

**A COMPARISON IN SHEAR BOND STRENGTH OF DIFFERENT
ADHESIVES BETWEEN ORTHODONTIC BRACKET-TOOTH
SURFACES AND ORTHODONTIC BRACKET-AMALGAM
RESTORATIONS**

SIRIWAN KUNTHARAPORN

**A THESIS SUBMITTED IN PARTIAL FULFILLMENT
OF THE REQUIREMENTS FOR
THE DEGREE OF MASTER OF SCIENCE
(ORTHODONTICS)
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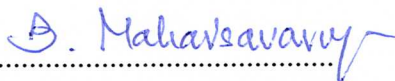
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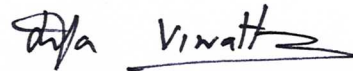
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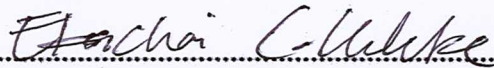
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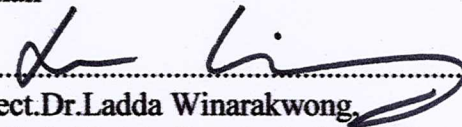
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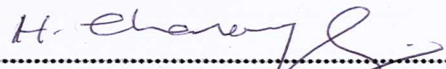
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A COMPARISON IN SHEAR BOND STRENGTH OF DIFFERENT ADHESIVES BETWEEN ORTHODONTIC BRACKET-TOOTH SURFACES AND ORTHODONTIC BRACKET-AMALGAM RESTORATIONS

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ABSTRACT

The objective of this thesis was to compare the shear bond strength (SBS) of different adhesives between metal orthodontic brackets on tooth surfaces and on tooth surfaces with amalgam restorations. Two hundred and fifty human upper premolars extracted for orthodontic purposes were randomly divided into two major groups; a tooth surface group and a 50% buccal amalgam restoration group. Brackets were bonded to the teeth using five different adhesives in each groups: I: UniteTM; II: Rely-a-bond[®]; III: Enlight[®]; IV: Transbond XTTM; V: Transbond PlusTM with self-etching adhesive. The teeth were thermocycled between 5° and 55° for 500 cycles, and consequently debonded using Instron testing machine to test the SBS. The enamel surfaces were examined under a stereomicroscope at 25X magnification and the amount of residual adhesive remaining on the teeth was determined with Adhesive Remnant Index (ARI) scores. The SBS values of all adhesives used on tooth surfaces were greater than the bonds of brackets on tooth surfaces with the amalgam restorations ($p < .05$). Significant differences among different adhesives did not exist in the tooth surface group ($p > .05$), but did exist in the amalgam restoration group ($p < .05$), in which Transbond XT and Enlight SBS values were significantly higher than Unite, Transbond Plus and Rely-a-Bond. In conclusion, the SBS values on tooth surfaces of all adhesives in this study were clinically acceptable (5.9-7.8 MPa). Three-step light-cured adhesives (Transbond XT and Enlight) had clinically acceptable SBS values on tooth surfaces with amalgam restorations. In a comparison of the ARI scores, all adhesives both on tooth surfaces and tooth surfaces with amalgam restorations had different failure modes. Mostly, adhesive failure was found between adhesive/bracket bases on tooth surfaces, and cohesive failure was found within adhesives on tooth surfaces with amalgam restorations, with the exception of Transbond Plus, in which the same failure modes were recorded both on tooth surfaces and tooth surfaces with amalgam restorations. Failures of the Transbond Plus group occurred mostly within adhesives and between adhesive/tooth surfaces and amalgams.

KEY WORDS: METAL BRACKET/ SHEAR BOND STRENGTH/ ADHESIVE/ ARI SCORE/ AMALGAM RESTORATION

73 pp.

การเปรียบเทียบค่าความแข็งแรงเฉือนระหว่างเบร็กเก็ตจัดฟันชนิดโลหะกับผิวฟันและเบร็กเก็ตจัดฟันชนิดโลหะกับผิวฟันที่มีวัสดุอุดอะมัลกัม (A COMPARISON IN SHEAR BOND STRENGTH OF DIFFERENT ADHESIVES BETWEEN ORTHODONTIC BRACKET-TOOTH SURFACES AND ORTHODONTIC BRACKET-AMALGAM RESTORATIONS)

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บทคัดย่อ

การศึกษานี้มีวัตถุประสงค์เพื่อเปรียบเทียบค่าความแข็งแรงเฉือนของวัสดุยึดติดต่างชนิดกันเมื่อใช้ยึดเบร็กเก็ตจัดฟันชนิดโลหะบนผิวฟันปกติและเทียบกับเมื่อใช้ยึดบนผิวฟันที่มีวัสดุอุดอะมัลกัม โดยรวบรวมฟันกรามน้อยบนจำนวน 250 ซี่ลงบดออกอะคริลิกเรซินและแบ่งออกเป็น 2 กลุ่มใหญ่คือ กลุ่มผิวฟันปกติและกลุ่มผิวฟันที่มีวัสดุอุดอะมัลกัม ซึ่งนำมาเตรียมโพรงฟันและอุดด้วยอะมัลกัมให้มีขนาด 50 เปอร์เซ็นต์ของพื้นที่ได้ฐานเบร็กเก็ต โดยเบร็กเก็ตจะถูกยึดด้วยวัสดุยึดติดชนิดเรซินต่างชนิดกัน 5 กลุ่มได้แก่ ยูไนท์TM รีไล-อะ-บอนด์[®] เอ็นไลท์[®] ทรานส์บอนด์ เอ็กซ์ทีTM และ ทรานส์บอนด์ พลัสTM แล้วนำไปผ่านเครื่องเปลี่ยนอุณหภูมิร้อน-เย็นระหว่าง 5 ถึง 55 องศา จำนวน 500 รอบ จากนั้นนำไปวัดค่าความแข็งแรงเฉือนด้วยเครื่องทดสอบอินสตรอน และนำไปประเมินตำแหน่งที่เกิดการหลุดของเบร็กเก็ตด้วยกล้องจุลทรรศน์กำลังขยาย 25 เท่าเพื่อตรวจดูปริมาณวัสดุยึดติดที่เหลืออยู่บนผิวฟันด้วยค่าครรชนีเออาร์ไอ ผลการศึกษาพบว่า ค่าความแข็งแรงเฉือนของวัสดุยึดติดแต่ละชนิดบนผิวฟันปกติมีค่ามากกว่าบนผิวฟันที่มีวัสดุอุดอะมัลกัมอย่างมีนัยสำคัญทางสถิติ ($p < .05$) และไม่มีความแตกต่างกันระหว่างชนิดของวัสดุบนผิวฟันปกติอย่างมีนัยสำคัญทางสถิติ ($p > .05$) แต่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติเมื่อยึดติดบนผิวฟันที่มีวัสดุอุดอะมัลกัม ($p < .05$) โดยทรานส์บอนด์ เอ็กซ์ที และเอ็นไลท์ จะให้ค่าความแข็งแรงเฉือนที่มากกว่า ยูไนท์, ทรานส์บอนด์ พลัส และ รีไล-อะ-บอนด์ สรุปได้ว่าวัสดุยึดติดทุกชนิดที่นำมาทดสอบมีค่าความแข็งแรงเฉือนเมื่อติดเบร็กเก็ตบนผิวฟันปกติอยู่ในระดับที่ยอมรับได้สำหรับการใช้งานทางคลินิก (5.9-7.8 เมกะปาสกาล) และวัสดุยึดติดชนิดที่แข็งตัวด้วยแสงที่ใช้การเตรียมผิวฟันแบบ 3 ขั้นตอนตามปกติ ซึ่งได้แก่ ทรานส์บอนด์ เอ็กซ์ที และเอ็นไลท์ มีค่าความแข็งแรงเฉือนบนผิวฟันที่มีวัสดุอุดอะมัลกัมอยู่ในระดับที่ยอมรับได้สำหรับการใช้งานทางคลินิก สำหรับการประเมินการหลุดของเบร็กเก็ตบนผิวฟันปกติกับผิวฟันที่มีวัสดุอุดอะมัลกัม วัสดุทุกชนิดที่ทดสอบ ยกเว้นทรานส์บอนด์ พลัส มีลักษณะการหลุดบนผิวฟันปกติกับบนผิวฟันที่มีวัสดุอุดอะมัลกัมแตกต่างกัน คือบนผิวฟันปกติส่วนใหญ่เกิดการแยกออกระหว่างวัสดุกับฐานเบร็กเก็ต ส่วนบนผิวฟันที่มีวัสดุอุดอะมัลกัมส่วนใหญ่เกิดการแยกออกภายในเนื้อวัสดุเอง แต่ทรานส์บอนด์ พลัส พบลักษณะการหลุดที่ไม่แตกต่างกันระหว่างบนผิวฟันปกติและผิวฟันที่มีวัสดุอุดอะมัลกัม คือพบการแยกออกภายในเนื้อวัสดุเอง และการแยกออกระหว่างวัสดุกับผิวฟันและอะมัลกัม

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CHAPTER I

INTRODUCTION

Since Buonocore introduced the acid-etching bonding technique in 1955, the concept of bonding resins to enamel has been consequently applied to all fields of dentistry, including the bonding of orthodontic brackets (1). By the 1970s, bonding of orthodontic brackets had become an accepted clinical technique. Bonding bracket has several advantages over banding, including ease of placement and removal, minimal soft tissue irritation and hyperplastic gingivitis, minimal danger of decalcification with loose bands, and being more esthetic (2). However, bond failure of brackets is one of problem occurrences including; increased treatment time, additional costs and unexpected additional visits (3). Therefore, the bond strength of adhesives should be sufficient to withstand the forces of mastication, the stress exerted by archwires, and patient abuse. At the same time, the bond strength should be at a level to allow for bracket debonding without causing damage to the enamel surface (4).

Moreover, adult patients in need of orthodontic treatment often have amalgam restoration, extensive buccal amalgam restoration which limit the surface area of enamel available for etching, and resin impregnation, obviously increase the difficulty of the bonding process. At present, it is understood that reliable bonding of orthodontic attachment to dental amalgam restorations is not possible with conventional orthodontic bonding techniques and bonding materials (5).

However, cost-benefit should be considered, whether the tooth with amalgam restoration should be re-restoration with composite filling, band or enhancing with intermediate resins to achieve adequate bond strength for orthodontic treatment (6-8). Amount of remained sound enamel surrounding a buccal amalgam filling may provide reliable bond to amalgam and neighboring acid-etched enamel.

Advances in the adhesive field have led to the development of new materials with improved properties, making more types and systems in choosing. Initiation of the polymerization process can be achieved chemically, as in the self-cure

materials or by the action of blue light. In dentistry, self-cure composites are usually available as two-paste systems, one containing the activator and the other the initiator, which are mixed together. Complete polymerization usually takes place within a few minutes. However, in orthodontics, an additional disadvantage of this method is that air bubbles can become incorporated into the composite during mixing. Obviously the presence of a significant air bubble under a bracket can be catastrophic. Therefore, no-mix systems have been developed where initiator and activator is separated, present in a fluid which is painted onto the etched enamel and bracket base and a paste which is then applied to the bracket. Mixing of the two components is achieved by the paste being squeezed to a thin layer. However, if the bracket is not pushed firmly into place because the layer of resin is too thick, which means that the separate components do not come into intimate contact resulting in incomplete polymerization (9).

Self-polymerizing materials have proved popular and reliable. However, one significant disadvantage to this system is that possible little manipulation of the working time. This can lead to problems with accurate bracket placement, particularly for the novice. Therefore, in parallel with other branches of Dentistry, light activated materials have become increasingly fashionable (10, 11). Recently, the clinical use of visible light-cured adhesive for direct bracketing was widely advertised by several manufacturers. This light-cured resin single paste system that consists of a ketone and amine as initiators. The ketone, camphoroquinone sensitive to blue light at a 470 nm wavelength, which catalyzes the polymerization reaction (12). The disadvantages of light-cured resin are associated with incomplete polymerization beneath the surface, the curability and diffuse penetration of the light for curing the resin under a metal, ceramic, or resin bracket are doubtful and questionable because the characteristics and thickness of the bracket material and thickness of composite resin may inhibit the light penetration (13, 14). However, both in vivo and in vitro testing have shown that the use of light-cured adhesives for bonding orthodontic brackets should produce bond strengths and failure rates similar to those of chemically cured adhesives (10, 11, 15, 16).

Conventional resin adhesive systems use the different agents; an enamel conditioner, a primer solution, and an adhesive resin to bond orthodontic brackets to

enamel. Orthodontists generally use the conventional acid-etching bonding technique to attach brackets to the enamel surface. The self-etching adhesives have recently become available and combining the functions of primer and adhesive components, not requiring a separate acid-etching step and thus eliminating the need for rinsing. The self-etching systems are capable of etching the tooth surface and simultaneously preparing it for adhesion. Combining conditioning and priming into a single step reduces the bonding time and increases the cost effectiveness for the clinician and indirectly for the patient. However, effective bonding by self-etching systems is controversial (17-27).

In addition to the ARI scores (28) more descriptive observations were recorded and categorized. These data are helpful in characterizing the bond failure, since there are several interfaces in which fracture may occur. The weak link of the bond may be at the tooth surface (adhesive failure at enamel surface; no cement on tooth), at the bracket (adhesive failure at bracket material surface; cement on tooth, not on bracket), or within the adhesive cement (cohesive failure within the cement; cement on both tooth and bracket surfaces) (29).

This study was therefore designed to compare the shear bond strength of recently introduced commercial adhesives between orthodontic bracket to tooth surface and orthodontic bracket to tooth surface with amalgam restoration without enhancing or intermediate resins and determined the bond failure by an adhesive remnant index (ARI).

CHAPTER II

OBJECTIVES

Objectives of this study

1. To compare shear bond strengths (SBS) of orthodontic brackets with various adhesives both on tooth surfaces and tooth surfaces with amalgam restorations.
2. To examine the difference between SBS on tooth surfaces and SBS on tooth surfaces with amalgam restorations.

Research design

An experimental study

Hypothesis

1. There is no statistical significant difference of shear bond strength of each adhesive type between the tooth surface and the tooth surface with amalgam restoration.
2. There are no statistical significant differences of shear bond strength among five orthodontic adhesives on the tooth surface and the tooth surface with amalgam restoration.
3. The site of bond failure is independent of adhesive types.
4. The site of bond failure is independent of surfaces.

Expected benefits

This study will provide useful informations regarding the shear bond strength of currently available adhesives on both normal tooth surfaces and tooth surfaces with amalgam restorations. Furthermore, the orthodontists may use these informations as a guideline in selecting appropriate adhesives for different clinical situations.

CHAPTER III

LITERATURE REVIEWS

History of bonding

In 1955, Buonocore found that pretreatment with 85 percent phosphoric acid for 30 seconds altered the tooth surface by dissolution of organic component of the enamel matrix creating microporosities in the enamel surface, allowing acrylic to adhere (30). Essentially acid etching changes the enamel surface from a low energy hydrophobic to a high energy hydrophilic surface, showing increased surface tension and wettability (31). Therefore, after introduced this acid-etching bonding technique, the concept of bonding resins to enamel has been consequently applied to all fields of dentistry (1).

In 1965, Newman applied these findings to the direct bonding of orthodontic attachments to the tooth surface (in vivo). He used an epoxy resin (diglycidyl ether of Bisphenol A with a polyamide curing agent) after etching with 40 percent phosphoric acid for 60 seconds (32). In 1967, Retief and Dreyer issued a detailed report on the in vitro investigation of epoxy resins as orthodontic adhesives. In 1969, Newman reported on the use of methyl methacrylate and in a clinical study. He described the adhesive under investigation as an acrylic containing ether links. In 1968, Mulholland and De Shazer reported on in vitro tests with Addent 35 (an early composite), but there were no clinical follow-ups and no specific reports of diacrylate adhesives for brackets (33). Miura et al. in 1971 described an acrylic resin, using tri-n-borane derivative instead of the more conventional amine-peroxide curing system. This has proved successful and is commercially available (Orthomite, Rocky Mountain). The advantage of this resin is its ability to polymerize in a moist environment (31). Until 1972, Cohl and Silverman et al. described the use of commercial UV-curing fissure sealant for this purpose (34). Although this system provides the dentist unlimited working time, it also has several disadvantages. One drawback is the extremely limited ability of ultraviolet light to penetrate either tooth structure or

composite resin (33). Tavas and Watts first described the use of visible light to cure composites used in orthodontic bonding in 1979. Visible light-cured composites provide ease of use, extended working time, improved bracket placement, easier clean up, and faster cure of the composite (29).

By the 1970s, bonding of orthodontic brackets had become an accepted clinical technique. Bonding bracket has several advantages, including ease of placement and removal, minimal soft tissue irritation and hyperplastic gingivitis, minimal danger of decalcification with loose bands, being more esthetic, no need of separation of adjacent teeth and closing post treatment band space, and facilitates possibility of exact mechanical positioning of brackets. However, some disadvantages of bonding bracket, including the satisfactory adhesives were often difficult to remove, and the surface area of attachment available for retention was greatly reduced (2, 31).

Adhesion and requirement of adhesion

Adhesion describes the attachment of one substance to another whenever they come into close contact with each other. Therefore, it can be defined as the force that binds two dissimilar materials together when they are brought into intimate contact. This is different from cohesion, which is the attraction between atoms or molecules within one substance. Adhesion is the attraction of molecules at surfaces, and the bond strength depends on the amount of force present at each contact site. At an atomic level, solids often have rough surfaces, which mean that they contact each other only at certain points. To get a better contact between the two materials, an intermediate layer, called an adhesive, has to be placed. The surfaces or substrates that are adhered to are termed the adherend (35).

The two main theories for the observed phenomena of adhesion are *the mechanical theory*, which states that the solidified adhesive interlocks micro-mechanically with the roughness and irregularities of the adherend's surface, and *the adsorption theory*, which includes all kinds of chemical bonds between the adhesive and the adherend, including primary and secondary valence forces. Primary forces are ionic and covalent bonding, and secondary forces are hydrogen, dipole interaction, and van der Waals forces (35).

The most important requirement for adhesion is that the two materials to be bonded to each other must be in sufficiently close and intimate contact. To achieve this requirement for solid bodies, liquids or flowable materials (the adhesives) can be used. Their intimate contact with the substrate depends on the wettability of the substrate, the viscosity of the adhesive, and the morphology and the roughness of the substrate (35).

The wetting of a surface by a liquid is usually characterized by the contact angle of a droplet placed on the solid surface. The better the liquid spreads over the surface, indicated by a contact angle of close to 0 degrees, the better the wetting takes place. The phenomenon of wetting depends on the difference between the surface tension of the adhesive and the surface tension of the adherend (35). The surface tension of that liquid is taken as approximately the free surface energy of the

substrate. When the free surface energy is higher than the surface tension, a liquid will wet completely, when it is lower, wetting will be incomplete (34).

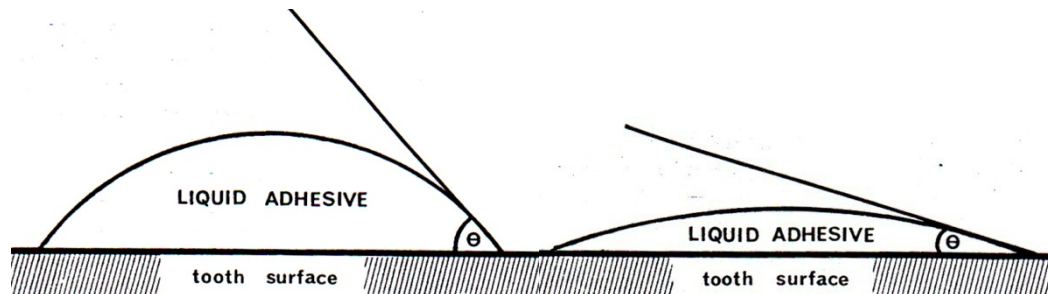


Figure 1 Contact angle between adhesive and the tooth surface. Left, high contact angle; right, low contact angle (34)

The viscosity is a term describing consistency. A thick, pasty material cannot be expected to readily flow over surfaces, viscosity interferes with establishing maximum wetting in spite of the fact that strong attraction may exist between molecules of a viscous adhesive and those of the adherend surface (36).

The irregularities at the surface can create capillary forces that support the diffusion of an adhesive into the rough surface. On the other hand, a deeply fissured surface might lead to air impaction if the viscosity of the adhesive is not as low as necessary to invade the rough surface (35).

It can be concluded that whenever the substrate has a high surface energy and the adhesive has a low viscosity, then the microporosities and irregularities of a surface can be filled to achieve micromechanical retention (35).

Surface conditioning

Bonding success or failure starts with the tooth surface. Proper conditioning of the surface, whether it is enamel, composite, porcelain, or metal, is critical. A surface that is not conditioned properly or that becomes contaminated cannot achieve satisfactory bond strength, regardless of adhesive type (37).

Enamel is highly mineralized tissue composed of hydroxyapatite about 96%, water 4%, and collagen 1% (38).

The first step in preparing enamel for bonding has to be to remove the surface layer of contaminants. It is standard procedure for the prophylaxis to be carried out with unflavored and unfluoridated pumice. The reason for using an unflavored abrasive is that most flavorings in dental paste come from essential oils, often containing glycerin. These substances can interfere with the work of the acid. Further observations suggest that the fluoride should be removed from the polishing agent because it reacts with the calcium hydroxyapatite of the enamel to form calcium fluorapatite, a substance much more resistant to acidic attack (33).

The untreated enamel surface is either smooths (meaning not retentive) or covered by plaque. Therefore, enamel has to be conditioned. Adhesive techniques started in 1955 when Buonocore first applied phosphoric acids to enamel and proved that this procedure resulted in an altered surface that increased the bonding of acrylic to human teeth. Subsequent studies suggested that the formation of resin tags at the interface to acid-etched enamel was the leading mechanism of adhesion to enamel (30). The goal of condition the enamel should be to create an effective etching pattern. The etching procedure which also includes sufficient rinsing with move all precipitates, results face with a dramatically area and, even more high surface energy. This helps the adhesive to get into conditioned enamel a micromechanical substrate. However, the high energy of the acid-etched enamel is also attractive to other liquids like blood, or sulcus fluid. Each contact of any liquid to the conditioned enamel will decrease the surface energy, resulting in a reduced wettability of a hydrophobic adhesive. As a conclusion, acid etching of enamel results in a rough and enlarged surface with high surface energy. Such a surface

promotes wettability and improved micromechanical retention and meets the requirements for effective adhesion (35).

However, the effect on the etching pattern in enamel depends on type, acidity, time, form and pattern of application of the etchant used.

Other demineralizing acidic agents have been tested for the simultaneous conditioning of enamel and dentin. The type concentration, and application time of the etchant are adapted to obtain a reasonable enamel etch pattern without cause extreme demineralization. Nitric acid (2.5%), citric acid (10%), maleic acid (10%), pyruvic acid (10%), polyacrylic acid (20%), and oxalic acid (1.5% to 3.5%) have been suggested. The bond-strength test results and the long-term clinical consequences indicate that weak mineral etchants and milder organic acids are not as efficient in their enamel conditioning as the traditional phosphoric acid (20, 39).

The acid etching with phosphoric acid (35%) removes about 10 μm of the top surface, exposing the prism cores (4 μm remaining diameter) to a depth of 10 to 20 μm . The surface free energy doubles to 72 dynes per cm (33).

The recommended etching time with a conventional 37% phosphoric acid liquid or gel is a minimum of 15 seconds and a maximum of 90 seconds per tooth (37). Wang and colleagues evaluated several phosphoric acid concentrations, from 2% to 80%, and found the best bond strength was achieved with 30-40% concentrations (40). Barkmeier and colleagues reported no significant difference in bond strength whether a 37% phosphoric acid was applied for 15 seconds or 60 seconds (41). Etching for longer than 90 seconds may be harmful, because overetching causes dissolution of the enamel rods and the formation of insoluble calcium phosphate crystals. Any enamel surfaces etched with phosphoric acid must be thoroughly rinsed (five seconds per tooth with a liquid etchant, 10 seconds per tooth with a gel) and dried before application of the bonding resin (37).

In addition, the gel formulations with the addition of a colored dye allows visually accurate placement of the etchant and ensures that the material is completely washed off the etched surface. However, whether the thickening agents that are added to produce the gel can be completely washed from the surface and whether the resulting residue has a significant effect on resin penetration (42). MacColl et al (43) found that there was no statistically significant

difference between 37% phosphoric and 10% maleic acid gel preparations and 37% aqueous phosphoric acid, these results suggest that the gel residue is of no clinical significance.

Four major etching patterns of enamel are reported in the literature. The first, type I, is created when the center of the prisms erode more rapidly than the interprismatic enamel. A second topography, type II, is created when the interprismatic enamel erodes more rapidly than the prism cores. Although types I and II patterns are complete reverses of each other, both are suitable for mechanical retention. Interestingly, both patterns are often found in adjacent areas of the same tooth surface, even in adjacent prisms. In the type III etching pattern no rod structures are evident. type III pattern results when the enamel being etched is composed of a homogeneous mass instead of the more commonly found prismatic structure. The type III pattern can be troublesome for bonding because it does not allow the resin to grip the enamel. However, applying an acid etchant not only roughens the outer surface, but actually dissolves it. It is possible to etch past this prismless layer using the etchant itself. Thus, the time needed to etch an area of enamel displaying prismless outer structure is considerably greater than an area of normal enamel (33).

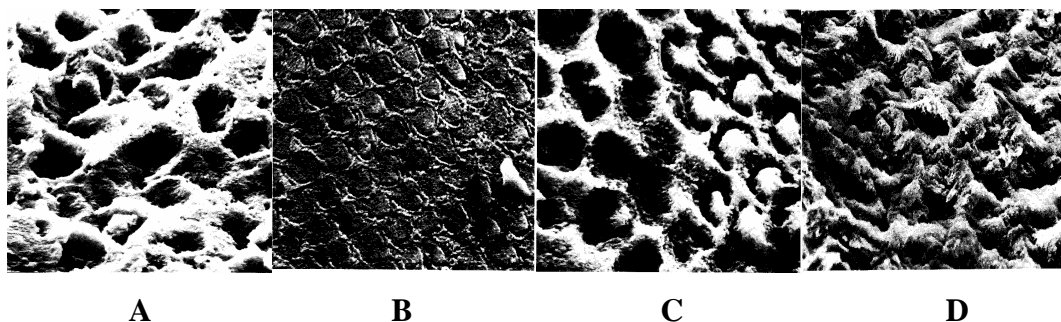


Figure 2 Etching pattern. A, Type I; B, Type II; C, Type I Type II; D, Type III (33).

Clinically, however, one has no impact on the type of prism core dissolution. The ultimate etching effect depends on the enamel instrumentation, the chemical composition, the fluoride content, the prismatic or aprismatic nature, and the tooth type (permanent or primary) (35). Hobson and McCabe (44) indicated that there were no significant relationship between bond strength and etch pattern, it is

difficult to explain the discrepancy between the poor quality of etch on the lower first molar and its large bond strength and vice versa with the upper first molar, so the development of a well-defined ideal etch pattern is not necessarily a pre-requisite for high-bond strength because the relationship between etch pattern and bond strength is complex.

The resin-enamel bond strength is mainly the result of the cumulative cross-sectional area of the tag-like resin extensions, macrotags and microtags, which infiltrate the etched enamel surface. Lengthening the tag does not increase the cumulative cross-sectional area or the enamel-resin bond strength. After efficient curing of the bonding resin, the infiltrating resin envelops apatitic crystallites in etched enamel to increase the bonding strength and also to make them resistant to cariogenic, endogenic, and exogenic acids (35). The hybridization theory can be used to explain the outcome of the current study and is further supported by the work of Leger et al. who reported a poor relationship between depth of resin penetration in etched enamel and the resulting resin-enamel bond strength, suggesting that enamel porosity is more important than a well-defined etch pattern. Hybridization is a process routinely used to achieve bonding to dentine by creating a mixture of adhesive polymers and dental hard tissues at a molecular-level (45, 46).

Adhesives

Type of adhesives

There are two basic types of dental resin currently in use for orthodontic bracket bonding. Both are polymer and are classified as methacrylate or dimethacrylate resin.

1. The methacrylate resin (Orthomite superbond, Directon, Bondeze, Genie, etc) are base on self-curing acrylics and consist of methymethacrylate monomer and ultrafine powder (47).

The basic composition of the components of these bonding adhesive

Polymeric components: the major constituent of this adhesive resin, is polymethyl methacrylate (PMMA), which has been widely used in dentistry for decades. This component contains no inorganic filler such as glass powder, silica, etc. Compared with resins such as composite, which contain a polyfunctional monomer and inorganic fillers. However, because it is unfilled, the resin film is resilient and extremely tenacious. It provides strong bonds that resist impact and torque better than the more brittle, filled resins.

Bonding monomer: 4 META are the bonding components which are MMA-soluble and which have a molecular structure in which hydrophilic and hydrophobic groups coexist. It is generally thought that such coexistence has the function of accelerating the penetration of monomers into tooth substrates.

Catalyst TBB (tri-n-butylborane): polymerization initiation for methyl methacrylate (MMA), reacts with oxygen in the air and water and oxidizes into peroxide. The peroxide further decomposes, and forms radicals, which in turn initiates polymerization of MMA.

Pretreatment of enamel surface 65% solution phosphoric acid, a short 30-second treatment permits excellent enamel bonds without excessive decalcification (47).

2. The dimethacrylate resin

Matrix: the most common organic polymer matrix is either an aromatic or urethane diacrylate such as bisphenol A diglycidyl dimethacrylate (Bis-GMA),

urethane-dimethacrylate (UDMA), triethylene glycol dimethacrylate (TEGDMA) or similar monomer. (38, 48-50) An organic chemical called a *diluent* is added to control the viscosity of the final product. C=C is the functional group of both bis-GMA and the diluent. The matrix of a dental composite polymerizes via chemical reaction called addition polymerization. Polymerization is activated via a chemical or light activation (48).

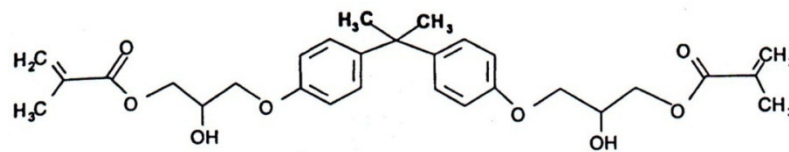


Figure 3 The structure of Bis-GMA

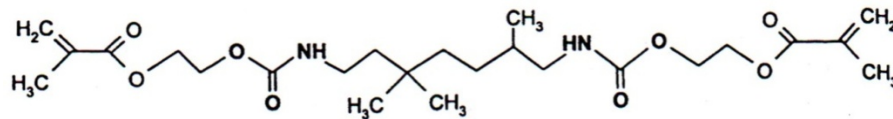


Figure 4 The structure of UDMA

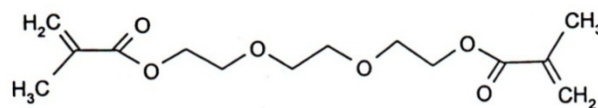


Figure 5 The structure of TEGDMA

Fillers: the combinations of inorganic composition such as quartz, barium or lithium aluminum silicate, barosilicate, zinc, strontium glasses (38, 49, 51).

Coupling agents: a bond between filler particle and matrix in the set composite is achieved by the use of an organic silicon compound, or silane coupling agent.

Initiators and accelerators: polymerization of composites may be achieved by chemical means (self-cure) or by visible-light activation. Dual cure is a combination of light and chemical curing. In chemically activated systems, an organic peroxide initiator (or catalyst), upon reacting with a tertiary amine accelerator, produces free

radicals that attack the double bonds of oligomer molecules and begin the process of additional polymerization.

Other ingredients: inorganic oxide pigments, polymerization inhibitors and stabilizers are added to the composite to lengthen shelf life.

Chemistry of Polymerization

Polymerization = A chemical reaction in which monomers of a low molecular weight are converted into chains of polymers with a high molecular weight (52). The polymerization reaction of acrylic resins, called additional polymerization, is very common in dental materials. Acrylic resins and composite materials restorative materials, sealants and adhesives all set via additional polymerization (48). The common factor relating all these materials is the same chemical structure of the reactive or function group (48). Additional polymerization is polymerized by a mechanism in which monomers add sequentially to the end of growing chain (52).

Functional groups

Monomer = A chemical compound capable of reacting to form a polymer (52). Monomers are molecules which have a reactive group that participates in the polymerization reaction, that group is called the functional group. The functional group is the carbon-carbon double bond (C=C). While C=C is the reactive part of the monomer, other atoms and side groups besides hydrogen can be bonded to either or both of the carbon atoms of the C=C. The side groups become pendants on the polymer chain and determine the physical properties of the resulting polymer (48).

Free radicals

Free radical = A compound with an unpaired electron that is used to initiate the polymerization (52). Free radical, an unpaired electron, is involved in the polymerization reaction (48).

Polymerization activation

Two types of the free-radical activation are used in the polymerization (or *cure*) of the unsaturated methacrylate groups of the resin composites (3):

Self-Cured or Chemical-Cured : These composites employ a two-paste system, one of which contains 1-2% benzoyl peroxide in the monomer portion as a free radical initiator. The activator in the other paste for these materials has usually been a tertiary amine, most commonly dihydroxyethyl-*p*-toluidine. The activator acts as an accelerator so that, on mixing, the benzoyl peroxide fragments into free radicals at room temperature, thus initiating polymerization. This type of system is most widely used in orthodontic adhesives (3, 52).

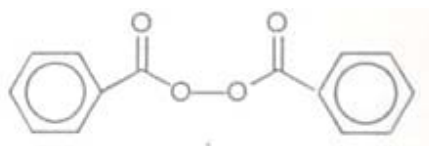


Figure 6 The structure of benzoyl peroxide

Light-Cured : These materials usually employ other types of free radical initiators, commonly an α -1,2-diketone such as benzil or camphoroquinone and an amine reducing agent such as *N,N*-dimethyl-amino-ethyl methacrylate. The radicalized ketone alone may initiate the photopolymerization. A reducing agent, the amine radical is responsible for initiating the polymerization and is more efficient than the radical formed from the ketone. These intensified radicals can thus significantly improve the degree of cure. The concentration of camphoroquinone photosensitizer is in the range 0.17-1.03 mass% range of the resin phase and that of *N,N*-dimethyl-amino-ethyl methacrylate reducing agent is 0.86-1.39 mass%. The combined photosensitizer/ reducing agent complex has an extended absorption band within the visible light spectrum (3, 52).

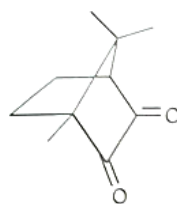


Figure 7 The structure of camphoroquinone

Although light-curing may be problematic with metal brackets, it was first shown by Tavus and Watts that a sufficient light may be transilluminated by the teeth to effect adequate photopolymerization of the material (3).

Steps in Polymerization

1. Initiation : - formation of a free radical
 - reaction of the free radical and the monomer
2. Propagation: growth or lengthening of the chain
3. Termination: termination of the polymerization

Initiation step

The first step of an addition polymerization is called “initiation”. There are two reactions involved in this step.

- a) Formation of the free radicals (polymerization activation)

An initiator molecule can become activated (changed into a free radical) by heat or a chemical reaction. Several types of chemical reactions are utilized in dentistry to form free radicals. One is a chemical reaction that begins when chemicals are mixed together. The other is a chemical reaction that is started by light (52, 53).

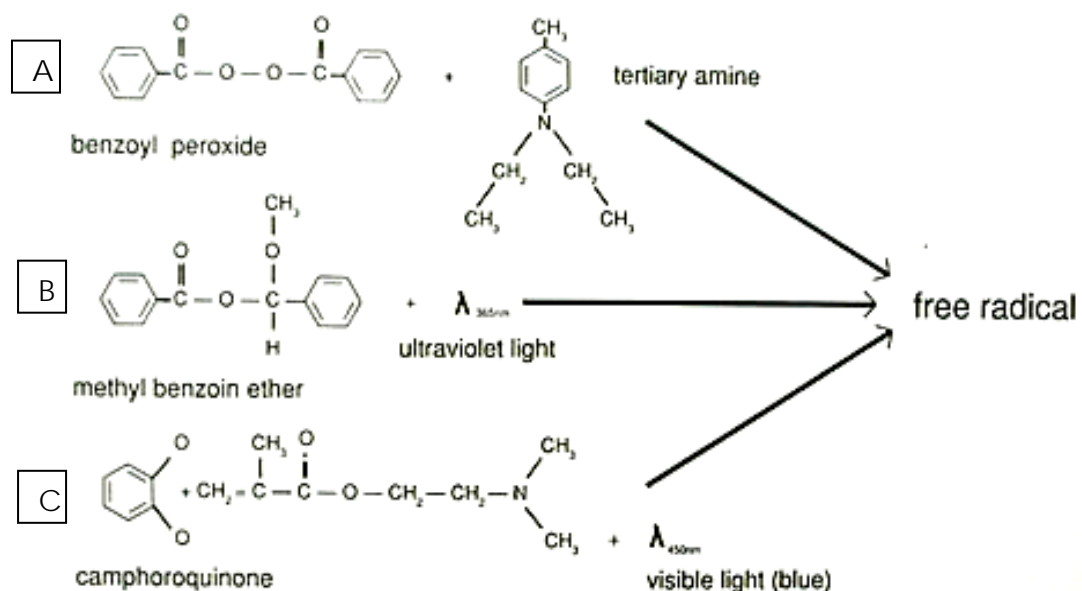


Figure 8 Routes for production of free radicals for A) chemically cured, B) ultraviolet light cured, and C) visible light cured composite resin (52).

b) Reaction of free radical and monomer

The second reaction in initiation of polymerization is for the free radical to react with the C=C of the monomer (52, 53).

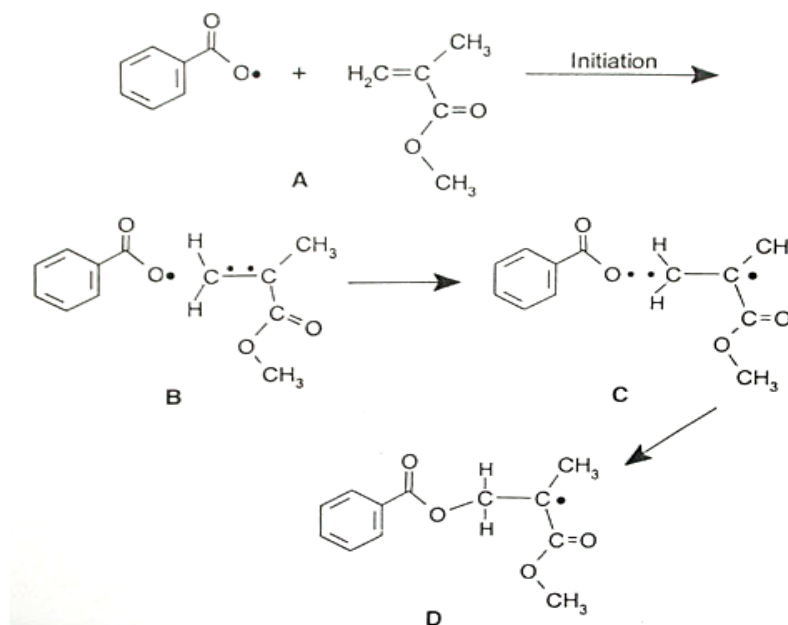


Figure 9 Initiation of a methyl methacrylate molecule (52).

The unpaired electron of the free radical approaches the methyl methacrylate molecule (A and B), one of the electron in the double bond is attracted to the free radical to form an electron pair and a covalent bond between the free radical and the monomer molecule (C and D). When this occurs, the remaining unpaired electron makes the new molecule a free radical (D).

Propagation step

The second step, the growth or lengthening of the chain. The free radical of the “initiated chain” reacts with a monomer, and the chain is one monomer longer (48). Consequently, polymerization propagation will predominantly add one molecule of monomer after the other to a growing polymer chain (54).

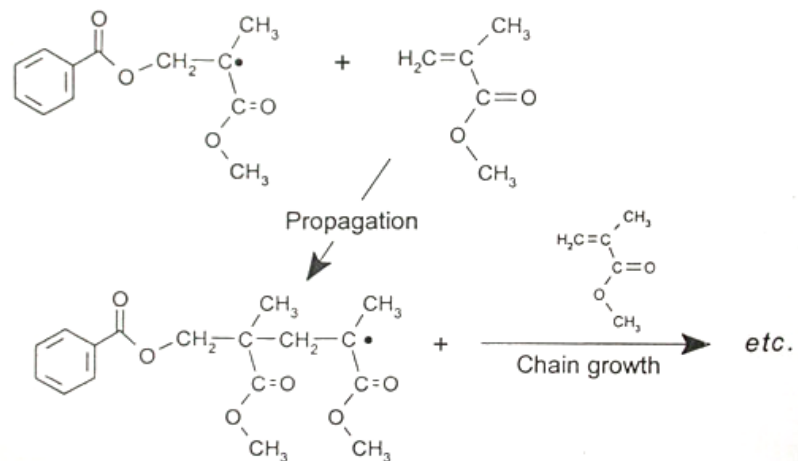


Figure 10 Propagation and chain growth (52).

As the initiated molecule approaches other methyl methacrylate molecules, the free electron interacts with the double bond of the methyl methacrylate molecule, and a new, longer free radical is formed.

Termination step

Two free radicals at the end of two growing chains may react. If they react, they will form a carbon—carbon bond as illustrated in Figure 6. No free radicals are left to continue growth of the chain. Termination of the polymerization of both chains is the result (48, 52).

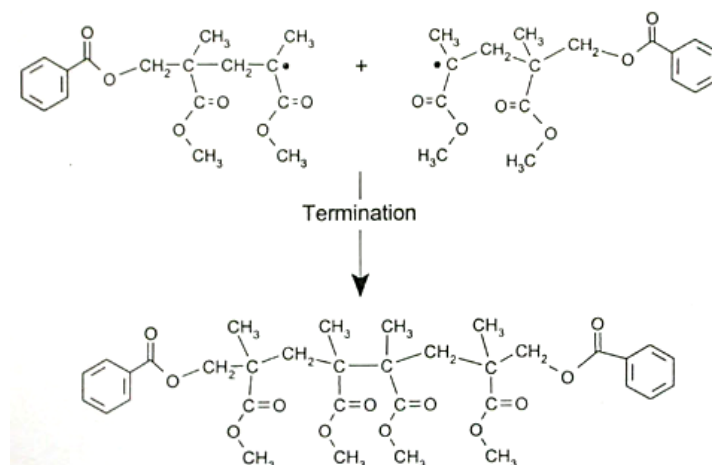


Figure 11 Termination occurs when two free radicals interact and form a covalent bond (52).

Degree of Conversion

The degree of conversion (DC%) is a measure of the percentage of carbon-carbon double bonds that have been converted to single bonds to form a polymeric resin during the polymerization reaction (3, 52). The higher the DC%, the better the strength, the wear resistance, and many other essential properties for resin performance. A conversion of 50% to 60% implies that 50% to 60% of the methacrylate groups have polymerized. However, this does not imply that 40% to 50% of the monomer molecules are left in the resin because one of the two methacrylate groups per dimethacrylate molecule could still have reacted and could be covalently bonded to the polymer structure (52). Conversion of the monomer to polymer depends on several factors, such as resin composition, the transmission of light through the material, and the concentration of sensitizer, initiator, and inhibitor. The transmission of light through the material is controlled by lamp intensity, absorption, and scattering of light by filler particles and opacifiers, as well as by any tooth structure interposed between the light source and composite (52).

The total DC% within resins does not differ between chemically activated and light activated composites containing the same monomer formulations, as long as an adequate light curing is employed (52). The previous researches had demonstrated that irradiance values of at least 300 mW/cm^2 are required to adequately cure a 2-mm-thick specimen of resin-based composite (55, 56). However, 300 mW/cm^2 might not always be adequate. Rueggeberg et al. (55) recommended an irradiance of at least 400 mW/cm^2 for 60 seconds to cure a 1-mm-thick sample of resin-based composite. The 20-second polymerization time seems to have been empirically adopted by the orthodontic community, even though the thickness of composite used in bonding orthodontic brackets to teeth is only a fraction of the suggested 2-mm maximum used in restorative dentistry.

In the clinical practice of orthodontics, time spent photo polymerizing brackets can be a major time management concern. Most manufacturers of orthodontic resin adhesives recommend a 20 seconds total polymerization time for bonding brackets (55). The increase in the duration of irradiation from 20 to 40 seconds resulted in a 0.5–2.2% increase in the final DC% for materials polymerized with the

conventional quartz–tungsten–halogen (QTH) light-curing unit and light-emitting diode (LED) light-curing unit. In addition, the higher degree of conversion might be due to the lower viscosity. Lower viscosity results in greater DC% because of increasing the mobility of molecules (57).

Setting Time

Chemically activated composite systems have setting times ranging from 3 to 5 minutes from the start of mixing. The setting time of chemically activated composite systems is determined at the time of manufacture by control of the concentrations of initiator and accelerator. However, studies show that even after a curing time of 24 hours, polymerization is incomplete and 25% to 45% of double bonds remain unreacted (3, 53). The setting time and the depth-of-cure of light-initiated materials depend on the intensity and penetration of the light beam. Polymerization is approximately 75% complete at 10 minutes after exposure to blue light, and curing continues for a period of at least 24 hours. At 24 hours, up to 30% of double bonds still remain unreacted (3, 53).

The light-cured adhesive bonded to metallic brackets was exposed to light primarily from the light-curing unit and secondarily from backscattering the artificial background surface (49). Light-curing from the edges of the bracket alters the direction of the free radical gradient and the polymerization shrinkage relative to those of the chemically cured, no-mix materials. In addition, the rapid setting reaction for the relatively thin layers of light-cured composites greatly reduces the time available for atmospheric oxygen to diffuse into the bulk resin and deactivate the free radicals produced by the photoinitiator. As a result, a better peripheral bracket sealing can be obtained with light-cured composites compared with chemically cured systems (49). For light- and dual-cured materials, the maximum light exposure is desirable. Maximum properties are generally reached about 10 minutes after polymerization; only small changes occur over the ensuing 24 hours (38).

Classification of Orthodontic Adhesives

Base upon the polymerization initiation mechanism, orthodontic adhesive may be classified according to the following groups (Table 1)

1. Chemically activated (chemically cured, autocured or self-cured)
2. Light-cured (also termed photocured)
3. Dual-cured (chemical activated and light cured)
4. Thermocured

Table 1 The classification of orthodontic adhesives

Adhesive	Polymerization initiation	Clinical handling	Properties	Comment
Chemically cured Two-phase	Mixing of the liquid and paste components	Laborious, time-consuming	Increased exposure of components to air induces oxygen inhibition. Mixing introduces defects in the form of air entrapment and formation of voids	Appeared first in the market. Representative product: Concise(3M)
Chemically cured One-phase (No-mix)	Application of liquid component on enamel and bracket base, No mixing is involved	Efficient application, limited time requirement	Limited data on degree of cure and bond strength. Inhomogeneous polymerization pattern due to sandwich technique involved in diffusion of liquid component into paste during application.	Development of these materials succeeded the two-phase systems. May not be recommended in applications where the adhesive thickness is increased, as in bonding molar tubes.

Table 1 The classification of orthodontic adhesives (Continued)

Adhesive	Polymerization initiation	Clinical handling	Properties	Comment
			Enamel and bracket sides of adhesive are more polymerized relative to middle zones	Representative products: System1(Ormco), Rely-a-bond (Reliance), Unite(3M Unitek)
Visible light-cured	Exposure to light-curing source	Permits increased working time for optimal bracket placement. Ideal for educational purposes. Time-consuming photocuring process. Decreased time by orthodontist and increased chair-time are involved. Photoactivation from the incisal and cervical edges is suggested	Degree of cure of stainless brackets bonded with light-cured adhesive is comparable to degree of cure of adhesive bonded to transparent aesthetic brackets. Bond strength has been studied extensively and supports their use	Available since the 1980s. Good alternatives to two-phase systems. Significantly more time-demanding than one-phase. Most manufacturers have marketed LC adhesives

Table 1 The classification of orthodontic adhesives (Continued)

Adhesive	Polymerization initiation	Clinical handling	Properties	Comment
Dual-cured	Initiation is achieved through exposure to light. Reaction proceeds following a chemically-cured pattern	Combines disadvantages of handling of both light-cured and chemically cured materials. The most time-consuming applications	Increased degree of cure and bond strength, but questionable clinical significance for their differences with light-cured materials	Introduced into the profession from prosthetic dentistry applications. Ideal candidates for bonding molar tubes
Thermo cured	Initiation occurs through exposure to heat	Not intended for direct bonding	Superior properties	Polymerization initiator system restricts their use to indirect bonding. Not commonly used

Chemically cured orthodontic adhesive systems have been used since the initiation of bonding in modern orthodontic history. The chemically orthodontic adhesives employ benzoyl peroxide as an initiator, which is activated by a tertiary aromatic amine such as dimethyl-*p*-toluidine or dihydroxyethyl-*p*-toluidine (3, 48, 53).

The two types of chemically cured systems are “no-mix” and “two-paste mix” (37). The two-paste adhesive, the most popular of the chemically cured systems, requires the mixing and application of two liquid bonding resins to the enamel and the mixing and application of two pastes to the bracket base. As long as the operator thoroughly mixes the two pastes and does not place brackets after the working time has expired, maximum strength can be consistently achieved (37). Two-Paste Mix Systems; Concise™, Phase II®. However, their application involves mixing the paste and the liquid components that introduces critical defects such as surface porosity and air voids in the bulk material. Any porosity, such as these air bubbles, weakens the set material and increases staining. Care must be taken to minimize these types of defects

in the final product (3, 53). Majjer et al. (58) have also commented that air entrapment behind the mesh of a metal bracket may significantly affect polymerization, because of the role of oxygen inhibition of free radical polymerization, and may produce lower bond strength between the bracket mesh and the composite material. Careful application of the material to the bracket base and/or the use of liquid-paste systems, may avoid air entrapment.

The development of the no-mix bonding resins, which are intended to minimize the mixing-induced defects and to reduce the steps required for placement of the material (3, 48, 53). The no-mix system has two components; a single liquid primer and a single paste. The primer is applied to the etched, dried enamel and the bracket base. The paste is then applied to the bracket base, and the bracket is placed on the tooth. The primer serves as the catalyst for the paste and is thus the major variable of this system. Even though the adhesive is called “no-mix”, the paste and primer are actually mixed directly on the tooth. Therefore, to achieve maximum strength, the bracket base must fit flush against the tooth surface. For a proper mix, it is also important to apply thin coatings of primer and paste to the enamel and bracket base (59). To summarize the important variables involved in using a no-mix system:

- The bracket base must be flush against the tooth surface.
- A thin coat of primer should be applied to the tooth surface and bracket base.
- A thin layer of paste should be applied to the bracket base.

No-Mix Systems; Rely-a-Bond®, Right-On®, System 1®, Unite™

Light-cure materials are the single-paste materials that are mixed by the manufacturer. Therefore, voids are minimized; a stronger is the result. Light-cure materials set when activated by a very bright light. As a result, the working time is variable and can be quite long if the operator desires. The setting time is short, because the working time of light-cure materials is determined by the user, they are widely popular and have replaced chemical-cure materials for most used (53). They offer the following benefits (37);

- Extended working time to position brackets and clean up flash.
- Ability to place archwires immediately.
- More efficient utilization of staff.

The extent of polymerization depends upon several factors: the exposure time, the photoinitiator concentration, the light intensity emitted by the curing unit at the peak absorbance wavelength of the photoinitiator, and the filler volume fraction. The highest intensity of the light source is obtained when the wavelength is 470 nm (i.e. a visible blue light). Light sources include halogen lights, plasma arc lights, argon lasers, and blue light-emitting diodes (3, 48, 53). Light-Cured Systems; Blugloo®, Eagle No Drift®, Enlight®, Light Bond®, Pad Lock®, Pro-Seal®, Transbond XT™

Several dental materials have both chemical- and light-cure capabilities. These materials are called dual-cure materials. With dual-cure materials, polymerization is started with a curing light, but material that cannot be reached by the intense light sets via the chemical-cure mechanism (53). Dual-cured systems combine the advantages of rapid initiation for photopolymerizing resin and high conversion rates for chemically cured resin. The dual-cured adhesive was found to provide significant higher bond strength compared to the chemically-cured and the light-cured materials 24 hours following activation (3).

From many developments and improvements in orthodontic adhesive formulations, the results present in the current availability of two-paste system, no-mix adhesives, and light-activated direct bonding materials. Fluoride-releasing visible-light-activated bonding agents, and more recently, adhesive precoated brackets have also been used for bracket bonding (51).

Mui B et al. (60) examined on 104 bovine teeth. The bracket were bonded with self-cured and light-cured composite, the results show that light-cured system produced higher initial bond strength than self-cured system. While, Chamda and Stein (61) showed there was no significant difference among the bond strengths achieved by the chemically-cured and light-cured systems at 10-minute, 60-minute, and 24-hour intervals.

Bonding protocol

Traditional orthodontic bracket bonding systems require the use of a three-step procedure involving three separate agents; an enamel conditioner, a priming agent, and an adhesive resin. The application of phosphoric acid for bonding orthodontic brackets has the advantage of increasing bond strength, but it can cause more enamel loss. The enamel loss during acid etching has been found to depend on the acid; the most commonly used is 37% phosphoric acid with etch times of 15 to 30 seconds per tooth. Wide variations in enamel surface loss from as little as 10 μm to 30 μm to as much as 170 μm have been reported (62). The surface enamel is permanently lost from the tooth surface during etching procedures that use acidic solutions. The adhesive resin can then infiltrate the etched and roughened enamel to establish a stable bond with the orthodontic bracket. After infiltrating the etched enamel, the adhesive resins must polymerize (63).

Self-etching primers were introduced in an effort to reduce the three-step procedure to two steps, effectively reducing chair time and increasing cost-effectiveness, and subsequently resulting in increased convenience and potentially reducing costs to the patient (23). Self-etching primers are aqueous mixtures of acidic functional monomers and other constituents. Self-etching primers have a low pH to facilitate the etching process. Because the primer and the etchant are combined, the etched tissue is simultaneously primed, eliminating the possibility of demineralized tissue that is incompletely infiltrated with resin. However, Self-etching primers do not leave a uniform frosty appearance on the etched surface, so it can be difficult to determine whether enamel surface is adequately etched (19). In a self etching primer, the active ingredient is a methacrylated phosphoric acid ester. The phosphoric acid and the methacrylate group are combined into a molecule that etches and primes at the same time. The phosphate group on the methacrylated phosphoric acid ester dissolves the calcium and removes from the hydroxyapatite. But rather than being rinsed away the calcium forms a complex with the phosphate group and gets incorporated into the network when the primer polymerizes. Agitating the primer on the tooth surface serves to ensure that fresh primer is transported to the enamel surface. Etching and monomer penetration to the exposed enamel rods are,

simultaneous. In this manner, the depth of etch is identical to that of the primer penetration. Three mechanisms act to stop the etching process; First, the acid groups attached to the etching monomer are neutralized in a similar way, as is phosphoric acid, by forming a complex with the calcium from the hydroxyapatite. Second, as the solvent is driven from the primer during the airburst step, the viscosity rises, slowing the transport of acid groups to the enamel interface. Finally, as the primer is light cured and the primer monomers are polymerized, transport of acid groups to the interface is stopped (20). Many studies have demonstrated that when self-etching primers are preferred, the degree of penetration by the adhesive to the etched enamel is less compared with the use of the conventional acid-etching technique. The more deeply the enamel surface is penetrated by the adhesive, the greater the penetration of the adhesive and the greater the risk of damage to the enamel (20, 39).

A characteristic and uniform etch pattern was observed in the resin samples of the 3-step procedure of phosphoric acid, Transbond XT primer group, revealing increased roughness and resin tags penetrating the demineralized enamel surface. The use of Transbond Plus, self-etching primer, produced a uniform etch pattern that was more conservative and less destructive to the enamel surface. Consequently, a regular resin tags distribution was observed. Enamel etching with phosphoric acid created an etch pattern characterized by a deep and uniform demineralization area. These demineralized areas were infiltrated by the resin of the priming solution, producing well-formed resin tags penetrating into demineralized surface. However, this may not be desirable clinically because there are concerns that such bonding levels may be higher than what is required for a successful orthodontic bonding (64).

Bond strength

In order to gain maximum strength from the materials being used, it is important to understand their strengths and weaknesses. In dentistry there are five types of stress that can act on restorations in the mouth (33).

These are as follows:

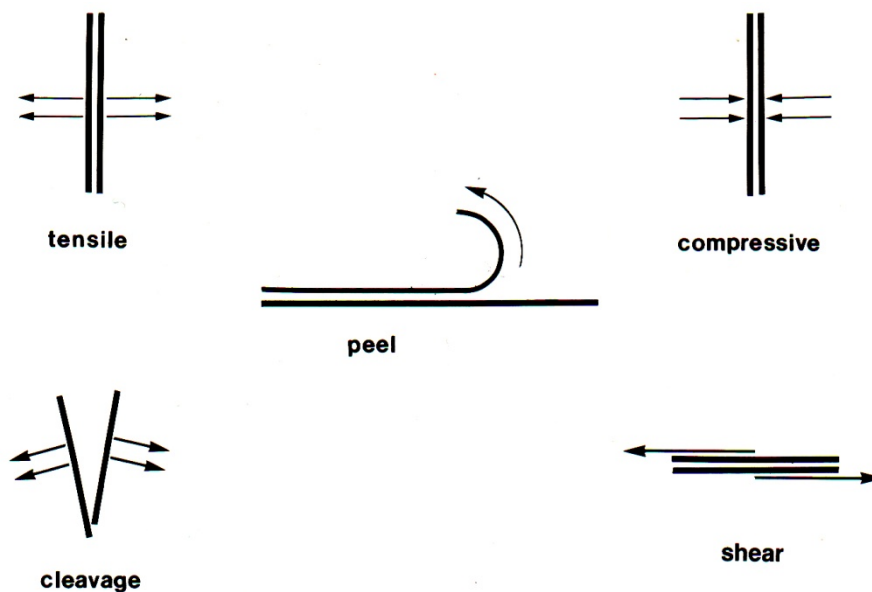


Figure 12 Types of stress that can act on restorations in the mouth

Composite luting agents exhibit great strength under almost all of these types of stress. If cleavage stress is applied, the composite cement may be overtaxed and fail. With compressive, shear, or tensile stress, the entire surface of the bond resists deformation. With cleavage forces, however, only the leading edge of the bond resists the pressure. It then is as if the entire force is concentrated on a single line, even a small force exerted on the surface area of a line becomes magnified to an infinite force per square inch. However, often very little is said about the torsional or cleavage strengths of dental materials. This is probably because standardized tests are available in dentistry for compressive, tensile and shear stress, but there does not yet exist a fully accepted standardized test in dentistry for cleavage (33).

In shear testing, the bracket is loaded by a blade in tension or compression

or wire in tension, so that the bracket slides parallel off the substrate. However, pure shear loading is difficult to achieve, and most shear testing includes components of peeling, tension, and torsion (3, 65). In tensile testing, the bracket is pulled perpendicularly off the substrate. Both shear and tensile loading modes are valid tests for studying orthodontic bonding. An adhesive system (bracket base and adhesive) cannot be selected on the basis of one test (tension, shear, or torque) alone. The various systems generally ranked differently, depending upon the test method utilized (66). The goal in bond testing should be to achieve a coefficient of variation (standard deviation /mean) in the range of 20% to 30% (3, 65).

In order for bracket bonding systems to achieve wide acceptance, they must meet certain requirements and specifications. It is only reasonable to expect a workable bonded system to be at least equal in strength and versatility to banded systems. A bonded system must be able to support shear and tensile forces in the magnitude exerted by all currently used orthodontic wires and auxiliaries which may extend into the pound range. The bonds created must maintain their initial strength for the duration of treatment, and the material used for the attachments must maintain its stability in the oral environment. The attachments themselves must be available in the same variety as are metal welded attachments (36).

The maximum bond strength recommended for successful clinical bonding is estimated to be 5.9-7.8 MPa (31).

Factor that may be influenced bond strength were; nature of the enamel surface and bracket base, thickness of the material tested (enamel to bracket base distance) which is related to seating pressure, continuity of the material under bracket, and nature of the mix such as accuracy of powder/liquid and primer/composite ratio (67).

To achieve good bonding, either mechanical retention or chemical bonds are used. Mechanical retention can be achieved either by acid etching, use of an abrasive unit (sandblasting), or by regular mechanical retention (beads or meshes) like the mesh welded to orthodontic metal brackets. Chemical bonding can be achieved by coating the surface with a silica or tin film to which a resin can be bonded (35).

Bond testing

In the majority of published articles, a value of bond strength is calculated as the quotient of the force at which debonding occurs (determined from the load drop on the mechanical testing machine) and the interfacial area of the adhesive or bracket base (3).

The units of bond strength are megapascals (MPa), kilograms per square centimeter (kg/cm^2), and pounds per square inch (lb/in^2 or psi). It is common to see bond force reported with units of Newtons (N), kilograms (kg), or pounds (lb). Bond strength is the bond force divided by the area of the bonded interface. (31, 65) However, retention can be improved by selecting a bracket with a larger bonding area (65).

Bond testing of orthodontic adhesives can be performed in vitro by using extracted teeth as substrates (clinical simulation model) or by studying the adhesive/bracket interface, for example, by debonding a bracket from a layer of adhesive retained in a plastic well (isolated interface model). Clinically, bond strength is studied indirectly by observing retention rates of various brackets and adhesives (3, 65).

Bond testing using teeth involves many variables that can affect the measured bond strength. These variables include; type of tooth (e.g., incisor, molar, human, bovine), fluoride content of tooth, disinfection and storage media of tooth before bonding, elapsed time of storage following bonding, type of loading (shear, tension, torsion, or peeling), configuration of specimen testing jig, crosshead speed of mechanical testing machine, and bonding area of the bracket. Unfortunately, no specifications (American National Standards Institute/American Dental Association or International Organization for Standardization) currently exist to standardize testing protocols for orthodontic bond strength measurements (3).

Bond Failure

Bond failure is determined using the application of force at a specific time after bond placement. In the mouth, bond failure may be the result of progressive damage to the bond over a long time period due to multiple applications of load until failure occurs such as by fatigue (44).

The examination of the bracket backings revealed that all or nearly all recessed areas between the mesh wires were filled with cement. Therefore, for the bracket interfaces, the percentage of cement covering the surface of the mesh was recorded. The failure mode analysis reported whether the failure was adhesive or cohesive. The adhesive failure occurred at either the adhesive/bracket interface or the adhesive/enamel interface, and the cohesive failure occurred within the adhesive material itself (29). Often, bond failures are a mixture of adhesive and cohesive failures. The percentages of the types of bond failure can be measured using a scale such as the Adhesive Remnant Index. This index specifies the amount of cement remaining on the tooth and bracket or band (3, 65).

Bonding to dental amalgam

Posterior teeth often have buccal class V dental amalgam restorations. Bonding of brackets or tubes when there is ample amount of sound enamel surrounding the dental amalgam restoration is not difficult. However, when the size of the restoration is extensive bonding to the dental amalgam surface is required (3).

Sandblasting the surface of the restoration, followed by use of the previously discussed adhesives, 4-META, 10-MDP/Bis-GMA, and intermediate resins, improves bonding to dental amalgam. However, the bond strength achieved is at best about half that for resin composite to etched enamel. It should also be noted that sandblasting the dental amalgam surface produces significantly bonding than that achieved with a polished dental amalgam surface. This follows from the considerable difference in micromechanical retention offered by the two surface preparation conditions. However, when compared to roughening with a diamond bur, sandblasting of dental amalgam surfaces did not produce better bonding. Roughening with a stone bur produces a surface topography on dental amalgam similar to that achieved with a diamond bur (3).

Zachrisson and Buyukyilmaz (6, 7) recently reviewed the techniques and materials available to orthodontists for bonding brackets to amalgam. Metal bonding adhesives that chemically adhere to precious and nonprecious metals alike have also been developed. 4-META polar molecule (4 methacryloxyethyl trimillitate anhydrid) that is thought to attract oxygen or hydroxyl groups in the metal layer to form hydrogen bonds.

Buyukyilmaz et al. (7) studied bond strength of orthodontic bonding to silver amalgam after subjected to aluminum oxide sandblasting using intermediate resin, either All-Bond 2 Primers A+ B or a 4-META product Amalgambond Plus (AP) or Reliance Metal Primer (RMP) and followed by concise. They found invariably occurred at the amalgam/adhesive interface. The strongest bonds were created to the spherical and lathe-cut amalgams (range 6.8 to 11.0 MPa). Bonds to the spherical amalgam were probably more reliable and the intermediate application of the 4-META resins AP and RMP generally created significantly stronger bonds to all three basic types

of amalgam products than the bonds obtained with the All-Bond 2 primers. Moreover, the effect of abrasive-particle size on bond strength to different amalgam surfaces was not usually significant. However, it is difficult, not to say impossible, for an orthodontist to differentiate between these types of amalgams in old restorations.

Gross et al. (8) study with any combination of amalgam surface treatment and intermediate resins/bonding adhesives. It would appear that the combination of C&B Metabond and Adlloy-treated amalgam to increased amalgam surface roughness provides adequate orthodontic bond strengths.

CHAPTER IV

MATERIALS AND METHODS

This research was designed to be an experimental study. It was conducted in vitro on extracted human premolar teeth.

Teeth

Two hundred and fifty freshly extracted human upper premolar teeth for orthodontic purpose were collected and stored in a solution of 0.1% (weight/volume) thymol to prevent dehydration and bacterial growth. To meet the criteria for use in the study, the teeth were selected only if they had intact buccal enamel, had no enamel hypoplasia or abnormal buccal surface anatomy that might affect the strength of the enamel, had no surface cracks from the extraction forceps, had free of buccal caries and restorations. The teeth were embedded in methy methacrylate and placed in PVC blocks (26 mm. in diameter, 15 mm. in height) similar position and direction, and exposed the buccal surface about 8 mm. (Figure 13 left) The teeth were divided into two major groups. First major group was tooth surfaces and second major group was tooth surfaces with buccal amalgam restorations, which were prepared by one operator. The preparations were cylindrical in shape (2.585 mm. in diameter, 1.5 mm. in depth) by cylindrical diamond bur 012, extended fifty percents of bracket base (5.25 mm²) and positioned in the middle location of bracket placement, remained fifty percents of the dimensions of the brackets were attached on the tooth surface. The preparations were packed incrementally with disperse alloy amalgam (Ventura, Madespa, Toledo, Spain) and burnished with a burnisher (Figure 13 right), waited for setting time at least 24 hours and then were roughly polished amalgam surface with diamond bur 012. Each group was randomly divided into five minor groups equally (25 sample per groups) for testing with five typed adhesives which were following;

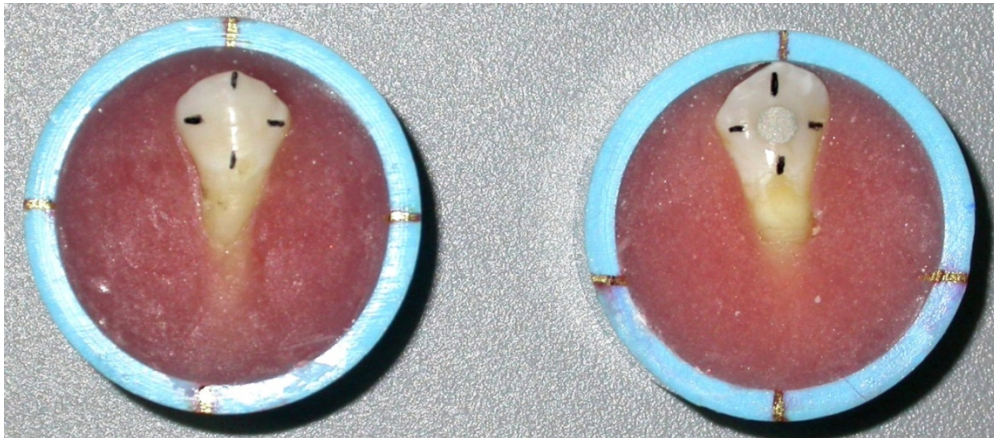


Figure 13 Specimen blocks. Left, normal tooth surfaces; Right, tooth surfaces with amalgam restorations

Brackets

All brackets used in this experiment were 0.022” x 0.028” slot pre-torque, pre-angulated edgewise stainless steel upper premolar brackets (Minidiamond, Ormesh™,Ormco Corporation, CA. USA.) that had 17-4 Stainless steel bracket body, 316-stainless steel foil/mesh base type. Total surface area of the base of each bracket was 10.5 mm². (3 x 3.5 mm.). Lot. 351-0514



Figure 14 Stainless steel premolar brackets (Minidiamond, Ormesh®, Ormco Corporation, CA. USA.)

Adhesives

Group 1 : Unite (Unite®3M Unitek, CA, USA.)
a chemically cured dimethacrylate resin

Group 2 : Rely-a-Bond (Rely-a-bond[®]Reliance, IL, USA.)

a chemically cured dimethacrylate resin

Group 3 : Enlight (Enlight[®]Ormco corporation, CA, USA.)

a low viscosity light-cured dimethacrylate resin

Group 4 : Transbond XT (Transbond XT[®]3M Unitek, CA, USA.)

a light-cured highly-filled dimethacrylate resin

Group 5 : Transbond XT + Transbond Plus Self Etching Primer

(Transbond Plus[®]3M Unitek, CA, USA.)

a light-cured dimethacrylate resin with self etching primer

Bonding

Before bonding, the enamel surfaces of each tooth were cleaned and polished for 10 seconds using rubber cup and slurry of nonfluoridated pumice. Then, the teeth were applied with 37% phosphoric acid gel (Scotchbond, 3M ESPE, USA) for 30 seconds and were rinsed thoroughly with water. Thoroughly dry etched surface with oil-and-moisture-free air until the surface appeared frosty white. The bonding protocols followed the manufacturers' instruction of each adhesive.

Unite; Brush a thin amount of primer on each tooth surface and on each bracket base. After that, the adhesive was applied on primed bracket base. The bracket was placed on the tooth and adjusted within 20 seconds. (Lot. 070618, Exp. 2009-05)

Rely-a-Bond; Apply a thin coat of primer to each elchcd dry tooth in the area which the bracket is to be bonded and the underside of each bracket base. Within 25 seconds, position the bracket to desired angulation and press firmly. (Lot. 0707032, Exp. 2009-11)

Enlight; The sealant was placed on the tooth and the bracket was bonded with the adhesive and light cure for 40 seconds. The curing time was then divided and finally the light was shone on the mesial and distal edges of the bracket. (Lot. 2778486, Exp. 2010-04)

Transbond XT; The primer was applied on the tooth. The bracket was bonded with the adhesive and light cured for 40 seconds by shining the light for 20 seconds on mesial and distal sides. (Lot.7YP/7EH, Exp. 2009-12)

Transbond XT + Transbond Plus Self Etching Primer; Apply this self-etching primer with saturated tip of applicator onto the tooth surface. Continue rubbing and swirling liquid onto enamel while applying some pressure for 3-5 seconds per tooth. Use an oil and moisture-free air source to deliver a gentle air burst 1-2 second to each tooth. The primer must be dispersed into a thin film on the tooth and the primed enamel surface should have a uniform, shiny appearance before being bonded. Then the bracket was bonded with Transbond XT light-cured adhesive. (Lot. 288407E, Exp. 2008-12)

Each bracket was pressed on the tooth surface until seated in the correct position. After seated the bracket, each tooth was checked to ensure that the material had been extruded around bracket base. An excess adhesive was then removed with a carver so that no adhesive overlapped the margin of the bracket base.

The bonding of all brackets was performed by the same operator to standardized technique as in clinical situation. The loading pressure, the force used to press a bracket on the tooth, was not measured to simulate the clinical practice. In addition, according to different viscosity of the adhesives if the same pressure was used while seating the bracket the different in thickness of adhesive layer would occur and affect the bond strength.

The light source used in this study for light-cured adhesives was EliparTM2500 (3M ESPE, USA) tungsten halogen light curing unit with a light intensity at 400 mW/cm². Before light activated each adhesive, the light curing unit was tested with a minimum output of 400 mW/cm². Light-curing time was 40 seconds by dividing the light for 20 seconds on mesial and distal sides.

Debonding Procedure

Test specimens were prepared at 23 ± 2°C. Debonding will be performed at room temperature after thermocycling; between 5 ± 2°C and 55 ± 2°C for 500 cycles.

The exposure to each bath is 20 seconds, and the transfer time between the two baths is 5-10 seconds.



Figure 15 Thermocycling machine

A steel rod with a flattened end attached to the crosshead of an Instron testing machine series IX. The rod applied an occlusogingival load to the bracket, producing a shear force at the bracket-tooth interface. The results of each test was recorded in MPa by a computer that was electronically connected to the testing machine at a crosshead speed of 5.0 mm per minute.

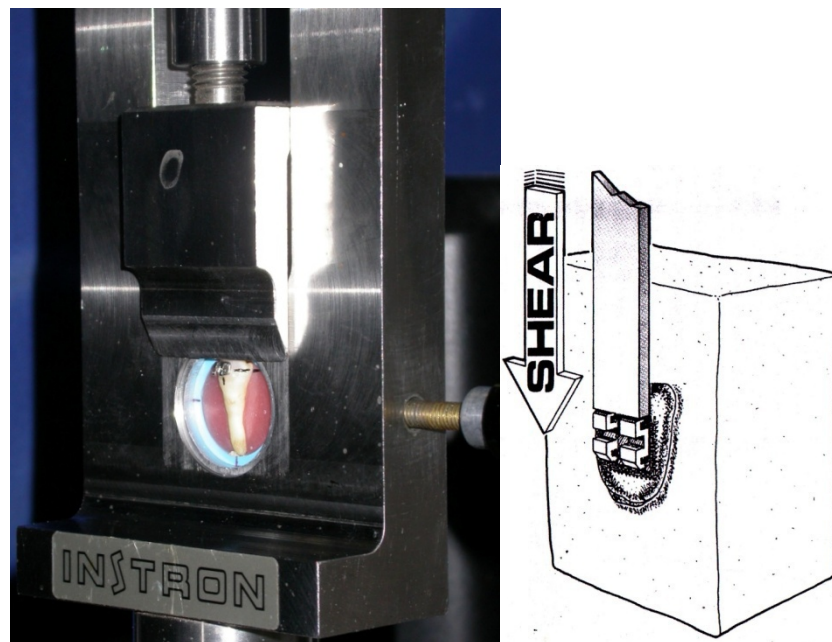


Figure 16 Instron testing machine series IX in shear testing

Adhesive Remnant Index (ARI)

After the brackets are debonded, the enamel surface of each tooth was examined under 25X magnifications of stereomicroscope to determine the amounts of residual adhesive remaining on each tooth. The adhesive remnant index (ARI) of

Artun and Bergland (1984) (28) used to quantify the amount of remaining adhesive using the following scale:

Score 0 = no adhesive left on the tooth

Score 1 = less than half of the adhesive left on the tooth;

Score 2 = more than half of the adhesive left on the tooth;

Score 3 = all adhesive left on the tooth, with a distinct impression of the bracket mesh.



Figure 17 Stereomicroscope used for residual adhesive examination on the bracket base

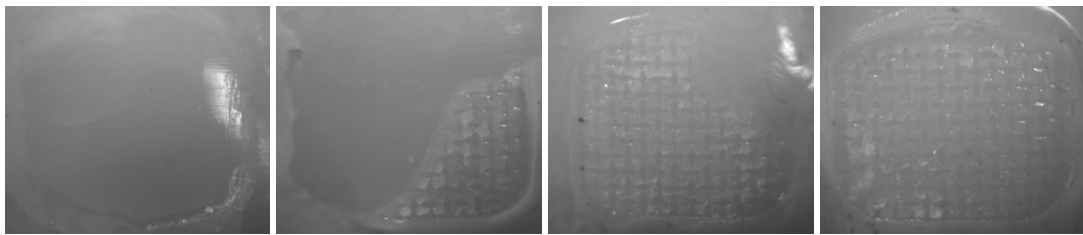
**Score 0****Score 1****Score 2****Score 3**

Figure 18 Samples of adhesive remaining on the tooth surface after debonding from stereomicroscope magnification X25; showed ARI index score 0 to 3 from left to right

Statistical Analysis

Statistics included the median, 25-75% quartile range, and minimum-maximum values of shear bond strength were calculated for each of the test groups. The data of the shear bond strength were tested for normality with the Kolmogorov-Smirnov method.

The differences in shear bond strength between tooth surface and tooth surface with amalgam restoration and the differences in shear bond strength among various adhesives were evaluated. The nonparametric Kruskal-Wallis test was used to determine whether significant differences existed between them. Then multiple comparisons by the Student-Newman-Keuls method was used to compare and identify which of the groups were different.

For failure site analysis, the descriptive statistic was used to explain the site of bond failure among various types of adhesives.

The overall test was interpreted for significant difference at p-value < .05

CHAPTER V

RESULTS

The shear bond strength

Shear bond strength values of five adhesives on tooth surfaces and tooth surfaces with amalgam restorations are presented in Figure 19

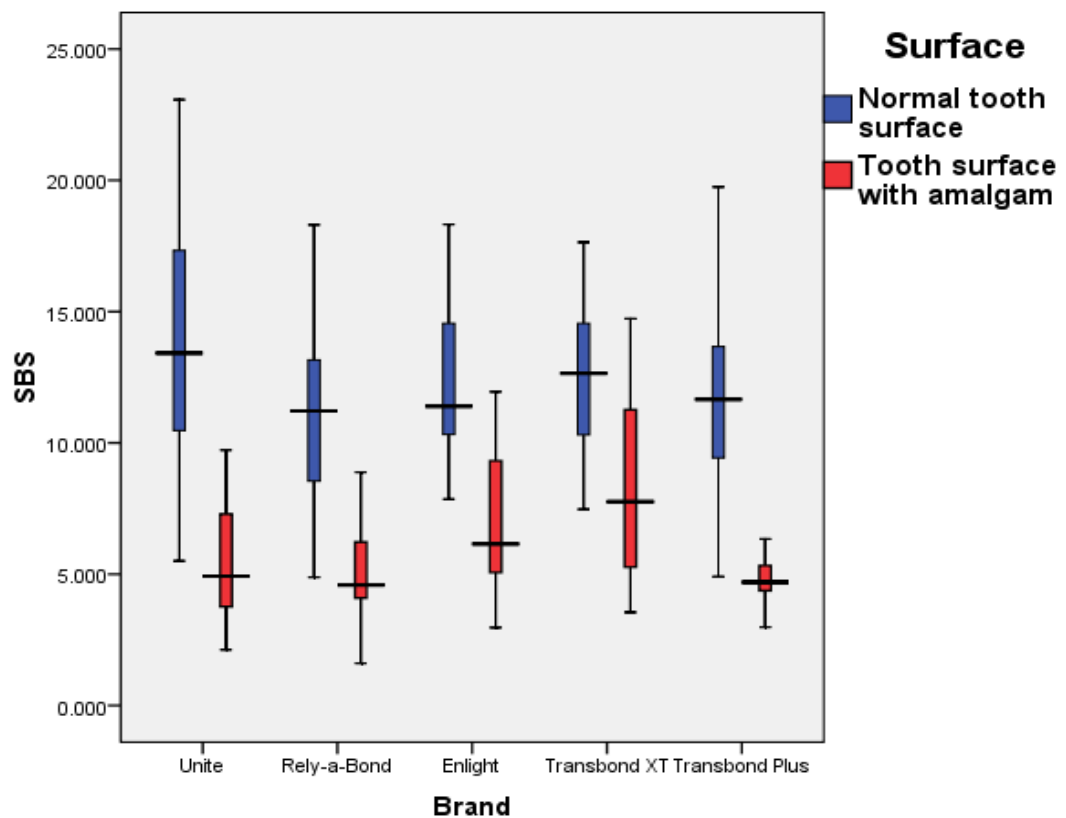


Figure 19 Shear bond strength (SBS) values of five adhesives on tooth surfaces and tooth surfaces with amalgam restorations

The median, 25%-75% range and minimum-maximum values for shear bond strength of various orthodontic adhesives are shown in Table 2, 3. The data analysis of all shear bond strength values is done in Megapascals (MPa).

To isolate the group or groups that differ from the others use a multiple comparison procedure. All Pairwise Multiple Comparison Procedures (Student-Newman-Keuls Method); same letter means no significant difference.

Table 2 Median, 25% and 75% percentile, minimum and maximum SBS values of five adhesives on tooth surfaces (MPa)

Brand	n	Shear bond strength (MPa)				
		Median	25%percentile	75%percentile	Minimum	Maximum
Unite	25	13.425 ^a	10.274	17.601	5.505	23.074
Transbond XT	25	12.653 ^a	10.281	14.607	4.886	18.309
TransbondPlus	25	11.659 ^a	9.382	13.981	7.859	18.321
Enlight	25	11.397 ^a	10.075	14.661	7.474	17.643
Rely-a-Bond	25	11.221 ^a	8.425	13.164	4.911	19.744

Table 3 Median, 25% and 75% percentiles, minimum and maximum SBS values of five adhesives on tooth surfaces with amalgam restorations (MPa)

Brand	n	Shear bond strength (MPa)				
		Median	25% percentile	75%percentile	Minimum	Maximum
Transbond XT	25	7.760 ^a	5.076	11.488	2.12	9.728
Enlight	25	6.150 ^a	5.061	9.322	1.604	8.881
Unite	25	4.924 ^b	3.688	7.441	2.967	11.946
TransbondPlus	25	4.695 ^b	4.282	5.391	3.55	14.729
Rely-a-Bond	25	4.585 ^b	4.071	6.243	2.984	6.341

Shear bond strength of various orthodontic adhesives on tooth surfaces were greater than on tooth surfaces with amalgam restorations. The median shear bond strength on tooth surfaces; Unite (median=13.43 MPa), Transbond XT (median=12.65 MPa), Transbond Plus (median=11.66 MPa), Enlight (median=11.40 MPa) and Rely-a-Bond (median=11.22 MPa) in order, while The median shear bond strength on tooth surfaces with amalgam restorations; Transbond XT (median=7.76 MPa), Enlight (median=6.15 MPa), Unite (median=4.214 MPa), Transbond Plus (median=4.70 MPa), Rely-a-Bond (median=4.59 MPa), The results of Kruskal-Wallis test indicated there was statistically significant greater SBS on tooth surfaces than tooth surfaces with amalgam restorations, and there were statistically significant difference in SBS among various adhesives on tooth surfaces with amalgam restorations. However, there were no statistically significant differences in the SBS among various adhesives on tooth surfaces at p-value < .05. The Student-Newman-Keuls method indicated that the SBS of brackets bonded on the tooth surfaces with amalgam restorations using Transbond XT (median=7.76 MPa) and Enlight (median=6.15 MPa) were statistical

significantly greater than bracket bonded using Unite (median=4.92 MPa), Transbond Plus (median=4.70 MPa) and Rely-a-Bond (median=4.59 MPa)

Site of Bond Failure

Failure modes and the frequency distribution of adhesive remnant index with ARI score are presented in figure 20

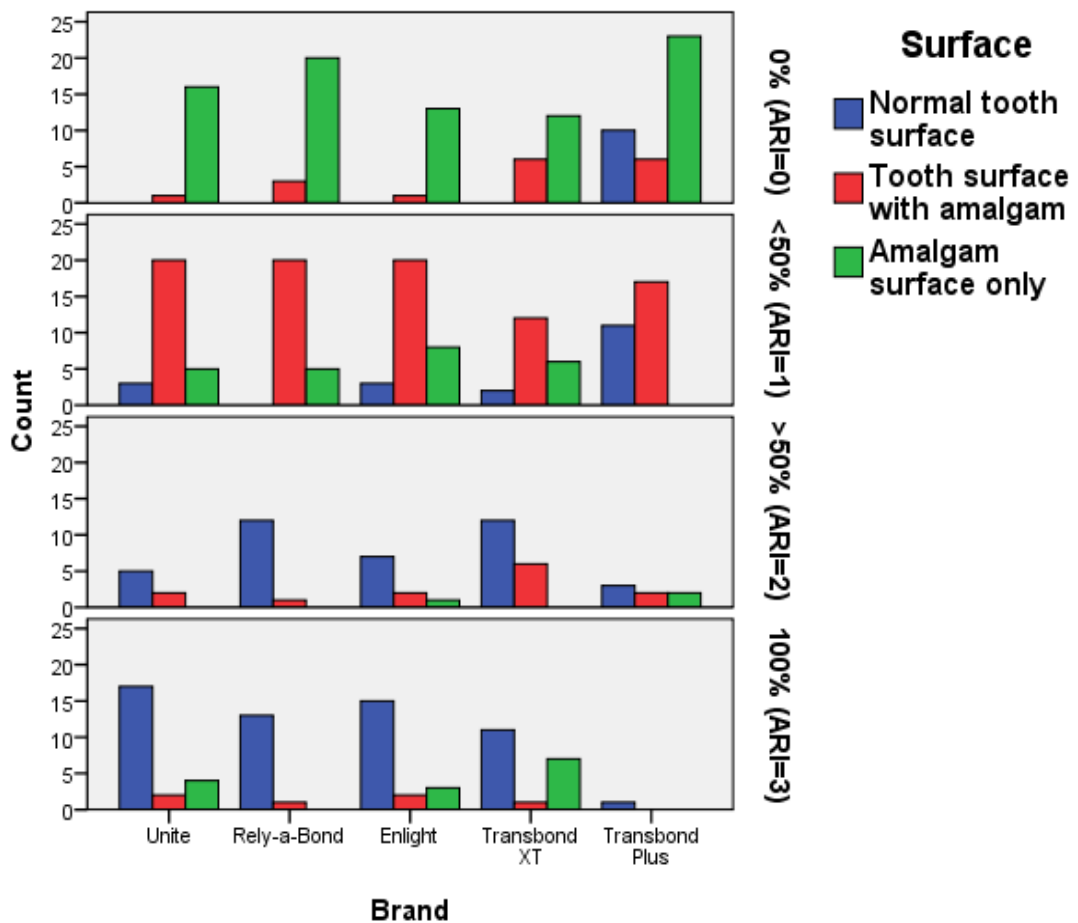


Figure 20 Frequency of ARI of five adhesives on normal tooth surfaces, tooth surfaces with amalgam restorations and amalgam restorations

The predominant modes of bracket failure on tooth surfaces more than 88% of samples were left on tooth surfaces more than 50-100%, while more than 84% of samples in Transbond Plus group left on tooth surfaces less than 50%. The

predominant modes of bracket failure on tooth surfaces with amalgam restorations more than 72% of samples left on surface less than 50%.

However, examination on amalgam restorations as total surface area only by using ARI scores in green bar charts. There were found that no adhesive remained on amalgam surfaces in most of samples as ARI=0 (Unite: 64%, Rely-a-Bond: 80%, Enlight: 52%, Transbond XT: 48%, Transbond Plus: 92%)

CHAPTER VI

DISCUSSION

Recently, the clinical use of adhesives for direct bracketing was widely advertised by several manufacturers. It is interesting that whether the development of new commercial bonding materials, having greater SBS and other properties than those previous adhesives. However, the literature contains large number of publications on in vitro bond strength testing of materials used in orthodontics. The results are often quoted by manufacturers to support their products. Little attention has been paid to the detail of the test procedures used. In addition, a review of literature revealed a large variation in materials and methods used for bond strength testing in orthodontics making comparison of paper difficult and often impossible (storage time before debonding, thermocycling, debonding device, bonding area, differences in bracket mesh etc.) Therefore, studies determining the bond strength are important mainly for their relative values and numerical comparisons are not always possible and such a fact may explain the differences observed (18, 68).

Materials and Methods

Teeth selection

Bond testing of orthodontic adhesives can be performed in vitro by using extracted teeth as substrates. A great variety of teeth have been used, but the most common were human premolar teeth (68). Although this experiment would like to simulate clinical model of such a tooth surfaces with amalgam restorations that almost commonly found at buccal surfaces of molar teeth, premolar teeth were common choice in extraction orthodontic cases and facilitated in collection of these teeth as samples. In addition, to fit the tooth into tested PVC block, each

apical root of tested tooth was cut not exceed than apical 1/3, avoiding any crack or fracture line propagation on tooth surfaces.

According to the usually designed brackets, they may be a tendency to press the bracket into contact with the highest point of curvature of enamel surfaces, squeezing out most of adhesive from this area. A poorly bonded bracket to tooth assembly due to the use of a bracket with improper curvature for the particular tooth is seen (36). To avoid this situation and to reduce variation of contours and emergence profiles between upper and lower premolar teeth, only upper premolar teeth were chose, and upper premolar brackets were used in our study. Although, the bracket form seems to play a part in bond failure, bracket bases were contoured. Nevertheless, the variation in tooth morphology might be lead to different thicknesses of adhesives (39).

The premolar teeth in this study were stored in a solution of 0.1 % (weight/volume) thymol at room temperature as suggested by Bishara et al. (69) to prevent dehydration and bacterial growth until the time of the experiment. Therefore, the storage times varied among the teeth in period of 3 months.

Brackets

All brackets used in this study were 0.022” x 0.028” slot pre-torque, pre-angulated edgewise stainless steel upper premolar brackets with 316-stainless steel foil/mesh base (Mini Diamond, Ormco Corporation, CA. USA.). They are commonly used in Department of Orthodontics, Mahidol University and widely used in many clinics.

Bonding

All these adhesives in our study are commonly used in Orthodontics Department, Mahidol University. Tested adhesives varied in curing systems, a chemically-cured and light-cured; bonding protocols, three-step and self etching primer; consistency which depended on manufacturers’ formula.

There were five types of adhesives. A chemically cured dimethacrylate resins, Unite (3M Unitek, USA.) and Rely-a-Bond (Reliance, IL, USA.); a low viscosity light-cured composite resin, Enlight (Ormco corporation, USA.); a light-cured highly-

filled composite resin, Transbond XT (3M Unitek, USA.); a light-cured highly-filled composite resin adhesive with self etching primer, Transbond XT + self etching primer as Transbond Plus (3M Unitek, USA.).

However, the bonding methods of each adhesive in this study were done according to the manufacturer's instructions. The bonding of all brackets was performed by the same operator to standardized technique as in clinical situation. The bracket placement position was marked before embedded each tooth in acrylic block. Each bracket was firmly pressed until completely seated in correct position that excessive adhesive extruded around bracket base in order to obtain the thinnest adhesive layer as much as possible. Then, excessive adhesive was removed, so no adhesive overlapped on the margin of the bracket base. The loading force for pressing a bracket on the tooth that used in this study was not measured to simulate the clinical practice. In addition, according to different viscosity of the adhesives with the same pressure was used while seating the bracket, the different in thickness of adhesive layers may occur and effect to the bond strength.

Generally, adhesive industry recognizes that thick adhesive layers give weaker joints than the thin ones. A number of reasons have been postulated for this, one of which is the probability of a greater number of imperfections such as voids and cracks in thicker layers. Internal stresses developed during polymerization of the adhesive will concentrate about these imperfections, increasing the likelihood of early failure under load related to the number and size of the imperfections. Another reason postulated for the weakness of thick adhesive layers is that they are more likely to become deformation than thin ones and thus will fracture later. Another consideration, the adhesive layer needs to be continuous, since a break in continuity of the adhesive layer could also result in a weak joint, which disastrous fractures through the adhesive may be propagated under relatively light loads. "Starved" joints may also be produced when bonding orthodontic brackets to teeth (36).

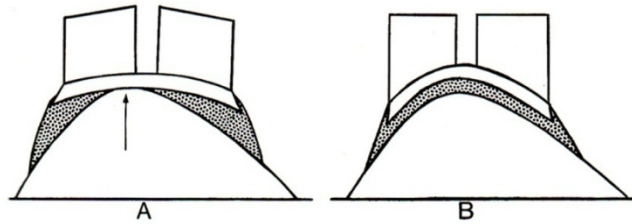


Figure 21 A, Discontinuity of adhesive layer caused by contact between projections both surfaces results in a weak “starved joint”. Regions of discontinuity may act as initiation points for propagation of fracture under stress; B, Stronger adhesive joint with continuous, thicker layer of adhesive (36).

Thickness of an adhesive layer under a bracket may be important for both final tooth position and bond strength. With increasing use of pre-adjusted brackets whose bases compensate for the differing thickness of teeth, it is important to ensure a consistently even layer of composite is placed under each bracket to take full advantage of the bracket design and to avoid the need for compensatory bends to be placed in the archwire (70). Evan and Powers (71) found that an increased layer thickness would result in lower bond strength, differences in film thickness may have also influenced differences between these composite resins.

Testing protocol

The characterization of bond strength tests can be made according to whether the mode of load application; shear, tension, or torsion. Use of shear loading has been very popular due to the relative simplicity of the experimental configuration and the presumably increased reliability of simulating debonding that occur from occlusal forces during treatment (72, 73). The values of bond strength have been calculated as the quotient of the debonding force and the area of the bracket base, thus neglecting any stress concentrations (3).

The discrepancy between statistically significant differences found for debonding forces and mean bond strengths may be attributed to several factors; the surfaces of bracket bases in contact with the adhesive deviate from an idealized

rectangular shape, there are variations in stress concentrations associated with differences in morphology and interfacial characteristics of the bracket-adhesive complex, significant differences in adhesive thickness can occur for smooth and rough bracket bases, resulting in inhomogeneous load application during testing, and several adhesives are tested with the same group of brackets, differences in rheological properties of the adhesives can lead to variation in the amount of microstructural porosity that is expected to be important for brittle failure(3).

The rate of force application as determined by the cross-head speed of testing machine is one of parameters that show large interstudy variation in dentistry. The typical range for shear bond strength (SBS) testing is 0.5 to 5 mm/min. The ISO Technical Report 11405:2003 “Guidance on testing of adhesion to tooth structure” recommends the standard rate of loading bonded specimens to be 0.75 ± 0.30 mm/min cross-head speed. However, this report does not specifically address the issue of orthodontic bond strength testing. A cross-head speed of 0.5 mm/min may be the most commonly used although this value lacks correspondence to clinical conditions (74). Fox et al. (68) suggested a crosshead speed of 0.1 mm/min to be included in a protocol for future bond strength testing in orthodontics. Hara et al. (75) concluded that cross-head speeds of 0.50 and 0.75 mm/ min result in more adhesive fractures, and are therefore preferable in SBS tests. They further stated that relatively high cross-head speeds develop abnormal stress distributions during the shear test, including cohesive failures in tooth substrate or in resin-based composite which would influence the bond strength values achieved. The viscoelastic nature of dental adhesives suggests that SBS values and failure modes could be affected by the rate of stress application. Slower crosshead speeds could allow an extended recovery period during which stress and strain are compensated for by the elasticity of the bonding agents, at lower speeds the resin behaves like a viscous material, deforming more as increased pressure is applied, with a resultant increase in SBS values. Conversely, the potential for SBS also exists with faster crosshead speeds. At more rapid crosshead speeds, the resin may perform as a brittle solid, with increased energy directed toward fracture of the specimen rather than molecular deformation and fracture (76, 77). Bishara et al. (78) determined the effect of changing the crosshead speed of the testing machine on SBS of orthodontic brackets to enamel while standardizing all the other variables, indicated

crosshead speed 0.5 mm/min developed more SBS values significantly than and 5.0 mm/min.

While the relatively slow crosshead speed typically used for bond strength tests does allow some self-adjustment of the experimental configuration during loading, it is expected that adhesive bond failures occur at much more rapid loading rates under clinical conditions. Moreover, viscoelastic behavior that is absent *in vivo* may occur at low strain rates for the adhesive; this is an important area for future investigation (3). However, Lindemuth and Hagge (79) found that no significant differences in SBS values of different crosshead speed at 0.1, 0.5, 1.0, 10.0 mm/min and failure mode on enamel. In the study of Klocke and Nieke (74), median ARI scores were identical for all experimental groups, indicating no influence of cross-head speed on the bond failure pattern in orthodontic SBS testing. In conclusion, the study showed that variation in cross-head speed between 0.1 and 5 mm/min do not have a significant influence on debonding forces in orthodontic bond strength testing. Furthermore, no influence of cross-head speed on remnant adhesive and, hence, bond failure pattern was observed.

Moreover, Klocke and Nieke (80) showed the differences in shear stress generated in the adhesive layer also resulted in significantly different shear stress at bond failure. SBS recorded dropped by 49.3% when debonding forces were moved from a position close to the enamel to the ligature groove and additional decrease in SBS was found merely 41.5% when forces were applied to the bracket wings. Therefore, debonding force location needs to be standardized in order to allow for interstudy comparisons. The validity of this suggestion is emphasized by the results of the study which showed that differences in force location due to bracket design will invariably lead to differences in SBS measurements.

However, in our study, cross-head speed at 5.0 mm/min was used as many recent studies (17, 21, 23-27, 81, 82). Debonding force was performed at bracket-tooth interface, because it was easily adjust, accurately repeat testing position for all samples and reducing bracket variation effect.

Shear bond strength

This investigation found no significant difference in SBS values among various adhesive on tooth surfaces, whether chemically-cured or light-cured included self-etching adhesives that agreed with previous reports (10, 11, 15, 16, 19, 22). These observation suggested that bond strengths of all adhesives in this study are sufficient to withstand normal orthodontic forces between 5.9 to 7.8 MPa as recommended by Reynold (31) Chamda and stein (61) concluded that the bond strengths for the chemically-cured system were initially low, but these increased with time. The light-cured sample displayed initial bond strengths of sufficient magnitude to withstand the immediate application of orthodontic forces, and these bond strengths also increased with time.

However, some studies reported self-etching primer and adhesive agents have substantially lower bond strength compared with conventional acid-etching and bonding systems. Bishara et al. (23) reported that self-etching primer plus Transbond XT provide significantly lower but clinically acceptable shear bond strength when compared with a conventional etching and primer technique before bonding brackets with Transbond XT adhesive paste. Grubisa et al. (83) found significantly higher bond strengths were seen in 35% phosphoric acid (15 seconds) plus Transbond XT than self-etching primer plus Transbond XT and phosphoric acid (15 seconds) plus Enlight bonding resin. However, the mean SBS of self-etching primer plus Transbond XT were not significantly different from Enlight.

The strength of cured adhesive depends on its composition, the degree of conversion, and the length of the polymer chain. Any unreacted resin monomer remaining in the adhesive might alter its mechanical properties. Evaluating the mechanical properties of an adhesive resin is therefore important in predicting the bonding ability to enamel (84).

Moreover, the SBS values of different adhesives on tooth surfaces with amalgam restorations found only light-cured adhesive groups: Enlight and Transbond

XT showed greater SBS values than chemically-cured adhesive groups: Unite, Rely-a-Bond and Transbond Plus.

Although, many previous studies indicated no significant difference between the bond strengths achieved by the chemically-cured and light-cured systems or between bonding with 3-step and self-etching protocols when bonding on tooth surfaces, tooth surfaces with amalgam restorations in our study had enamel surface for micro-mechanical interlocking with adhesive reduced to 50% or about 5.25 mm². MacColl et al. (43) concluded that no statistically significant difference in SBS values existed between the larger base sizes, which indicated that shear bond strength is independent of surface area between 6.82 and 12.35 mm² as in range of bracket base area on tooth surfaces in our study as 10.5 mm². However, a reduction in bond strength was associated with the reduction of base surface area from 6.82 to 2.38 mm². It is possible that when reduced the bracket base area, may be affected SBS values.

In addition, most previous studies in bond strengths of composite resins to amalgams have used water storage at 37°C for 24 hours. However, in our study debonding were performed after 5° to 55° thermocycling, temperature changing in oral cavity simulation model, provide interesting information on differences in coefficient of thermal expansion and temperature-dependent degradation of the bonds. (6) The coefficient of thermal expansion of amalgam ($25 \times 10^{-6}/^{\circ}\text{C}$) is greater than enamel ($11.4 \times 10^{-6}/^{\circ}\text{C}$), resulting in more expansion and contraction in amalgam than enamel (85). Therefore, the different in coefficient of thermal expansion of enamel and amalgam when subjected to temperature changing may be affected SBS values.

Bond failures that occurred at the amalgam/adhesive interface were those that had low bond strengths. Conversely, good bond strengths were associated with amalgam fractures on debonding. A dilemma exists in that what normally would be considered adequate orthodontic bond strength to enamel may be excessive for amalgam. In addition, future studies in orthodontic bonding to amalgam should concentrate on methods that provide adequate bonds to withstand occlusal forces while not damaging the amalgam restorations (8).

However, all previous SBS studies of bracket on amalgam restoration were tested on amalgam rods or all bonded area were on amalgam surfaces (5-7), therefore,

SBS were low if without intermediate resins/bonding adhesive enhancers for provided chemical bond with metal (6-8) or using special treating materials to amalgam surfaces (5). While, in our study was investigated whether recent adhesive materials sufficient to provide SBS values on tooth surfaces with partial amalgam restorations without intermediate resins/bonding adhesives enhancers and using simple-cost effective amalgam surface treatment.

The site of bond failure

Our results of ARI indicated that brackets bonded both on tooth surfaces and tooth surfaces with amalgam restorations showed possible bond failure mode at the enamel/adhesive interface, the bracket/adhesive interface, and within the adhesive as cohesive failure. Although, some SBS values of samples in our study were obtained more than the maximum bond strength that should be less than the breaking strength of the enamel which is about 14 MPa (86), there were no damage within enamel surfaces and between enamel surfaces with amalgam restorations.

The bracket failure at each of the two interfaces has its own advantages and disadvantages. As an example, a bracket failure at the bracket/adhesive interface is advantageous since it leaves the enamel surface relatively intact; however, considerable chair time is needed to remove the residual adhesive with the added possibility of damaging the enamel surface during the cleaning process (62). Conversely, when brackets fail at the enamel/ adhesive interface, less residual adhesive remains. This might be advantage to the clinician because it will require less time to clean the teeth after debonding but the enamel surface can be damaged when failure occurs in this mode if bond strength were great (25, 87).

Most of debonded brackets on tooth surfaces occurred at the bracket/adhesive interface as ARI 2 and 3 which remained adhesive remnant left on tooth surfaces more than 50-100%, except Transbond Plus occurred at the enamel/adhesive interface.

Joseph and Rossouw (88) suggested that polymerization of chemically-cured adhesive (System One) has been more effective and retention also been greater, this

could be the reason for the higher SBS values and the greater percentage of mixed failures (90%). When high shear bond strengths are obtained, the process of debonding may exert some extra influence on the attained site of failure. High shearing forces induce a fracture plane that would propagate through the union, at the resin/bracket area, increase the number of resin/enamel and mixed failures. So enamel prism fractures may also be observed in this group.

Jou et al. (89) studied Light Bond (LC composite resin), found 70% of the failures were at the bracket /adhesive interface. This is probably because of incomplete polymerization of the resin just below the metal base of the bracket (90). The inability of visible light to cure material behind the bracket mesh may be responsible, in part, for the site of failure. Polymerization of light-curing materials for orthodontic bonding, even with longer illumination times, does not result in the same degree of polymerization that is obtained by direct illumination (91).

Owen et al. (29) evaluate the site of bond failure for two visible light-cured composites, Transbond XT and Enlight by using light microscopy. The results of this study demonstrated ARI were similar with the mean values at about 2, which indicates that more than half of the adhesive remained on the tooth.

Bishara et al. (17) studied the SBS values of the brackets that were bonded with Transbond XT with conventional and self-etch primer in vitro finding that the use of a self-etch primer to bond orthodontic brackets to the enamel surface resulted in a significantly lower than the use of conventional. However, the comparison of ARI scores indicated that there was significantly more residual adhesive remaining on the teeth that were treated with the new self-etch primer than the conventional adhesive system.

While, most of debonded brackets on tooth surfaces with amalgam restorations were occur within the adhesive as ARI 1.

In addition, more than 50% of all specimens had no adhesives remained on amalgam surfaces. The different in bonding mechanism between adhesives on the enamel and the amalgam seem to be the possible reason why SBS values of adhesives on tooth surfaces were significantly higher than on tooth surfaces with amalgam restorations. Although, the bond strength on amalgam surfaces in this investigation was not as strong as on enamel surfaces, some specimens

demonstrated the residual adhesive remained on all of amalgam surfaces as ARI = 3. However, there was no intermediated resin or bonding adhesive enhancer which provided chemically bond, so, it might be occurred from the mechanical interlocking between rough amalgam surfaces with diamond bur and adhesives. Sperber et al. (5) found that the topography of the surface of amalgam restoration is highly conducive to the promotion of any form of mechanical interlocking of bonding resin. It appears that surface preparation played a more significant role in the determination of SBS to amalgam than did the type of resin used for bonding. However, it was evident that alterations in surface topography were highly effective in altering bond strength.

Moreover, most of specimens that had residual adhesive ($ARI \geq 1$) were from Enlight and Transbond XT, light-cured adhesives with 3-step procedure. The reason in bonding of light-cured groups overcame chemically-cured groups may be occurred from the better in polymerization. The application of catalyst or initiator in the primer composition of no-mix chemically-cured may not be completely contacted the paste composition, especially, in some deep grooves on rough amalgam surfaces to initiate the polymerization. Furthermore, the different in viscosity of adhesives was also another factor in different SBS values. Under the same seating force, the low viscosity adhesive spread more extensively than higher one, causing to form the better mechanical bond. However, the cause of lower bond strength of self-etching primer group on amalgam surfaces than three-step procedure was still unclear in mechanism, the further study should be taken.

Nevertheless, clinicians should also remember that this was an in vitro study, the results are not necessarily the same as those that would be obtained in the oral environment. It needs to take into consideration to the other properties of the adhesive before using them. In vitro, bracket bonding is performed under ideal conditions. On the other hand, in vivo, there are many factors that are difficult to control. Sometimes, contamination on enamel surface is unavoidable.

Clinical Implication

The present findings suggest that

- SBS values on tooth surfaces of all adhesives in this study; Unite, Rely-a-Bond, Enlight, Transbond XT and Transbond Plus were clinically acceptable levels in following the manufacturers' instruction of each adhesive.
- In case of bonding brackets on tooth surfaces with amalgam restorations, not exceeding a half of the bracket base area. It should be bonded by rough surface of the amalgam with diamond bur and choose adhesives that provide appropriate bond strength such as the light-cured adhesives with 3-step bonding protocol; Enlight and Transbond XT. Instead of using intermediate resins/bonding adhesives enhancers, chemical-bond intermediate materials or using other surface prepared methods to increase mechanical interlocking.
- According to the ARI scores, adhesives remnant on tooth surfaces with amalgam restorations that need to remove or enamel-amalgam crack and fracture may less than bonding on tooth surfaces.

Further Study

Although our study obtains more knowledge about SBS of recent adhesives, it should be tested more types of adhesives, as the other new generation of orthodontic adhesives. Moreover, it should be compared them with acrylic resin that have chemical bond, improves bonding to dental amalgam restoration. Including, tested in the other positions of amalgam restoration is also one interesting field that should be taken.

CHAPTER VII

CONCLUSION

- SBS of all adhesives in this experiment on tooth surface were statistically significantly greater than tooth surface with amalgam restoration.
- No statistically significant difference in SBS among various types of adhesives in this study on tooth surface. All of them showed clinically acceptable SBS levels.
- Light-cured adhesive with 3-step bonding protocol groups; Enlight and Transbond XT on amalgam restoration showed statistically significantly greater SBS than compared with the others.
- The bond failure on tooth surface and tooth surface with amalgam restoration, All of adhesive in testing except, Transbond plus were different mode of failure, Failure at the bracket/adhesive interface were predominant on tooth surface, failure within the adhesive were predominant on tooth surface with amalgam restoration.
- Transbond plus were not different in failure mode compared with tooth surface and tooth surface with amalgam restoration, the predominant mode of failure were occurred within the adhesive and at enamel or amalgam/adhesive interface.

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