in modern resins is based on C=C double bonds. (Anusavice et al. 2012)

The first stage of polymerization is induction where polymerization of resin composites is induced by free radicals that are single or groups of atoms possessing an unpaired electron. The free radicals are generated by the delivery of the photons of the curing light with a wavelength of 400-500 nm. During the polymerization, a free radical with unpaired electron approaches the C=C double bond of high-electron density. After extraction of the electron, a bond is formed between the radical and monomer, leaving the other electron of the double bond unpaired. Thus, a new free radical site is formed at the other end of the molecule. The resin matrix undergoes additional free-radical-induced polymerization where methacrylate monomers are linked to polymer chain. (Anusavice et al. 2012, Leprince et al. 2013, Floyd and Dickens 2006) Active polymerization continues until all monomers have reacted or the system reaches stability (Burtscher 1993).

All mechanical properties of resin composites are measures of the resistance to material deformation, fracture under pressure or induced stress. Desired properties of adhesive resin used in orthodontic field varies among purpose. Long-lasting, wear-resisting adhesives are ideal for example bonding lingual retainers when on the other hand bonding brackets require different properties. In case of bonding orthodontic brackets bond strength and safe debonding without enamel fractures are in key role. (Sifakakis et al. 2017, Anusavice et al. 2012).

Degree of conversion (DC%) is the percentage of reacted C=C double bonds. Mechanical properties of the adhesive including flexural modulus of elasticity, tensile strength and compressive strength depend on the degree of cure (Eliades et al. 1987, Eliades et al. 1995 (1)). In addition, DC% modulates solubility, biocompatibility, color stability and degradation of the adhesive. Indirect measuring techniques, such as the surface microhardness and bond strength tests, measure the mechanical properties of the adhesive but do not provide information about polymerization of the material. (Purushothaman et al. 2015)

DC% of a resin adhesive is commonly measured with fourier-transformed infrared (FT-IR) spectroscopy ATR (attenuated total reflectance) accessory. IR-spectroscopy scans the sample with infrared radiation which induces vibration in C=C double bonds and is used to specify the molecular structure of the sample (Leprince et al. 2013, Zhang et al. 2016). The DC% can be calculated from the aliphatic C=C peak (1638 cm^{-1}) and the aromatic C=C peak (1608cm^{-1}) of bis-GMA using Equation 1.

$$DC\% = \left[1 - \frac{C_{aliphatic} / C_{aromatic}}{U_{aliphatic} / U_{aromatic}}\right] \times 100\%$$

Equation 1

$C_{aliphatic}$ = absorption peak at 1638 cm^{-1} of the cured sample
$C_{aromatic}$ = absorption peak at 1608 cm^{-1} of the cured sample
$U_{aliphatic}$ = absorption peak at 1638 cm^{-1} of the uncured sample
$U_{aromatic}$ = absorption peak at 1608 cm^{-1} of the uncured sample

Free radical polymerization commonly results a DC% of 50-60%, if the irradiation energy has been sufficient throughout the material. (Anusavice 1996) In case of bonding metal brackets with light-curing adhesives, propagation of irradiation energy, e.g. curing light, through the material is prevented. Therefore, the DC% remains relatively low, at the level of 35-45% (Eliades et al. 1995 (1), Shinya et al. 2009).

Incompletely polymerized resin composite may have adverse health-related effects. Bisphenol-A (BPA) that is commonly used in the monomers of dental resin composites, e.g. as precursor of bis-GMA, has been shown to have estrogene-related effects (Kloukos et al. 2013). Incomplete polymerization may result in leaching of residual monomers in the oral cavity (Eliades et al. 2011, Sunitha et al. 2011).

Although bis-GMA is not hydrolyzed spontaneously or by enzymatic activity to any estrogen-like substance, some other monomers after being hydrolyzed, such as monomers of bis-DMA, may be enzymatically hydrolyzed to bisphenol-A, which have shown estrogenic effects (Fleisch et al. 2010).

2.2.5 Light curing of resin dental systems

In order to polymerize light activated dental resin composite, electromagnetic energy is required. Energy is brought in the composite within the photons that are emitted from the light source of the curing device. Photons deliver the energy required for activating the free radicals via photoinitiator molecules. Electromagnetic energy is sinusoidal and travels at the speed of light. Frequency of radiation equals to the number of complete sinusoidal waves traversing through a set distance, and the wavelength equals to the physical length of each complete wave. Association between electromagnetic energy and wavelength of different type of radiation is shown in Figure 5. (Rueggeberg et al. 2017)

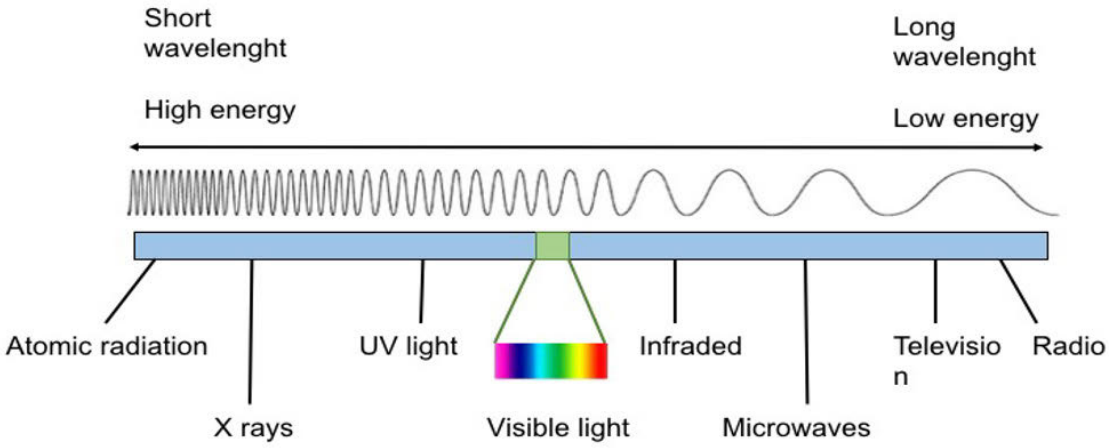

Figure 5. Electromagnetic spectrum.

In dental light curing units (LCUs), the wavelength of the emitted light is within visible light spectrum, usually 400–500 nanometers. Absorption of photons to the resin rises the photoinitiator molecules (CQ) to an excited stage and initiates the tertiary amine system to form free radicals (Chen et al. 2007). Light-emitting diodes (LEDs) came available at late 1990's and they have largely replaced halogen lamps in dental LCUs. In the LED technique, doped semiconductors produce narrow spectrum of light (400–500 nm) which the initiator molecules absorb (Dunn and Taloumis 2002, Desmet et al. 2006).

An improper light curing may result in weak bond strength and incomplete polymerization of the resin (Mutluay et al. 2014, Price et al. 2015). In good dental

practice, the light curing tip should be placed as close as possible to the composite surface. However, this may not always be possible, e.g. when curing adhesive under metal bracket or in deep cavity. Beolchi et al. (2015) showed that the output densities of various LCUs varied widely, and the irradiance power was significantly diminished when the distance between the material surface and the light curing tip increased.

The level of irradiance required for proper curing depends on characteristics the composite, e.g. on the number of fillers, shade and thickness of the material (Rueggeberg et al. 1994). The minimum energy density required to polymerize 2 mm thick composite is 12–16 J/cm^2 (Peutzfeldt and Asmussen 2005, Gritsch et al. 2008, Beolchi et al. 2015). The energy is influenced by the irradiation time and the output power of the LCU. For example, irradiation of 20 s with a light intensity of 800 mW/cm^2 results in energy level of 16 J/cm^2. By prolonging the curing time, higher energy density can be achieved. With modern LCUs curing times from 10 s to 40 s are recommended, depending on material thickness and properties. Irradiation produces heat and with longer curing times, risk of thermal pulp damage must be taken into consideration. Zach and Cohen (1965) found that a rise in pulp temperature of 5.5 °C induced pulp necrosis in 15% of the teeth of rhesus-monkeys. However, there is no agreement on the limit of temperature rise that will cause irreversible pulp damage. (Rueggeberg et al. 2017)

3 Aims

This study investigated behavior of light cured adhesive resins in bonding of orthodontic brackets and factors, which have been suggested to influence the curing and related properties of the bonded brackets.

The specific aims and working hypothesis were:

1. To investigate the degree of conversion (DC%) under the orthodontic brackets, and to study the attenuation of light in the adhesive resin with and without high aspect ratio glass filler. The hypothesis was that glass fillers improve the curing of the adhesive resin.
2. To measure optical properties of enamel and dentin, particularly how the layer thickness, level of hydration, and the presence of a smear-layer affect attenuation of light. Working hypothesis was that the light attenuation follows the Beer-Lambert law, and that a level of low hydration and presence of smear layer decrease light transmission.
3. To compare bond strength of metal brackets when bonded with conventional methods and with transillumination technique. The hypothesis was that conventional light curing results in higher bond strengths.
4. To measure the DC% of adhesive under ceramic and plastic brackets after curing through the transparent brackets. Hypothesis was that direct curing through the ceramic brackets provides a high degree of conversion.

4 Materials and Methods

Table 1. Materials used in studies I–IV.

MATERIAL	MANUFACTURER	COMPOSITION	STUDY
Transbond™ XT	3M Unitek (Monrovia, CA, USA)	Bis-GMA TEGDMA EBPADMA Silane treated quartz Silane treated silica Diphnyliodonium Hexafluorophosphate	I, III, IV
Transbond XT primer	3M Unitek (Monrovia, CA, USA)	Bis-GMA TEGDMA Triphenylantimony 4-(dimethylamino)-benzeneeethanol DL-camphorquinone Hydroquinone	III
Enlight	Ormo (Orange, CA, USA)	Bis-GMA 3-trimethoxysilylpropyl methacrylate	I
everStick® NET	Stick Tech Ltd. (Turku, Finland)	Bis-GMA, PMMA, glass-fiber	I
Ortomat Mini-Mat Standard brackets	Ortomat Herpola, Scafati, Italy	Stainless steel	I, III, IV
File Eze®	Ultradent Products, Inc., South Jordan	19,5% ethylenediaminetetraacetic	II, III
Scotchbond™ Universal Etchant 32%	3M ESPE (Deutschland, Neuss, Germany)	Water Phosphoric acid Polyvinyl alcohol	III

Bis-GMA indicates bisphenol-A glycidyl methacrylate, TEGDMA triethylene glycol dimethacrylate, EBPADMA bisphenol-A-bis(2-hydroxyethyl ether) dimethacrylate

The materials used to fabricate the specimens in studies I-IV are listed in Table 1. Brackets used in the study IV are listed in Table 4.

4.1 Preparation of test specimens

4.1.1 Brackets and matrices (I)

A stainless steel bracket was measured and pieces of metal matrices were hand cut into four different sizes, M0 corresponding the size of the bracket, M0.5 being 0.5 mm, M1 1.0 mm, and M2 2.0 mm larger in length and width. The exact dimensions of each specimen are given in Table 2. The M0-group was added to ensure that the metal matrices acted in similar manner as the brackets.

Table 2. Dimensions of the bracket (br) and the light shielding matrices (M0, M0.5 M1 and M2). From original publication I.

CODE	BR	M0	M0.5	M1	M2
Dimensions (mm)	3.1x4.2	3.1x4.2	4.1x5.2	5.1x6.2	7.1x8.2

4.1.2 Tooth specimens (I, II, III)

The teeth used in the tests were extracted incisors, premolars and third molars, collected from the Dental Teaching Clinic, Oral and Dental Health Care of the City of Turku, and stored in Chloramine-T after extraction. They were sound without visually detectable cracks, caries or fillings.

To study light curing by transillumination through dental hard tissues of different thickness, the third molars were cut with a histological saw (Secotom-50, Struers A/S, Ballerup, Denmark) into vertical slices with a thicknesses of 0.5 mm, 1.0 mm, 1.5 mm, 2.0 mm, 3.0 mm or 4.0 mm, five each. After cutting, the specimens were stored refrigerated in distilled water. In addition to enamel and dentin, the specimens contained remnants of pulp tissue.

Furthermore, forty third molars were cut into slices vertically, in buccolingual direction, each to thickness of 1 mm, resulting in 20 specimens containing only enamel and 20 specimens containing only dentin, without visible remnants of other tissues. The cutting was performed with a histological saw (Secotom-50, Struers A/S, Ballerup, Denmark) and the specimens were finished on a polishing machine (LaboPol-1, Struers A/S, Ballerup, Denmark) with a 500 grit-SiC-paper. The final test specimens were round in shape with an average diameter of 5.5 mm (standard deviation 0.58). Before use, the specimens were stored in oil-free distilled water. Four experimental groups for both enamel and dentin were created to study the effect of increased layer thickness to transmitting light (1 mm, 2 mm, 3 mm and 4 mm, n=5 in each group). Light transmittance of the groups, both moist and air-dried, was

measured. Thereafter, the specimens were treated with 19.5% EDTA (ethylenediaminetetraacetic acid) for 1 minute on both sides in order to remove the smear layer and expose dentin tubules and enamel rods. After EDTA treatment, the measuring of light transmittance was repeated using both moist and air-dried specimens for 1 mm layer thickness only.

To study the DC% after transillumination through various layer thicknesses of dental hard tissues, a total of 60 extracted sound human third molars were cut into 1 mm thick enamel and dentin slices, 20 each. Tooth slices were prepared as described earlier in the present chapter and the thickness of the slices was ensured with a digital caliper with accuracy of ± 0.02 mm. All the slices were treated with 19.5% EDTA for 1 minute from both sides and carefully rinsed with tap water.

4.1.3 Bonding protocol (III)

Bracket debonding was studied using extracted human incisors and premolars, embedded inside acrylic cylinders with the labial surface (bonding surface) perpendicular to the longitudinal axis of the cylinder. Enamel surface was etched with 32% phosphoric acid for 30 s, rinsed for 15 s with oil-free tap water and dried. Primer was applied according to the manufacturer's instructions. Adhesive was applied to the back of the bracket and the bracket was pressed firmly onto the labial surface of the teeth. Excess adhesive was removed and light curing was carried out with hand held LCU. Four study groups were formed: metal bracket bonded to incisor 1) with the conventional curing method (Group 6, n=10) or 2) by transillumination (Group 7, n=10), and metal bracket bonded to premolar 3) with the conventional curing method (Group 8, n=6) or by transillumination (Group 9, n=6).

In the groups 6 and 8, the adhesive was cured 20 s from the mesial and distal sides of the bracket, and in the groups 7 and 8, the adhesive was cured for 40 s through the tooth (transillumination). After bonding, the teeth were stored in distilled water at 37°C in the dark incubator for 24 h before measuring the debonding force.

4.2 Analyses

4.2.1 Determination of DC% under metal brackets

The degree of cure (DC%) was measured with FT-IR (Frontier™ FT-IR, PerkinElmer®, Beaconsfield Bucks, UK) using the ATR sampling accessory and the Spectrum™-program (v. 10.4.2, PerkinElmer®). To analyze DC% against time, the first scanning was performed before curing, the second immediately after curing, and the following scans every three minutes up to 15 minutes after curing.

Before measuring the DC% of the experimental groups, DC% of the adhesive without a shielding bracket or metal plate was measured as a control. A small amount of adhesive (thickness of 1 mm) was placed on the sensor and cured for 40 seconds with a hand held LCU (LED, Elipar™ S10, 3M ESPE, St. Paul, MN, USA) using an output power of 1880 mW/cm^2.

The DC% of the experimental groups was then measured with and without the glass-fiber weave. The specimen was structured similar with the study of Shinya et al. (2009) so that first a small amount of adhesive was placed on the ATR sensor, and then bracket/matrix was firmly placed on the adhesive and excess adhesive was carefully removed. Adhesive was cured 20 seconds from the sides of the bracket.

After measuring the DC% of the adhesive in the experimental groups, a glass-fiber weave was added. A small amount of adhesive was placed on the sensor, a hand-cut piece of glass-fiber weave was placed on top of the adhesive and a small amount of adhesive was placed on the bottom of the bracket. The bracket was firmly placed on top of the adhesive and glass-fiber weave, excess adhesive was removed and the adhesive was cured for 20 s from both sides of the bracket (Figure 6).

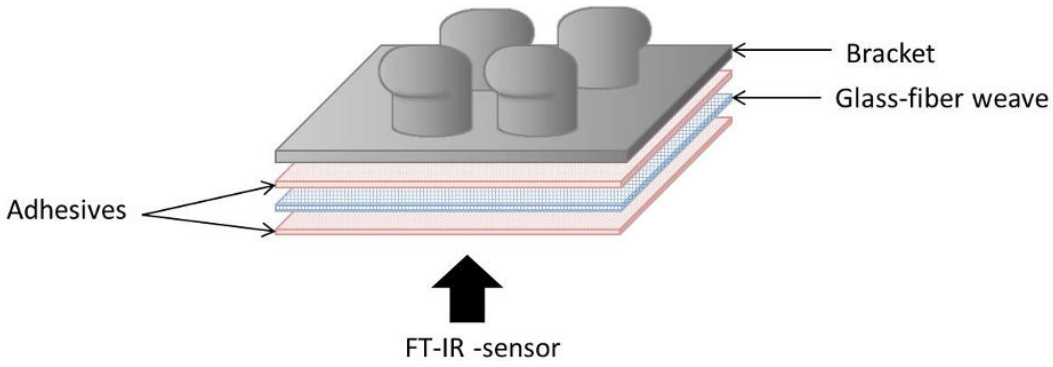

Figure 6. A schematic drawing of a bracket with glass-fiber weave. FT-IR sensor was used to measure the DC% of the adhesive resin. From original publication I.

4.2.1.1 Determination of DC% of the adhesive after curing with transillumination (III)

The DC% of a light curing adhesive (Transbond™ XT), was measured after it was cured through slices of dentin and enamel, and through the entire tooth. Five experimental groups, differing in composition and thickness (Table 3), were formed to measure the light curing efficacy through dental hard tissues. In the control group (Group 1) the adhesive was directly cured without any solid material between the LCU tip and the adhesive. Groups 2 and 3 were created to test the light attenuation through increasing thickness of dental hard tissues and to simulate light attenuation through a natural tooth. In groups 4 and 5, sound incisors and premolars were placed

between the LCU tip and the adhesive with the labial surface of the tooth specimen facing the adhesive and the FT-IR sensor.

Table 3. Group numbers and description used to study the DC% after light curing with transillumination through layers of dental hard tissues (III). Modified from original publication III.

GROUP NUMBER	DESCRIPTION	THICKNESS
1	control	No solid material between the light curing and the adhesive
2	Enamel-dentin-enamel	3 mm
3	Enamel-dentin-dentin-enamel	4 mm
4	Incisor	5.6 mm
5	Premolar	8.2 mm

Protecting molds were fabricated to keep the piled specimens together and prevent the light straying around the sample. Molds were made from Lab-Putty (Coltène/Whaledent AG, 9450 Switzerland) leaving round holes in the middle (Ø 6 mm). Similar molds were individually made to each tooth in groups 4 and 5.

A small amount of adhesive (Transbond™ XT) was applied on the FT-IR sensor, the putty mold was placed around the sensor, the specimen was placed inside the mold and firmly pressed against the FT-IR sensor. The same procedure was repeated with the incisors and premolars (groups 4 and 5). The DC% was measured before curing, immediately after curing and every 3 minutes up to 15 minutes from the curing. The LCU used in this study had an output power of 1960 mW/cm^2 (Elipar™ S10, 3M ESPE, St. Paul, MN, USA). All the groups 1–5 contained three subgroups with curing times of 20 s, 40 s and 60 s (n=5 in each). The enamel and dentin slices were randomly selected to each slice combination, and each enamel slice was used twice: first on the top of the combination and then on the bottom. Once the slice had become contaminated with the adhesive resin, it was no longer used.

4.2.1.2 Determination of DC% under ceramic and plastic brackets (IV)

Four tooth-colored brackets were chosen to study the DC% of the adhesive when the curing took place either through the bracket or from the sides. Brackets used in this study were ceramic Inspire Ice, Fascination and Ovation C, fiber-glass reinforced Elegance with a metal slot and stainless-steel bracket Ortomat Mini-Mat (Table 4). All the brackets were maxillary incisor brackets.

Table 4. Brackets used in the study (IV).

BRAND	CODE	MANUFACTURER	COMPOSITION	STRUCTURE
Inspire ICE	ICE	Ormco, Orange, CA, USA	Ceramic (aluminum oxide)	Monocrystalline
Fascination	FC	Dentaurum, Ispringen, Germany	Ceramic (aluminum oxide)	Polycrystalline
Ovation C	OV	Dentsply Sirona, Philadelphia, USA	Ceramic (aluminum oxide)	Polycrystalline
Elegance	EL	Dentaurum, Ispringen, Germany	Glass-fiber reinforced polycarbonate with metal slot	
Mini-Mat Ortomat	SS	Ortomat Herpola, Scafati, Italy	Stainless steel	

Although the curing times of Transbond™ XT, recommended by the manufacturer, are 3 seconds from both the mesial and distal side of a metal bracket and 3 seconds through a ceramic/plastic bracket, longer curing times were used in this study to make the results better comparable with the existing literature.

Each bracket-group was divided into two subgroups: 1) light curing for 10 seconds from the sides of the bracket and 2) light curing for 10 seconds through the bracket (n=6 in each). A small amount of adhesive (Transbond™ XT) was placed on the sensor, the bracket was pressed on top of the adhesive and any excess adhesive was removed before light curing. The first scan was performed before curing, the second immediately after curing and following scans every three minutes up to 15 minutes after curing.

4.3 Light transmission

4.3.1 Light transmission through dental hard tissues (I, II, III)

Transmittance of light was measured with MARC®-spectrometer and analyzed with BlueLight®- program (MARC® Resin Calibrator, BlueLight® analytics inc., Halifax, Nova Scotia, Canada). A dentin specimen was placed on the sensor with the pulpal cavity included within specimen with thickness of 3.0 mm and 4.0, facing downwards and the light curing tip was placed on the specimen (Figure 7).

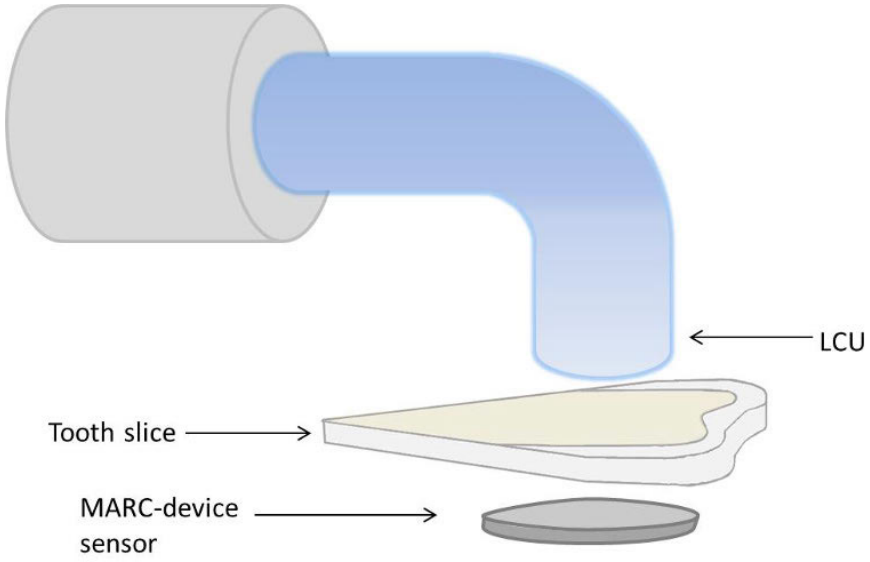

Figure 7. A Schematic drawing of measuring the irradiance power through a slice of tooth. From original publication I.

Transmittance of light through slices of enamel and dentin with thickness of 1 mm, 2 mm, 3 mm and 4 mm were measured, both as moist and air-dried. In addition, 1 mm thick slices of EDTA treated enamel and dentin were measured for light attenuation, both as moist and air-dried (Table 1). The effect of increasing thickness was studied by piling randomly selected specimens on the sensor. The LCU tip was held as close as possible to the specimen and the transmitted irradiance was measured as explained above. The maximum power output of the light curing unit (LED, Elipar™ S10, 3M ESPE, St. Paul, MN, USA) used in the study was 1869 mW/cm^2, with a wavelength of 455 nm ± 10, when the LCU was set tightly against the sensor.

In addition, light transmittance through extracted incisors (n=10) and premolars (n=6) was measured. Each tooth was placed with the labial surface facing the sensor and the LCU tip facing the lingual surface of the tooth. Thickness of tooth crown was measured perpendicular to the longitudinal axis of the tooth with a digital slide gauge (Vernier, Millikan Way, Beaverton), with an accuracy of 0.02 mm. Standardized distance from the light curing tip to the sensor was 7 mm for the incisors and 10 mm for the premolars.

Furthermore, transmission of light through dental hard tissues and entire teeth (Groups 2–5, Table 3) was measured with MARC® spectrometer and analysed using BlueLight® program following the same protocol as explained above. Prefabricated molds were used when light transmittance was measured to inhibit the light stary around the sample to the sensor. Maximum power output of the LCU used in the ´study was 1960 mW/cm^2 (Elipar™ S10, 3M ESPE, St. Paul, MN, USA).

4.3.2 Light transmission through brackets (IV)

Transmission of light through brackets shown in Table 4 was measured using a MARC® spectrometer and the data was analysed with BlueLight® program. Brackets were placed on the sensor of the spectrometer with the base facing the sensor and the tip of the LCU on top of the ligature wings. The number of specimens in each group was 6. The hand held LCU used in this study had an output power of 1850 mW/cm^2 (Elipar^{TM} S10, 3M ESPE, St. Paul, MN, USA).

4.4 Mechanical test

4.4.1 Debonding of orthodontic bracket (III)

Debonding force was measured with universal testing machine (LLOYD Instruments LR30K plus, Ametek Inc., Berwyn, US) from incisal to apical direction, with a crosshead speed of 1.0 mm/min. The loading tip was positioned as close as possible to the enamel surface and enamel-bracket junction causing predominantly shear type of stress at the interface. The sensor used was 2500 N, and the load was recorded in newtons (N). The influence of the surface area was ignored.

4.4.1.1 Adhesive remnant index (ARI)

Fracture analysis was carried out after every bracket debonding. Adhesive remnant index (ARI) was scored with a USB microscope (eScope, Oriental Inspiration Limited, Hongkong, China) immediately after the bracket failure. ARI was scored as follows: 0 = no adhesive on the tooth, 1 = less than ½ of the adhesive on the tooth, 2 = more than ½ of the adhesive on the tooth, 3 = all of the adhesive on the tooth and 4 = enamel fracture.

4.4.2 Surface microhardness (IV)

The specimens were tested for the surface microhardness of the adhesive (Vickers hardness number (VHN) Struers, Duramin) immediately after taking the last DC% measurement, 15 minutes after the initial curing. The bracket and polymerized adhesive were gently removed from the sensor with the adhesive surface remaining intact and were examined for the surface microhardness (Vickers hardness number (VHN), Struers, Duramin). A VHN intender produced a rectangular shape that was recorded with x10 or x40 light microscope and measured. The surface hardness was determined by dividing the press load (press load 1.96 N with a pressing time of 10 s) with the measured area of indentation. For each sample 4 indentations were

performed, with two indentations from the sides of the bracket and two from the center of the bracket.

4.5 Statistical analyses

The data of the DC% of the adhesive under metal brackets and matrices with various sizes used with glass-fiber weave (study I) was analyzed with a three-way-analysis of variance (ANOVA) and Tukey's HSD test. The normally distributed data of the DC% after transillumination (study III), bracket debonding (study III) and DC% under ceramic brackets (study IV) were analysed with a two-way analysis of variance (ANOVA) and Tukey's post hoc test. The correlation between surface microhardness and the DC% under ceramic brackets (study IV) was analyzed with linear regression analysis.

The normally distributed data of light transmission through dentin and enamel slices of various thicknesses (study II) was analysed by using Pearson correlation coefficient, a two-way ANOVA and Tukey's post hoc test. The data of light transmittance through tooth slices of 3 mm and 4 mm (study I) was not normally distributed and was therefore analyzed with a Kruskall-Wallis test and regression curve estimation according to the thickness of the specimen discs. The limit of statistical significance was set to 0.5.

5 Results

5.1 DC% of the orthodontic adhesive

5.1.1 DC% under metal brackets with and without glass-fiber net (I)

DC% under metal brackets and matrices remained lower compared to the control group with direct light curing of the adhesive. Adding a glass-fiber weave between the bracket and the adhesive increased the DC% in all groups (Table 5).

Table 5. Mean degree of cure (DC%) and standard deviation (SD) of adhesives Enlight and Transbond™ and standard deviation at the 15 min time point with and without the glass fiber weave. From original publication I.

	ENLIGHT			TRANSBOND™		
	No glass fiber weave	With glass fiber weave	Difference	No glass fiber weave	With glass fiber weave	Difference
CONTROL	58.3 (1.0)			46.5 (1.4)		
BR	39.7 (2.8)	50.2 (2.3)	10.5	32.7 (2.3)	39.6 (1.4)	6.8
M0	43.6 (2.6)	47.9 (2.9)	4.3	37.0 (1.4)	40.9 (2.1)	3.9
M0.5	12.6 (6.4)	43.8 (3.5)	31.2	30.8 (1.0)	38.8 (1.4)	7.9
M1	8.1 (9.0)	38.2 (6.3)	30.1	14.4 (3.8)	33.1 (1.7)	18.7
M2	0.1 (0.9)	7.7 (10.0)	7.6	2.2 (3.1)	10.4 (11.9)	8.2

The three-way ANOVA revealed a significant difference in DC% at 15 minutes time point in all the factors and their interactions (adhesive, glass-fiber weave, matrix/bracket size, Table 6). Tukey's post hoc test revealed a statistically significant difference between all the other groups ($p<0.05$) except in BR- and M0-groups which did not differ each other significantly ($p<0.05$).

Table 6. Results of the three-way ANOVA on the effects of the adhesive, glass fiber weave and bracket/matrix group on DC%. Large F and small p indicate statistical significance. From original publication I.

	F	p
ADHESIVE	1.49	0.226
GLASS FIBER WEAVE	171.02	<0.001
BRACKET/MATRIX	188.23	<0.001
ADHESIVE*GLASS FIBER WEAVE	14.95	<0.001
ADHESIVE*BRACKET/MATRIX	8.53	<0.001
GLASS FIBER WEAVE*BRACKET/MATRIX	15.09	<0.001
ADHESIVE*GLASS FIBER WEAVE*BRACKET/MATRIX	5.06	0.001

5.1.2 DC% of the adhesive cured with transillumination through dentin and enamel (III)

The mean values of DC% are presented in Table 7. Tukey's post hoc test revealed that there was no statistical difference between groups 3 and 4.

Table 7. The degree of monomer conversion (DC%). Vertical superscript letters describe statistical difference between the groups with same curing time (20, 40 and 60 s). Horizontal lowercase letters describe statistical differences between different curing times among the groups (1-5). Modified from original publication III.

GROUP CODE	20 S	40 S	60 S
CONTROL (GROUP 1)	48.2 (0.3) $_{a}^{A}$	48.5 (0.9) $_{a}^{A}$	49.3 (0.5) $_{a}^{A}$
ENAMEL-DENTIN-ENAMEL (GROUP 2)	40.8 (0.9) $_{a}^{B}$	44.0 (0.9) $_{b}^{B}$	45.2 (1.9) $_{b}^{B}$
ENAMEL-DENTIN-DENTIN-ENAMEL (GROUP 3)	33.7 (2.8) $_{a}^{C}$	40.2 (1.8) $_{b}^{C}$	41.8 (2.5) $_{b}^{C}$
INCISORS (GROUP 4)	31.4 (7.0) $_{a}^{C}$	39.4 (2.9) $_{b}^{C}$	41.8 (2.9) $_{b}^{C}$
PREMOLARS (GROUP 5)	20.9 (6.7) $_{a}^{D}$	28.8 (3.0) $_{b}^{D}$	33.9 (1.3) $_{c}^{D}$

In all experimental groups the DC% increased significantly when the curing time was increased from 20 seconds to 40 seconds ($p<0.05$). In groups 2, 3 and 4 no significant difference was detected between curing times of 40 s and 60 s. Only in group 5, prolonging the curing time from 40 s to 60 s resulted in a significant increase in the DC% ($p<0.005$).

5.1.3 The DC% of the adhesive under ceramic and plastic brackets (IV)

A two-way ANOVA revealed no statistical difference in the DC% between the groups ICE, OV and FC when the light curing was executed through the bracket but EL-group had a significantly higher DC% ($p<0.05$) (Table 8). The tooth-colored brackets did not show significant differences in the DC% when the light curing was performed from the sides of the bracket. With the exception of the SS-group, curing through the brackets resulted significantly higher DC% than curing from the sides of the bracket.

Table 8. The DC% and standard deviations after curing directly through the bracket or indirectly from the sides. Vertical superscript letters describe the statistical differences between the groups and the horizontal lowercase letters describe the statistical difference between the curing direction among the groups.

BRACKET LABEL	DIRECT CURING	INDIRECT CURING	DIFFERENCE
INSPIRE ICE	48.3 (0.8)$^{A}_{a}$	34.2 (3.8)$^{A}_{b}$	14.1
FASCINATION	49.1 (0.4)$^{A}_{a}$	36.4 (1.6)$^{A}_{b}$	12.7
OVATION C	47.9 (0.8)$^{A}_{a}$	34.6 (2.0)$^{A}_{b}$	11.5
ELEGANCE	52.0 (5.0)$^{B}_{a}$	33.8 (1.7)$^{A}_{b}$	18.2
STAINLESS STEEL	28.9 (3.8)$^{C}_{a}$	29.9 (5.5)$^{B}_{a}$	1

5.2 Light transmission

5.2.1 Light transmission through dentin and enamel (I, II, III)

The transmittance of the curing light decreased as the specimen thickness increased. With a slice thickness of 0.5 mm the transmitting light intensity was 445.8 mW/cm^2 (SD 72.8), with 1 mm thickness 320 mW/cm^2 (SD 65.9), with 1.5 mm 203.2 mW/cm^2, with 2 mm 85.8 mW/cm^2 (SD 20.2), with 3 mm 21.4 mW/cm^2 (SD 8.6) and with 4 mm 0.0 mW/cm^2 (SD 0.0), respectively (Figure 8). The differences between the groups (0.5 mm to 4 mm) were statistically significant ($p<0.001$).

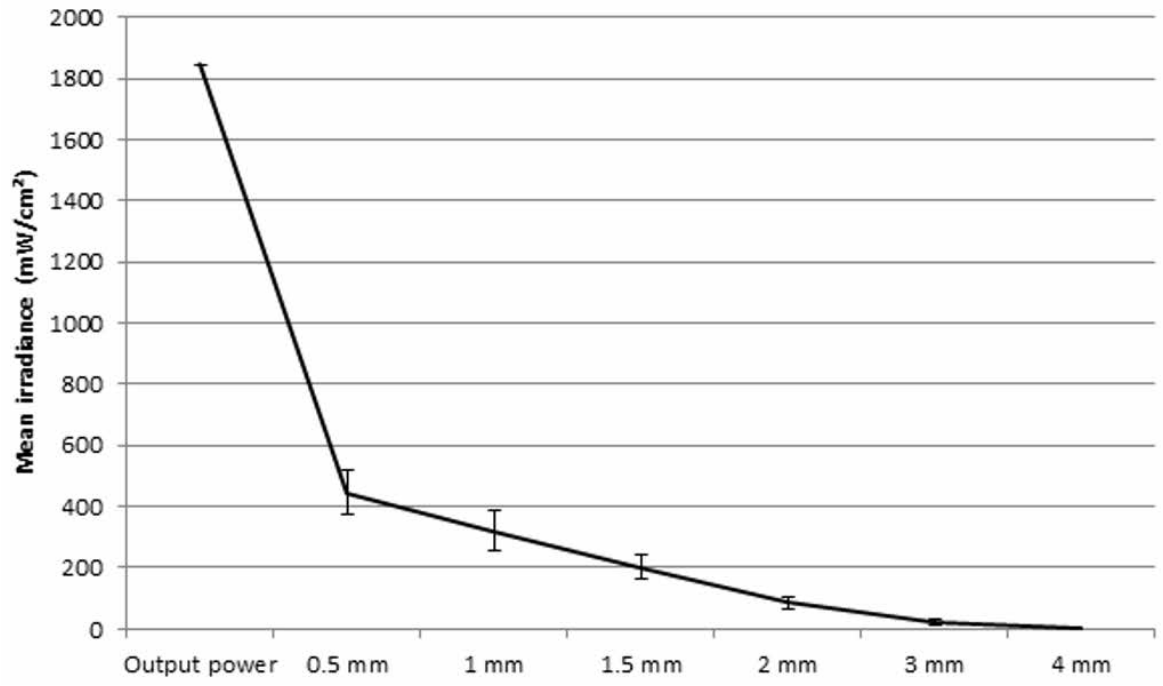

Figure 8. Mean irradiance (mW/cm^2) through dental slices of different thickness. From original publication I.

In the light transmission through slices of dentin and enamel, the two-way ANOVA showed a significant difference between enamel and dentin group ($p<0.005$) and between moist and air-dried specimens ($p<0.05$). Mean irradiances are shown in Figures 9 and 10, and the values are presented in Table 9.

Table 9. The average mean irradiances and standard deviations (mW/cm^2) of each group (n=5) (II). Superscript letters describe statistically significant difference between the groups. From original publication II.

	ENAMEL		**DENTIN**	
SPECIMEN THICKNESS	Moist	Air-dried	Moist	Air-dried
1 mm	500.6 (58.0)^{A,a}	389.0 (49.9)^{B,a}	398.2 (38.4)^{C,a}	251.0 (66.7)^{D,a}
2 mm	209.2 (11.6)^{A,b}	117.0 (24.4)^{B,b}	164.8 (21.6)^{C,b}	49.4 (16.8)^{D,b}
3 mm	92.0 (21.8)^{A,c}	36.0 (5.5)^{B,c}	51.8 (18.5)^{C,c}	15.4 (6.6)^{D,c}
4 mm	40.8 (9.7)^{A,d}	12.8 (1.9)^{B,d}	19.4 (3.1)^{C,d}	0.0 (0.0)^{D,d}

The increase of specimen thickness significantly decreased the transmitting irradiance (mW/cm^2) ($p<0.005$) and when the thickness of the air-dried dentin specimen reached 4 mm, no light transmission was detected.

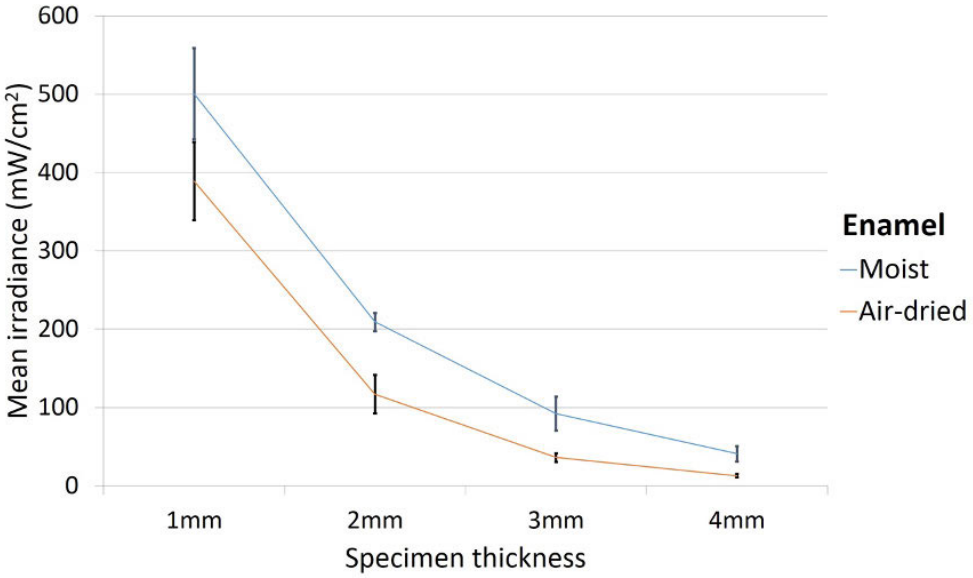

Figure 9. Mean irradiances (mW/cm^2) through enamel specimens of different thicknesses. From original publication II.

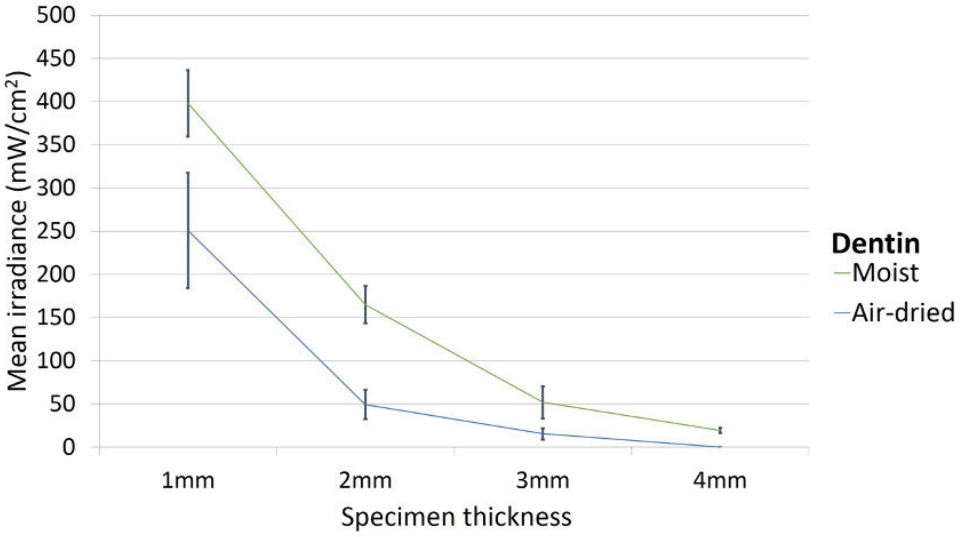

Figure 10. Mean irradiance (mW/cm^2) through dentin specimens of different thicknesses. From original publication II.

Transmission of curing light through a 1 mm thick test specimens after EDTA treatment was significantly higher ($p<0.05$) compared to a non-treated specimen in all groups excluding the group of moist enamel. However, due to large standard deviation, there was no statistical difference between enamel and dentin after EDTA treatment. Light transmission through EDTA treated enamel and dentin specimens, both moist and air-dried, are shown in Figure 11.

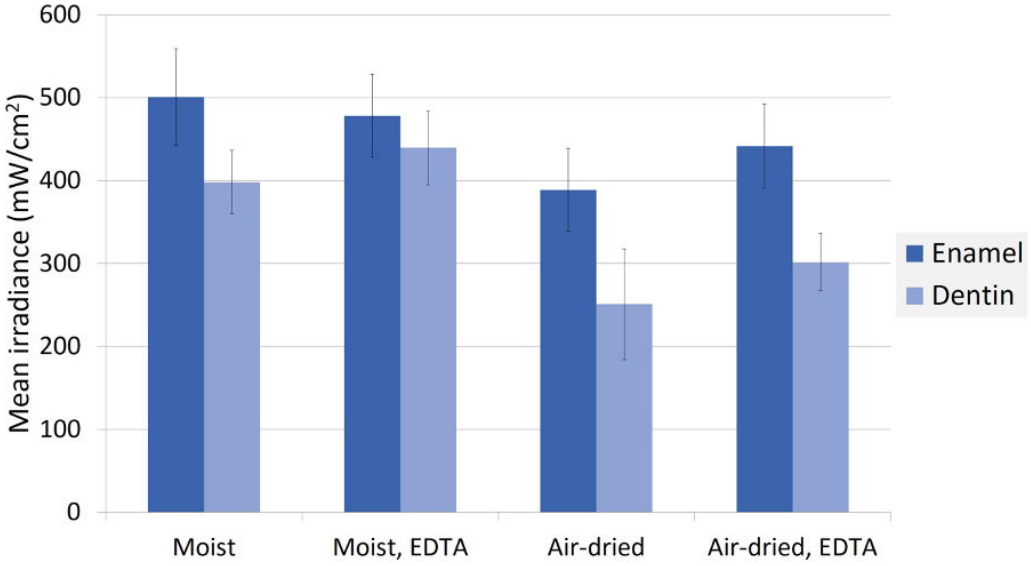

Figure 11. Mean irradiances (mW/cm^2) through 1 mm thick specimens before and after EDTA treatment both moist and air-dried. Vertical bars demonstrate standard deviation. From original publication II.

Light transmission through incisors was 37.6 mW/cm^2 (SD 26.6) and through premolars 6.2 mW/cm^2 (SD 6.9). In the premolars, majority of measurements were below the limit of detection, the highest detected transmitted value being 18 mW/cm^2. The average thicknesses of the incisors were 5.6 mm (SD 0.91) and of the premolars 8.2 mm (SD 0.37). The correlation between incisors and premolars was analyzed by regression analysis (Figure 12). The coefficient of determination was R^2=0.065 and the correlation coefficient was r=0.81 (P<0.001).

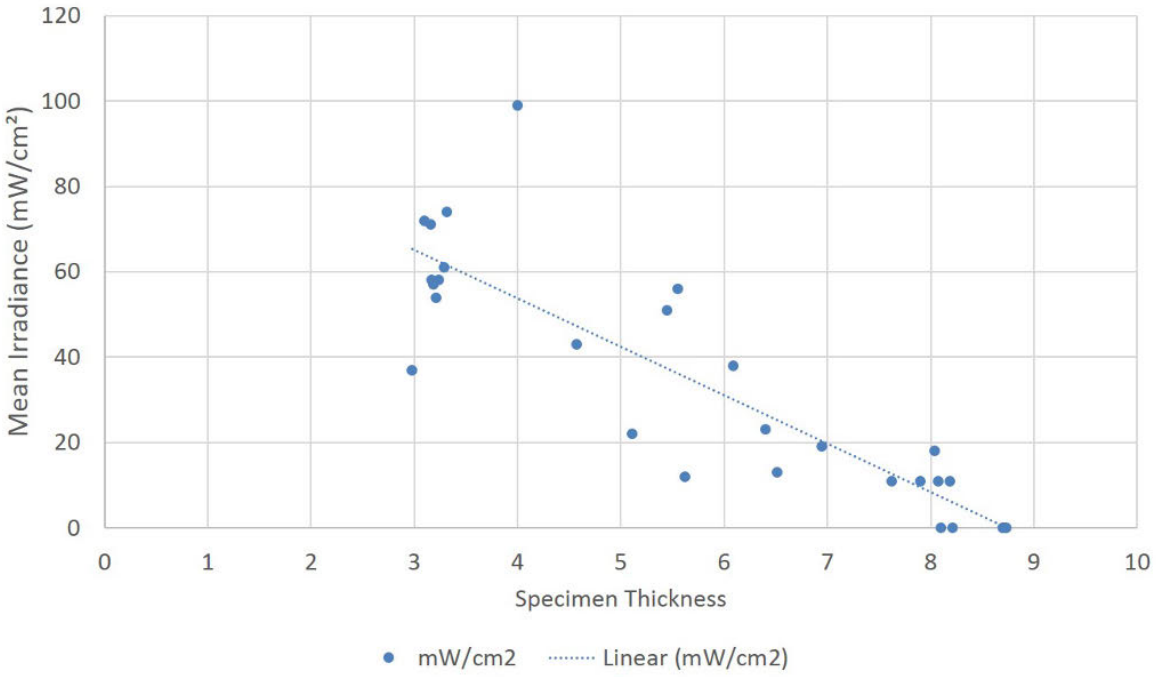

Figure 12. Mean irradiances (mW/cm^2) through extracted human incisors and premolars. From original publication II.

To test transmittance of light through a tooth *in vitro*, different combinations of enamel and dentin discs were used (groups 2–5). The results are shown in Table 10. Two-way ANOVA revealed a statistical difference in light transmission between the groups ($p<0.001$).

Table 10. Light transmission through 3 mm, 4 mm and extracted human incisors and premolars. Group codes are explained in Table 3. Modified from original publication III.

	MEAN (mW/cm²)	SD
LCU TIP FULLY AGAINST THE SENSOR	1958.4	32.4
ENAMEL-DENTIN-ENAMEL (GROUP 2)	91.0	16.5
ENAMEL-DENTIN-DENTIN-ENAMEL (GROUP 3)	22.2	3.0
INCISORS (GROUP 4)	37.6	26.6
PREMOLARS (GROUP 5)	6.2	6.9

5.2.2 Light transmission through ceramic and plastic brackets (IV)

Light transmission through the tested brackets are shown in Table 11. No statistically significant differences were detected between Elegance, Fascination and Inspire ICE whereas Ovation C differed significantly from Elegance, Fascination and Inspire ICE

($p<0.005$). As could be expected, light propagation by scattering along the metal bracket (SS) was significantly lower compared transmittance through transparent brackets ($p<0.005$). Inspire ICE transmitted the highest amount of light, 642.5 mW/cm^2, whereas the measured light intensity under metal bracket was only 86 mW/cm^2.

Table 11. Mean irradiances through the brackets and standard deviations. Vertical superscript letters describe statistical difference.

BRACKET LABEL	MEAN (mW/cm^2)	SD
INSPIRE ICE	642.5^A	41.4
FASCINATION	632.8^A	30.5
OVATION C	559.3^B	49.6
ELEGANCE	638.5^A	18.1
STAINLESS STEEL	86.0^C	4.4

5.3 Mechanical properties

5.3.1 Bracket debonding (III)

For conventionally cured incisor brackets (Group 6) the debonding force was 78.0 N and for those cured with transillumination (Group 7) 114.4 N. In premolars, the debonding force for conventionally cured bracket adhesive (Group 8) was 75.1 N and for that cured with transillumination (Group 9) 77.3 N. Debonding forces are shown in Figure 13. Analysis of variance revealed a statistically significant difference between groups 6 and 7 ($p<0.05$).

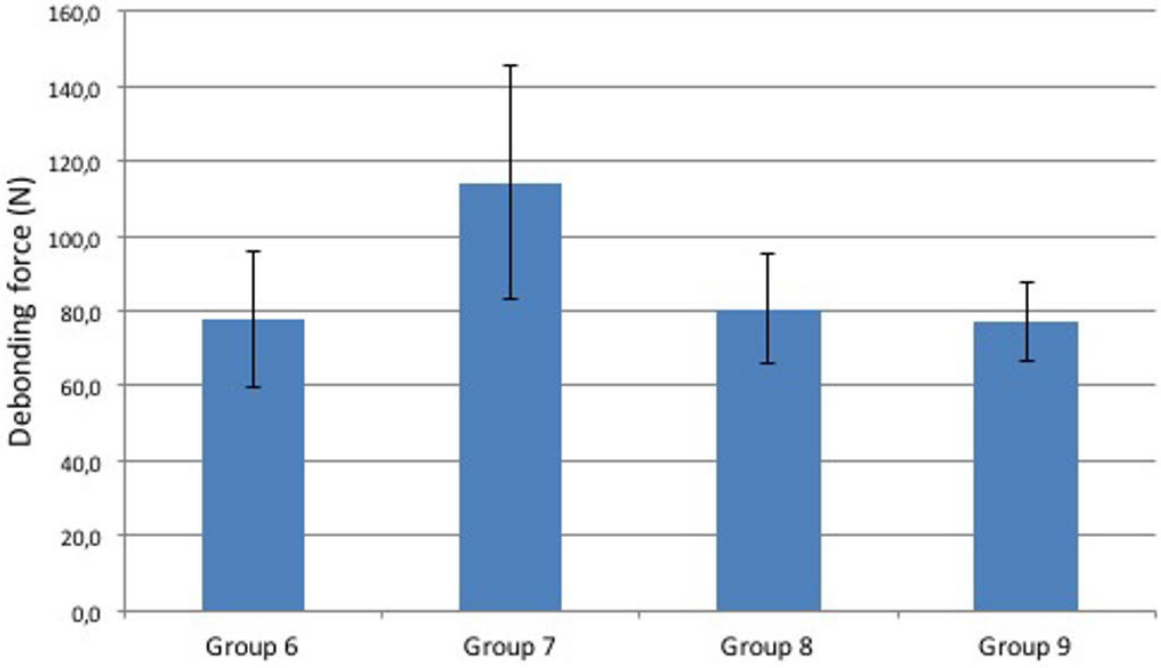

Figure 13. Debonding forces (N) for groups 6-9. Vertical bars represent standard deviation. From original publication III.

5.3.1.1 Adhesive remnant index (ARI)

Adhesive remnant index (ARI) scoring for the debonded brackets is shown in Figure 14. Stereomicroscope photos of enamel surfaces after debonding in groups 6–9 (III) are presented in Figures 15 and 16. In 48.1% of the cases, over ½ of the adhesive remained on the tooth surface and in 40.1% of the cases the adhesive remained entirely on the tooth surface. No case was detected with the adhesive completely removed with the bracket or fractured enamel.

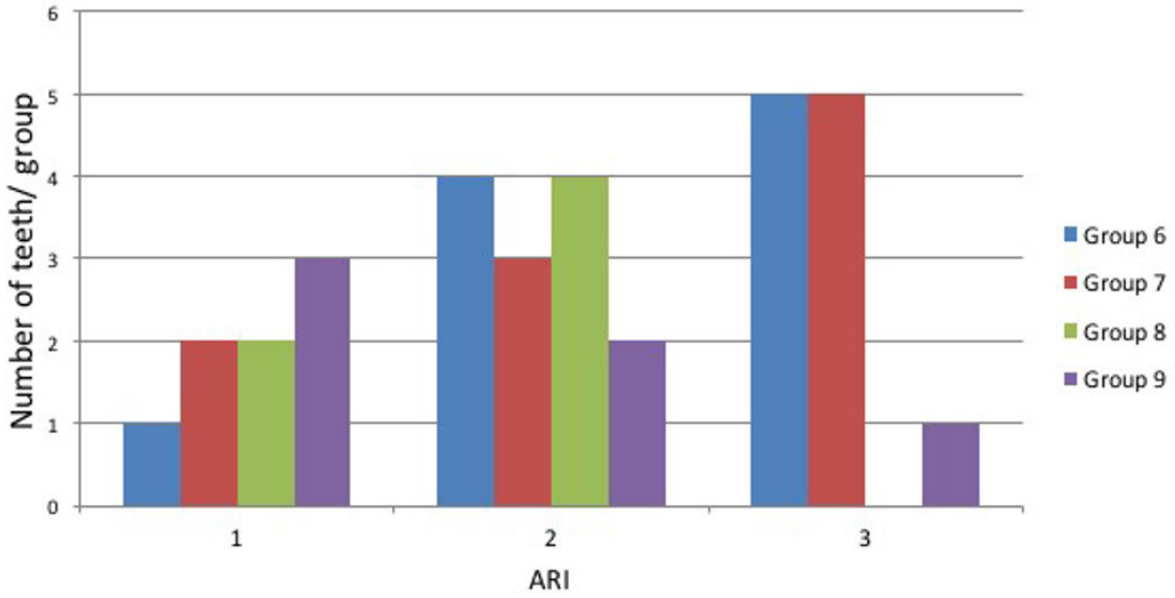

Figure 14. Adhesive remnant index (ARI) scoring for the groups 6-9. From original publication III.

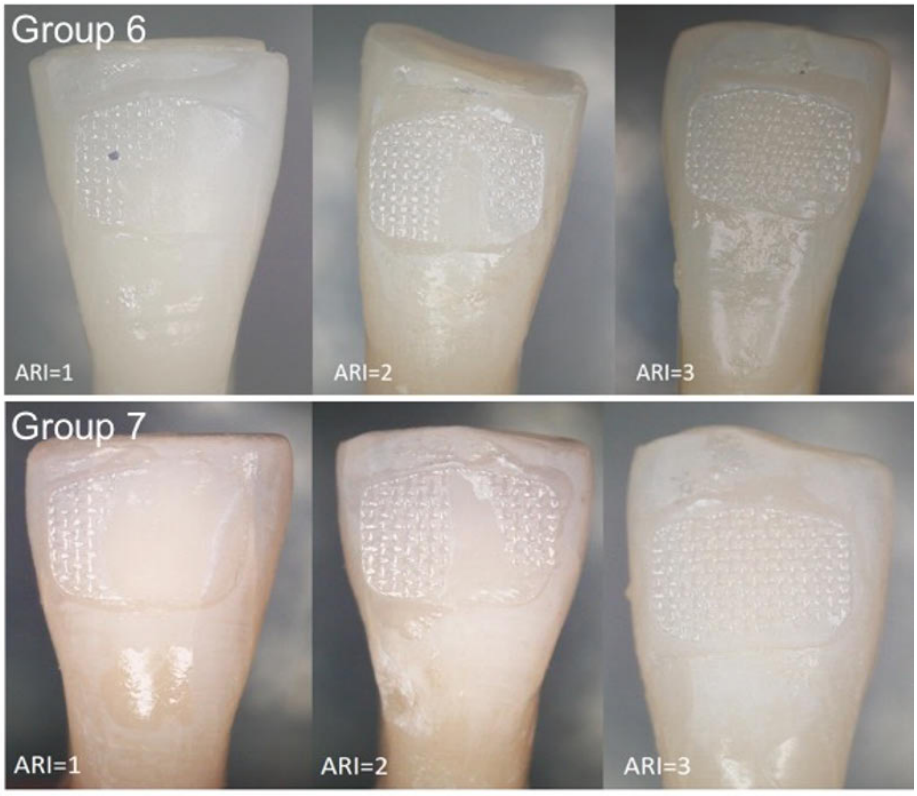

Figure 15. Stereomicroscope photos after bracket debonding and ARI scoring criteria. From original publication III.

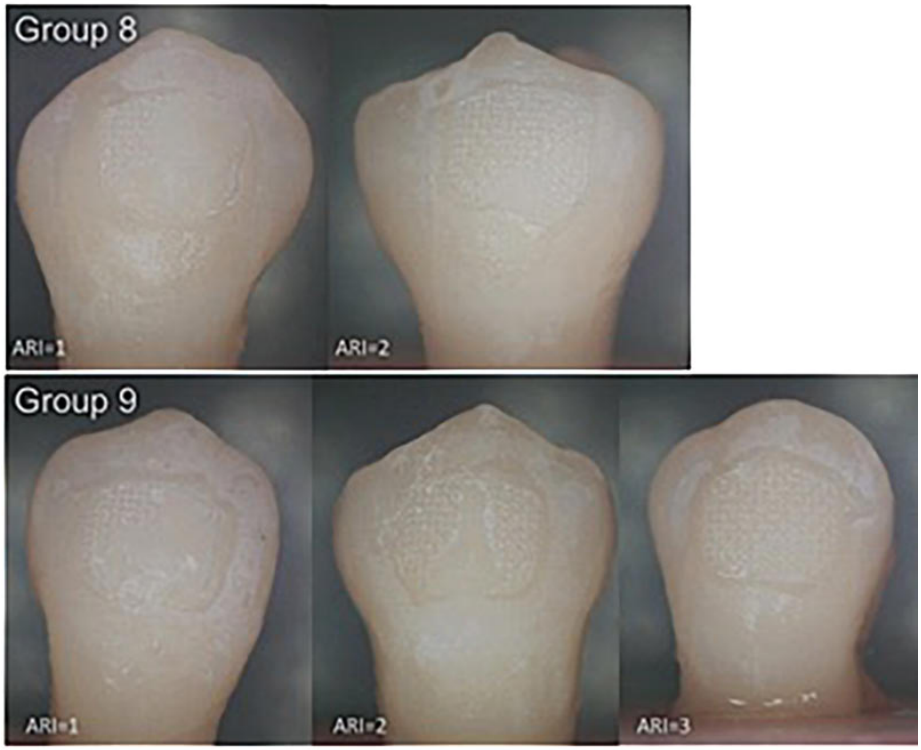

Figure 16. Stereomicroscope photos after bracket debonding and ARI scoring criteria. From original publication III.

5.3.2 Surface microhardness (IV)

Linear regression analysis revealed a statistically significant association in the light curing direction and surface microhardness of the adhesive at the center of the bracket ($p<0.05$). Curing through the bracket resulted in higher surface microhardness throughout the bracket area (both at the edge and center of the bracket) in other groups than SS-group ($P<0.05$). Mean microhardnesses measured from the edges and center of the bracket by light curing direction and groups are shown in Figure 17. The difference between the surface microhardness at the edges and at the center of the bracket within the same specimen was not however statistically significant in groups ICE, OV, FC and EL. However, in case of stainless steel brackets (SS-group) the center of the adhesive remained softer than the edges with both curing methods ($P<0.01$).

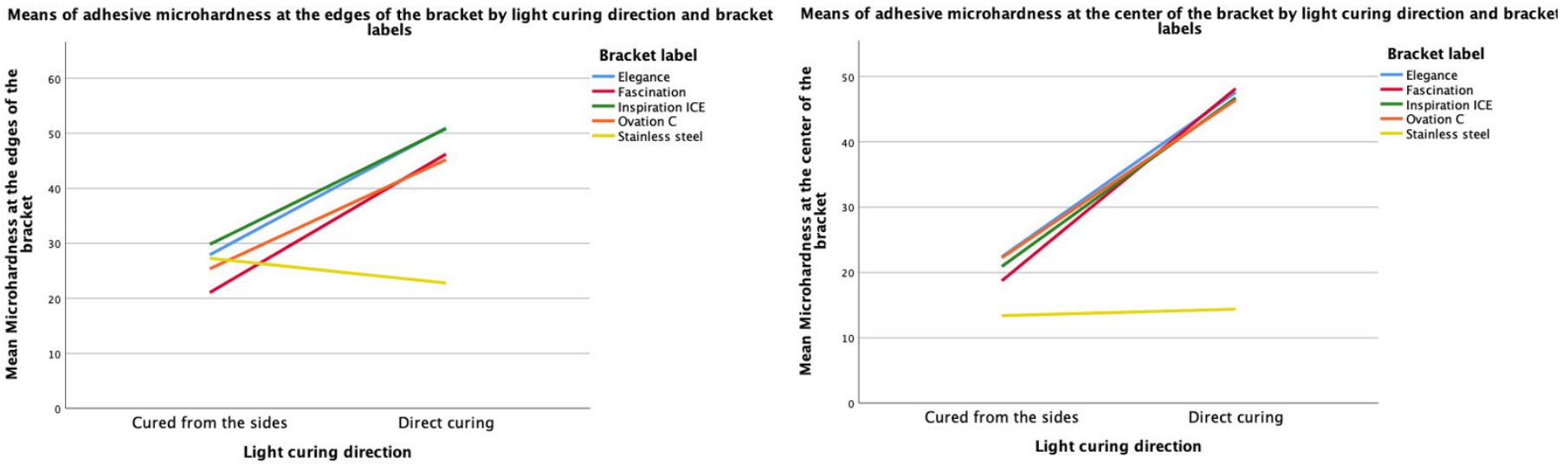

Figure 17. Mean surface microhardness of the adhesive by light curing direction and groups. Crossing lines indicate the significance of light curing direction between groups.

6 Discussion

6.1 Light curing of orthodontic adhesive

There has been a trend to increase the intensity of the LED curing units, even up to a power level of 3200 mW/cm^2. This has encouraged the adhesive manufacturers to shorten the recommended curing time to as low as 3 seconds per side (e.g. 3M Unitek Transbond^{TM} XT). Reduced curing times are in the interest of clinicians, since the bonding of the brackets is a time-consuming procedure, and a decrease in the curing time means shorter chair-time. In the present series of studies, LCUs were used with an average output power of 1800-1900 mW/cm^2, which is high compared to most of the previous studies. The "golden standard" of bracket bonding has been curing from the edges of the bracket for 20 seconds per side (Altmann et al. 2016). However, the technological development proceeds faster than research, and clinicians are left without firm evidence-based guidelines. Previous studies have shown that reducing the curing time down to 10 seconds per side can still provide satisfactory bond strengths (6-8 MPa) (Cerekja 2011, Lamper et al. 2012). On the other hand, a significant difference in bond strength was found between curing times 10 s and 40 s when using a high-intensity (1000 mW/cm^2) LED curing unit (Swanson et al. 2004). Furthermore, it has been shown that reduced curing time and increased light intensity resulted in lower DC% of the adhesive compared to situation where the same level of energy was received with longer curing time and lower LCU intensity (Amato et al. 2014). Previous studies suggest that a curing time of 3 seconds results in decreased bond strength (Cerekja and Cakirer 2011, Almeida et al. 2018).

6.2 Light transmission through dental hard tissues (I, II, III)

Light curing of orthodontic adhesive under a metal bracket is conventionally performed from the sides of the bracket. Several attempts have been made to improve the efficacy of light curing and enhance the conversion of the adhesive, e.g. testing of different adhesive compositions, adding glass-fiber weave under the bracket, and prolonging the curing time. In addition, transillumination has been suggested to be a potential method to increase the DC% under brackets (Tavas and Watts 1979, King

et al. 1987, Cheng et al. 1989). However, only few studies have investigated transmission of the curing light through dental hard tissues. Oesterle and Shellhart (2001) found that light transmittance through extracted human incisors with an average thickness of 4.21 ± 0.4 mm was only 4.0 ± 4.9 mW/cm^2 when the light intensity of the LCU was 450 mW/cm^2. In the present study (II), using a power level of 1870 mW/cm^2, transillumination through incisors resulted in light transmittance of 37.6 ± 26.6 mW/cm^2, and through premolars 6.2 ± 6.9 mW/cm^2 .

When light curing of the orthodontic adhesive is executed with transillumination, light propagates first through enamel, then through dentin and then again through the enamel on the other side of the tooth. The results of the present study showed that light transmission through enamel is significantly greater than through dentin. Enamel is more translucent than dentin (Brodbelt et al. 1980) and it allows more light to pass through. In the present study (II,) the light transmittance through enamel and dentin of thickness of 1 mm was 500.6 mW/cm^2 and 398.2 mW/cm^2, respectively. The enamel attenuated 73% of the transmitting light and dentin 79%. Air-drying of the samples increased the attenuation of light because the water around the enamel rods and in the dentin tubules was replaced with air. The refractive index for water is 1.33 and 1.00 for air, which probably explains the effect. When bonding brackets, the etched enamel surface is air-dried and protected from moisture. Etching reveals the HAP crystals, and the air drying of the surface removes the water between the enamel rods and HAP crystals. However, the dentin beneath the enamel remains intact, and in normal condition dentin tubules are filled with fluid and nerve receptors.

The Beer-Lambert law states the relation between absorbance and material concentration, but the linearity of the relationship is limited if the media is highly scattering, as is the case with enamel and dentin. In present study, it was shown that the increase in specimen thickness, in both enamel and dentin, whether as separate specimens (II) or in vertical tooth slices (I), significantly decreased the amount of transmitted light, as could be expected on the basis of the Beer-Lambert law. The trend in light attenuation was similar in both tests (I, II). Hence, it can be suggested that the light attenuation through enamel and dentin follows the Beer-Lambert law within the wavelength of the blue light that was used.

When light propagates through a turbid media, it is composed of absorbed transmitted and reflected light. (Gobrecht et al. 2015) In the case of enamel, the reflected light is blue light and the phenomenon is called Rayleigh scattering. Rayleigh scattering appears when light scatters from electrically polarized particles that are smaller than the wavelength of light, resulting in scattering as visible blue light. (Johnston et al. 1996) In addition, the scattering of the curing light is affected by the surface texture. In the present study (I, III) the surface treatment may have

influenced to the results along the edge-loss effect, since there was no mold to inhibit light scattering from the edges of the specimen. (Faria-e-Silva et al. 2008)

The EDTA treatment did not significantly affect light propagation through air-dried enamel which was hypothetical; hence the hydroxyapatite crystals contribute light propagation through enamel the most, rather than the enamel rods (Vaarkamp et al. 1995). The light attenuation was only 2% greater when the dentin tubules were obliterated (I), thus the results of this study support the suggestions of Turrioni et al. (2012) and Kienle et al. (2006) that the light propagation through dentin is a result of the scattering effect of the intertubular dentin.

The joule (J) is a symbol of energy or work that is done when an object is moved for distance of one meter with a force of one newton. According to the Newton's laws of mechanics, the energy (J) is the product of power (W) and time (T). Thus the energy density (J/cm^2) is the product of power density (W/cm^2) and exposure time (Yoon et al. 2002). Power density is the rate of delivered photons per surface unit and determines the rate of free radicals generated when the exposure time determines the total number of photons and thus the total of free radicals. It is known, that free radicals initiate polymerization and so the number of free radicals are responsible for the DC%. (Querrero-Santos et al. 2013) However, when the power density of the LCU remains low, the increase in curing time does not result as sufficient polymerization. (Musanje, Darvell 2003) It has been suggested that the energy density between 12–16 J/cm^2 is required for sufficient bracket bonding (Staudt et al. 2006, Beolchi et al. 2015). The LCU with an output power of 1900 mW/cm^2 required curing time for resin based composite system is 8.4 s. According to the results of the present study (II), bonding by transillumination only would require a curing time of 426 s to reach the energy of 16 J/cm^2.

6.3 Degree of conversion of the orthodontic adhesive

6.3.1 DC% of the adhesive under metal bracket with and without glass-fiber weave (I)

In the conventional bonding method of metal brackets light is directed from the sides. It can be assumed that the adhesive at the bracket edges receives more light than that at the center of the bracket. Although it could be assumed that highly polymerized edges of the adhesive would prevent the leakage of monomers, the shrinkage during polymerization of the adhesive must be taken into consideration. The resin adhesive is composed of organic matrix of polymeric chains and cross-linking agents, inorganic filler particles and a coupling agent. Fillers are important in restorations because they reduce shrinkage and stress of the adhesive during polymerization and

enhance properties of the composite, such as color and translucency (Kaisarly and El Gezawi 2016). They have a less important role in orthodontic adhesives, but high polymerization shrinkage can lead to microleakage at the adhesive-enamel or at the adhesive-bracket interface (Arhun et al. 2006, Buyuk et al. 2013). Microleakages are reported to increase the risk of "white spot lesions" in orthodontics. (Arhun et al. 2006, Buyuk et al. 2013, Canbek et al. 2013). In addition to the polymerization shrinkage, microleakage can be caused by changes in oral temperature that are leading to volumetric changes of the composite (Atash et al. 2017). It can be assumed that unpolymerized monomers can leach to the oral cavity through microleakage. It is therefore important to obtain a high level of conversion of the adhesive under the entire bracket.

The results of the present study that a glass-fiber weave under the bracket enhanced the DC% of the adhesive (I) were similar with Shinya et al. (2009) and Durgesh et al. (2015). The tested glass-fiber fabric contained non-directional fibers. It seems that best effect can be achieved by using unidirectional glass-fibers with the light directed perpendicular to the glass-fibers (Durgesh et al. 2015). There are two wavelength dependent coefficients. The refractive index describes how light is refracted by the material and the extinction coefficient describes the attenuation of light per unit length. It has been shown that polymerization increases the refractive index of the bis-GMA-TEGDMA resin system and decreases the extinction coefficient. (Lehtinen et al. 2008, Vallittu 2015). Another possible reason could be the increase in the thickness of the adhesive layer which could have enhanced the transmittance of the curing light to the center of the bracket. Jain et al. (2013) found that when the adhesive thickness was decreased from 0.99 mm to 0.83 mm the bond strength of the bracket increased significantly. Nevertheless, the results of Durgesh et al. (2015) indicated that a glass-fiber weave provides higher bond strengths compared to bonding without a glass-fiber reinforcement.

The DC% of the adhesive decreases as the distance from the light curing tip increases due to the attenuation of radiant energy as it is transmitted through the material (Yap 2000, Felix and Price 2003, Aravamudhan et al. 2006). The DC% of a resin based composite without light scattering objects, such as brackets, is ranging between 55-75% with conventional light curing (Eliades et al. 1987, Viljanen et al. 2004). In the present study (I), the DC% decreased significantly when the size of the light shielding matrix was increased indicating that efficacy of the light curing is significantly reduced by light obstructing objects. Therefore, it can be assumed that light propagation under a normal bracket is similarly decreased. When bonding stainless steel brackets with the conventional method, the DC% of the adhesive under the bracket is commonly 35-45% (Niepraschk et al. 2007, Shinya et al. 2009), which is significantly lower than the level of conversion with direct curing of the adhesive.

6.3.2 DC% of the adhesive under tooth-colored brackets (IV)

The light transmission through the tested transparent brackets varied from 560 mW/cm^2 to 643 mW/cm^2 when the output power of the LCU was 1850 mW/cm^2. The tooth-colored brackets attenuated 65–70% of the transmitting light. Of the brackets tested in the present study Inspire ICE, Fascination and Elegance are clear in appearance whereas Ovation C is more opaque and creamy. No significant difference was observed in light transmittance between Elegance, Fascination and Inspire Ice, whereas transmission through Ovation C was significantly lower. It can therefore be concluded that the crystalline structure did not affect the light transmittance. However, only few bracket labels were tested. The filler content of the brackets was found to affect the light transmittance. This is in line with the findings of Eliades et al. (1995) that the structure, morphologic factors and bracket composition significantly influence the transmission of curing light. Santini et al. (2016) found that transmitted light energy through monocrystalline and polycrystalline ceramic brackets were significantly different. However, the results of Aldossary et al. (2018) indicated that light energy attenuation through ceramic brackets it not affected by the crystalline structure of the bracket.

The DC% of the adhesive under tooth-colored brackets was higher when the adhesive was cured directly through the bracket compared to curing from the mesial and distal sides of the bracket despite an additional curing time of 10 seconds. DC% values after curing straight through the bracket varied between 48-53% and those after curing from the sides between 34-36%. Surprisingly, the DC% under stainless steel bracket after straight irritation resulted with similar DC% level than curing from the sides of the bracket. This was most likely caused by the large size of the LCU tip that was wider than the metal bracket. There was no significant difference in DC% between Inspiration Ice, Ovation C and Fascination. However, The DC% of the glass-fiber reinforced bracket (Elegance) was significantly higher. Glass-fibers have shown to enhance light scattering and surface irregularities increase the scattering even more (Vallittu 2015) which may explain the high level of DC%.

The mechanical properties of cured adhesive, e.g. the flexural modulus of elasticity, tensile strength and compressive strength, depend on the degree of cure (Eliades et al. 1995, Aromaa and Vallittu 2018). The results of the the present study (IV) showed that the surface microhardness correlated significantly with the direction of light curing which in turn, influenced the level of polymerization. No statistically significant difference in microhardness was observed between the edges and the center of the transparent brackets, possibly because the brackets allowed a more even penetration of light and conversion of the adhesive. However, with stainless steel brackets the adhesive remained softer at center of the bracket than at the edges, in both curing directions.

6.3.3 DC% of the adhesive cured with transillumination (III)

The DC% of the adhesive under metal brackets varied between 35-45% when curing was carried out from the sides of the brackets (I, IV). Curing with transillumination through human incisors or test specimens containing various layers of dentin and enamel resulted in DC% values between 31% to 45% (III). However, light curing through premolars resulted in poor DC% values when the scattering of light was inhibited and the polymerization of the adhesive took place only by transmitted light.

The results of the present study (III) that light curing through the incisors at straight angle towards the labial surface resulted with sufficient bond strength corroborate the findings of Dobrin et al. 2018. In addition to the direction of curing light, curing time affects the bond strength (Oesterle and Shellhart 2001, Heravi et al. 2013). In the present study (III) curing times of 20 s, 40 s and 60 s were tested. The results showed that the DC% increased as the curing time became longer. However, the relation of the DC% and the curing time was not linear because the proceeding polymerization limits the mobility of the monomer (Elliott et al. 2001, Bang et al. 2004).

6.4 Debonding force of stainless steel brackets (III)

Shear and tensile bond tests are commonly used *in vitro* studies to evaluate adhesive performance. These tests serve for different purposes and the results are not comparable with each other. Factors that affect the test results include the time between bonding and debonding, storing of the specimen, thermocycling, light curing protocol, adhesive composition, etching and bracket type (Bishara et al. 2005). Fox et al. (1994) and Bishara et al. (2005) concluded that changes in the study design, e.g. crosshead speed of the test machine, can significantly affect the test results. In the present study (III), the bond strength was measured by the means of debonding force (N), rather than by the more commonly used shear bond strength (MPa). This was due to the nature of the bracket debonding protocol in the present study, where the force is not divided equally to the whole specimen area, but rather only to the bracket-adhesive junction. Therefore, the applied force cannot be called as shear, as the it is not purely vertical but also itinerant to the bracket.

The results of the present study (III) showed that the brackets cured directly from the lingual side of incisors had higher debonding forces than brackets cured at mesial-distal direction from the labial side of the tooth. In case of premolars there was no statistical difference in debonding force whether cured with conventional or transillumination technique. Relatively high debonding force observed in premolar brackets cured with transillumination premolars could be explained by scattering of light and it can be assumed that the use of optical contact would have decreased the debonding force. However, scattering cannot be considered as an independent

phenomenon since transmitted light is composed of absorbed, transmitted and reflected light. Results of the study (III) are in line with earlier findings indicating that light transmission is effective in bonding metal brackets to incisors (Oesterle et al. 2001, Heravi et al. 2013, Dobrin et al. 2018). In premolars, the debonding force of brackets bonded with transillumination was comparable to that with conventional curing, but the DC% remained low, even with a curing time of 60 seconds. Thus, light curing through premolars cannot be recommended for clinical use.

The prevalence of bracket failures varies between 6.0–17.6% during orthodontic treatment with fixed appliances (Millett et al. 1998, Sunna and Rock 1998, Reis et al. 2008). Bracket failure can occur at three interphases: at enamel-resin junction, within the resin matrix and at bracket-resin junction. Bond failure is most likely to occur within the adhesive interface because of the stress concentration and resin film defects (Wang et al. 1994, Sunna and Rock 1998). In the present study (III), the adhesive remaining index (ARI), scored after every bracket debonding, indicated that the adhesive was left completely on the enamel surface in almost 50% of the premolars and incisors. In 40% of the teeth, more than half of the adhesive was left on the enamel. Not once was the adhesive completely removed with the bracket.

Findings of the present study (III) showed that the bracket failures were caused by a weak bracket-adhesive interface rather than by a weak bond between enamel and adhesive. A sufficient bracket bond strength is considered to be between 5.8 MPa and 7.9 MPa (Reynolds 1975) which is low compared to the bond strength between adhesive and enamel that can be as high as 20 MPa (Arab et al. 2018). Surface area, mesh layer design and retention base grooves are features of the bracket base that influence the bonding strength. Maccoll et al. (1998) found no differences in SBS with stainless steel brackets when the surface area of the varied between 6.82 and 12.35 mm^2 but SBS decreased significantly with a base smaller than 6.82 mm^2. Wang et al. (2004) showed that retention grooves, circular concaves and mesh layers influence on the bracket bond strength. Ceramic brackets with flat base design were found to have weaker bond strengths than stainless steel brackets (Zielinski et al. 2014, Stasinopoulos et al. 2018).

6.5 Clinical considerations and future perspective

Bracket failures due to accidental debonding are common during orthodontic treatment. Ceramic brackets seem to be 60% more prone to failure than stainless steel brackets (Stasinopoulos et al. 2018). Bracket failures compromise the duration of the orthodontic treatment and have been found to be an important predictor of the duration of fixed appliance treatment and patient compliance (Beckwith et al. 1999, Skidmore et al. 2006). Skidmore et al. (2006) found that rebonding of 2 brackets extended the treatment time by approximately 2.2 months and resulted in increased

costs and chair-time. This can jeopardize patients' motivation and complicate the treatment.

Pre-coated orthodontic brackets (POBs) are individually packaged with optimal amount of adhesive. (Lee and Kanavakis 2016). The adhesives used with pre-coated brackets contain more fillers than commonly used adhesives such as the Transbond™ XT in order to increase the viscosity of the adhesive and to achieve better bond between the adhesive and enamel (Hassan 2010). *In vivo* and *in vitro* studies on bond strength of pre-coated brackets have given conflicting results. While one study indicated that conventionally bonded stainless steel brackets had higher bond strength that POBs (Sfondrini et al. 2002), others have found no difference between POBs and conventionally bonded brackets (Wong and Power 2003, Vicente and Bravo 2007, Hassan 2010). The advantages of POBs are the consistent quantity and quality of the adhesive, fast bonding, easy clean-up after bracket placement, good aseptics and reduced waste (Lee and Kanavakis 2016).

Shinya et al. (2009) found that the use of a glass-fiber weave under the bracket enhances the DC% of the adhesive. However, a pre-cut glass-fiber weave is not commercially available and cutting and placing of the glass-fiber weave is time consuming and technically challenging. Since the bond strengths of POBs are sufficient, adding a pre-cut glass-fiber weave to the pre-coated bracket base could result in an ideal product for fixed appliance orthodontics.

The orthodontic usage of glass-fibers of reinforcements has mainly been limited to anchorage and retainers (Goldberg and Burstone 1992, Shinya et al. 2009). FRCs have been shown to have better resistance against fatigue and fractures compared to conventional resin composites (Garoushi et al. 2006) but the fillers in the resin matrix and the glass-fiber reinforcement typically make FRCs highly viscous which is not a favorable feature in orthodontic adhesive. However, a new flowable glass-fiber reinforced composite has recently been investigated in laboratory conditions (Lassila et al. 2019). Because of the many advantages of the FRCs, bonding brackets with a flowable fiber reinforced composite would be of interest for orthodontists.

7 Conclusions

The main conclusions of the present study were:

1. Propagation of the curing light under metal brackets is considerably decreased when the dimension of the bracket is increased. Usage of glass-fiber weave under orthodontic bracket enhances the degree of conversion of the adhesive and it could be therefore suitable for clinical use.
2. Transmittance of the curing light through the dental hard tissues seems to follow the Beer-Lambert law. A significant difference in light transmittance between enamel and dentin was observed.
3. Bonding of brackets on incisors with the conventional or transillumination technique resulted in similar bond strength and polymerization of the adhesive. Bracket debonding was likely to result from a failure in bracket-adhesive rather than adhesive-enamel interface in both bonding protocols.
4. Light curing through transparent brackets resulted in higher DC% than curing from the sides of the bracket.

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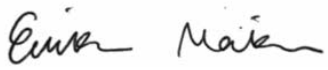

Erika Mäkinen

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