Shear Bond Strengths of two new Self-Etching Primers

By

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1 Introduction

Buonocore introduced the use of an acid etch in dentistry in 1955. Newman (1965) used the acid etch technique to bond orthodontic brackets to teeth ultimately eliminating the need for banding of teeth, which allowed clinicians to more efficiently treat patients with greater patient comfort, elimination of pretreatment tooth separation, improving oral hygiene and esthetics, and reducing chair time. (Buonocore, 1955; Newman, 1965)

Dental bonding agents consist mainly of three components: (1) etchant (2) primer and, (3) adhesive. These ingredients can be either contained separately or within a single package. Over time, the procedure for bonding has constantly evolved. In the mid to late 1980s, the 4th generation bonding agents consisted of the three components packaged separately which were called the *multiple-bottle bonding agent.* This led to the more familiar 5th generation bonding agents of the 1990s where the primer and adhesive were placed as a *one-bottle system.* (Kugel & Ferrari, 2000; Farah & Powers, 2004) Currently, *self-etching primers (SEPs)* are the 6th and 7th generation bonding agents. The 6th generation self-etching primers still require mixing of the primer and adhesive components with no separate etching required. (Farah & Powers, 2004; Powers *et al*, 2006) The latest 7th generation self-etching primers are single component bottles that requiring no mixing or etching. (Farah & Powers, 2004)

New bonding agents are continually being tested, released and marketed towards restorative dentistry for bonding composite resins to dentin and enamel. A standardized approach in laboratory testing of new self-etching primer systems is quite difficult to achieve due to variations in methodologies and techniques. However, it is still of importance to initiate preliminary *in vitro* studies of new bonding agents to provide clinical insight as to how they may actually perform. Bonding agents should possess adequate bond strength to prevent debonding during normal occlusal function, but should not be so strong that once treatment is completed, debonding them would damage the enamel. (Cehreli *et al*, 2005) Without the input from *in vitro* studies, research in orthodontic bonding will not progress and orthodontics will not be able to utilize and take advantage of possibly more effective and efficient bonding agents entering the market.

2 Literature Review

2.1 Bond Testing in Orthodontics

Adequate orthodontic bond strength of a bracket to tooth structure is an important aspect of clinical orthodontics – without it practising orthodontics can become frustrating for both the practitioner and the patient. Loose brackets will cause increased treatment time, added costs of materials, and additional visits by the patient. Bond strengths of orthodontic attachments must be able to withstand the masticatory forces of the patient and any orthodontic forces placed by the orthodontist. However, the orthodontic bond strength cannot be too strong since easy removal of the bracket is needed at the end of treatment with minimal damage to the tooth. (Proffit *et al*, 2007)

Preliminary testing of a new bonding system is usually performed *in vitro* using extracted teeth, normally human or bovine. *In vitro* testing allows for a more controlled environment where sensitive equipment can be used to precisely measure the bond strength of an attachment to a substrate. The measured bond strengths are reported as megapascals (MPa), kilogram per square centimeter (kg/cm^2) or pounds per square inch (lb/in^2). Bond strength can also be reported as *bond force* in units of Newtons (N), kilograms (kg) or pounds (lbs). Bond strength is the bond force divided by the area of the bonded interface (e.g. $1\ Pa = 1\ N/m^2$, $1\ MPa = 1\ N/mm^2$). Thus, experimental studies using a universal testing machine (e.g. Zwick GmBH) can measure the force (N) needed to debond a bracket with a known bracket base area (mm^2) to give a bond strength value in N/mm^2 or MPa. (Powers *et al*, 1997)

In the laboratory, there are several methods to cause a debond in order to study bond strengths. These include: *shear bond strength, tensile strength* and *torsional strength* tests. Both shear and tensile testing are valid ways to study orthodontic bond strengths, whereas torsional testing is difficult to perform and not so common. Testing shear bond strength involves loading the bracket with a blade so that the bracket slides parallel off the tooth surface. However, a pure shear load is difficult to achieve since debonding involves components of peeling, tension and torsion. Also, once a debonding force is applied at a distance from and perpendicular to the adhesive-bracket junction it is actually measuring the *shear-peel bond strength*. Most studies reporting shear bond strengths are in fact testing shear-peel bond strength due to the difficulties of obtaining a pure shear load at the bracket base. (Katona, 1994; Powers *et al*, 1997)

Also, in bonding studies it is important to observe where the bond failure has occurred. There are two types of bond failures that can occur. First, *cohesive failures* which occur within the material itself may happen within the tooth, bracket (e.g. a ceramic bracket) or most commonly within the adhesive resin. The second type of failure is *adhesive failures* which can occur between the tooth/adhesive or bracket/adhesive interface. In order to determine the frequency of where bond failures are occurring within a study – a scale such as the Adhesive Remnant Index (ARI) can be used. (Powers *et al*, 1997) The original ARI was described by Årtun and Bergland (1984), however, since then it has been updated and modified in bonding studies. (Årtun & Bergland, 1984; Bishara *et al*, 1999b) Determining where most of the bond failures occur using the ARI would help identify whether the bond strength is strong enough to bond composite resin to the enamel for placement of orthodontic brackets. During debonding, it is desirable for the site of failure to occur between the adhesive resin and the bracket base which avoids damage to the enamel. Failure at the enamel/adhesive interface is undesirable since it can fracture or tear the enamel as the bracket is pulled away from the tooth surface. (Proffit *et al*, 2007)

2.1.1 Minimum recommended bond strength

The clinical bond strength of a bracket is important since it needs to be strong enough to resist the normal masticatory functions of the patient as well as the applied orthodontic forces placed by the orthodontist. At the same time, the bond strength needs to be weak enough to allow easy debonding at the end of treatment with no/minimal enamel damage. Recommendations of the minimum bond strengths provide clinical meaning to the bond strengths measured during *in vitro* studies. The minimum value of 6-8 MPa was first recommended by Reynolds in 1975. This recommendation was deduced if a typical bracket has a bonding area of $16mm^2$ and the average force transmitted to a bracket during function is between 40 – 120 N (1 MPa = 1 N/mm^2). Thus, the minimum bond strength needed to withstand the applied force of 120 N is 7.5 MPa. (Reynolds, 1975; Powers *et al*, 1997) However, this conclusion was made over 30 years ago and since then there have been advances in materials, computer technology and testing systems. According to Wiltshire & Noble (2010), an "ideal bond strength" is difficult to define because each individual differs in their masticatory forces, eating habits, and intra-oral environment. They recommended that to achieve the minimal reliable clinical bond strength, *in vitro* bond strengths should be at least 3-4 MPa. It is also important to note that during *in vitro* testing it is key to not only look at the means but to also examine the range of values; in particular the low end of the range. (Wiltshire & Noble, 2010) This recommendation was based on clinical trials using glass ionomers to bond orthodontic brackets. It was demonstrated that there was no significant differences in failures rates between the glass ionomer (3.3%) and conventional orthodontic resin (1.6%). (Fricker, 1994)

2.2 Bond Strength Testing Standardization

With the increase in new bonding agents into the market, there has been an increase in *in vitro* studies measuring bond strengths of these products and their potential use in orthodontics. However, there has been a lack of consensus of experimental protocols standardizing these experiments which has led to variability in bond strengths of similar products. In order to evaluate bond strengths, laboratory studies have used a variety of different methods and conditions during testing. The major classification of bonding studies can be divided into three categories:

1. Test environment: *in vivo, in vitro, ex vivo*
2. Substrate: *enamel, composite resin, porcelain, amalgam*
3. Loading mode: *shear, tensile, torsion, shear-peel*

Choosing these different variables depends mainly on the purpose of the study, however, most studies on new bonding systems are performed *in vitro* using an enamel substrate and measuring the shear bond strength due to the relatively simplicity of the test and its close simulation to the clinical environment. (Eliades & Brantley, 2000)

However, there are several other protocol parameters and variables that can vary at different stages of a study and because of this lack of standardization the measured bond strengths can vary. Such variability in testing protocols can include:

- Tooth selection type
- Storage time
- Storage medium

- Preparation of the tooth surface
- Thermocycling

Another stage of a bonding study that can introduce variability is during the actual bonding of the bracket to the substrate. Two major variables that are not currently standardized are the amount of composite resin placed on the bracket base except for the Adhesive Pre-coated (APC) systems by 3M Unitek and now TP Labs Inc. and the amount of force placed on the bracket during seating. Finally, the actual testing of the bond strengths involve choosing the rate of loading or cross-head speed, the technique used to apply the shear stress (e.g. wire loops, steel blades or rods), and bracket base design. All these factors may influence the outcome of the research trials. As with any *in vitro* study, it is impossible to precisely duplicate the oral environment in a controlled setting. Such factors that are difficult to replicate *in vitro* include pH, microflora, temperature variation, as well as stresses from activated archwires and occlusal forces. (Powers *et al*, 1997; Stanford *et al*, 1997; Eliades & Brantley, 2000)

Thus, it is apparent that due to the variability in research protocols for studying bond strengths, a standardized test protocol is needed to allow for proper interpretation and comparison of test results by different researchers. Nevertheless, bonding studies provide an insight into the possible clinical performance of new bonding agents and with these studies the advancement of orthodontic bonding research will continue to move forward.

2.3 Conventional Bonding System

2.3.1 Composition

The first step in conventional bonding is to treat the enamel with an acid etchant, usually a phosphoric acid gel that comes in varying concentrations – most commonly 37% or 35%. The second step is the application of the *single bottle bonding agent* that consists of two main components: the primer and the adhesive. Primers are basically hydrophilic monomers dissolved in a solvent while adhesives are hydrophobic, dimethacrylate oligomers (i.e. Bis-GMA) usually diluted with a lower-molecular weight monomer such as triethylene glycol dimethacrylate (TEGDMA). The adhesive is compatible with both the monomers of the primer and composite resin. Other components within the bottle are the solvent used to carry the primer monomer which can be acetone, water, ethanol-water, or solvent-free, as well as initiators/accelerators such as camphoroquinone which initiates the polymerization reaction. Bonding agents are usually unfilled allowing for easier flow into the dissolved enamel allowing for increased micro-mechanical retention. However, in recent products some bonding agents include filler particles called *nano-fillers* which may actually increase *in vitro* bond strengths. (Craig & Powers, 2002; Powers *et al*, 2006; Van Noort, 2007)

2.3.2 Enamel Bonding

The conventional bonding system (5th generation bonding agents) consists of two steps in order to bond an orthodontic bracket to a tooth. The first step involves placing an acidic enamel conditioner or acid etch such as 37% phosphoric acid on the enamel surface to remove the smear layer. The enamel conditioner also differentially dissolves the enamel's hydroxyapatite crystals thus roughening the surface which improves its wettability in preparation for the application of the primer-adhesive solution. After placement for approximately 15 seconds, the enamel conditioner is thoroughly washed away and the enamel surface is dried to produce a frosty appearance indicating the properly dissolved enamel. The second step involves placement of a thin layer of the primer-adhesive *single bottle bonding agent* which is able to freely flow into the enamel irregularities thus becoming micro-mechanically locked once polymerized. This micro-mechanical retention comes from both micro- and macro-tags of the polymerized adhesive. (Powers *et al*, 2006)

During the bonding procedure, the bonding agent can experience high polymerization shrinkage due to their higher percentage of co-monomer. It has also been found that they experience an increased level of water sorption and an increased thickness of the oxygen-inhibition layer due to their large surface area to volume ratio and the clinical procedure of their placement on enamel. It has been thought that air-thinning of the bonding agent allows for reducing the thickness of the bonding agent, however, it increases the bulk oxygen concentration which can introduce defects that can lead to increased water sorption and monomer leaching. Air-thinning can also cause unevenness in the bonding layer which may lead to stress distribution problems. This layer of oxygen inhibition can cause harmful effects on the physical properties between the bonding agent and the composite resin. (Brantley & Eliades, 2001)

In order to reduce these detrimental effects two approaches can be used to reduce the amount of oxygen diffusion into the polymerizing composite. First, shortening the time the bonding agent is exposed to air prior to complete curing can reduce oxygen inhibition. This can be done with a more powerful curing light, use of a larger concentration of the photoinitiator, or use of a synergist or co-initiator to accelerate the surface cure. The second approach is to block the bonding agent from oxygen. This can be done with either curing under an inert gas or placement of petroleum jelly or a glycerol coating to protect the composite resin from oxygen. However, these methods can be impractical and difficult to perform clinically. (Brantley & Eliades, 2001)

2.4 Self-Etching Primers

To reduce chair time and efficiency during the bonding procedure, manufacturers have developed self-etching primers to be used in restorative dentistry. (Trites *et al*, 2004) This one-step system of etching and priming is often referred to as the

6^{th} generation bonding agents which still requires mixing of the primer and adhesive components with no separate etching required (e.g. Transbond Plus Self-Etching Primer). Recently, 7^{th} generation self-etching primers have been released which is a single component bonding system requiring no mixing and etching. (Farah & Powers, 2004)

The use of self-etching primers in orthodontics has been found to be effective when bonding brackets to tooth enamel. (Bishara *et al*, 2001; Arnold *et al*, 2002) There are several advantages of using self-etching primers during orthodontic bonding. These include allowing the primer and etchant to simultaneous penetrate into the enamel, reducing technical errors, eliminating cross-contamination and shortening clinical chair time due to the elimination of several steps such as enamel etching. (Fritz *et al*, 2001) Furthermore, self-etching primers has shown to have a shallower etch pattern indicating less enamel loss to the tooth and also less bond strength. This may be the reason for less enamel fractures during debonding with self-etching primers as compared to conventional bonding. (Paschos *et al*, 2008)

Self-etching primers are composed of an aqueous mixture of polymerizable acidic monomers consisting of methacrylated phosphoric acid ester which contains both the acidic component for etching and a monomer component as the primer. (Bishara *et al*, 2001) Their low pH facilitates enamel etching and because the solution also contains the primer, the etched enamel is simultaneously primed. The acidic monomers are able to bind to the enamel calcium and when polymerized they form the resin tags needed for micro-mechanical retention. Neutralization occurs rapidly as the solution binds to the calcium ions from hydroxyapatite crystals, which stops the demineralization process. (Velo *et al*, 2002; Holzmeier *et al*, 2008; Minick *et al*, 2009) Therefore, due to the neutralization of the acid, there is no need to rinse the enamel surface prior to bracket bonding. This rapid neutralization may cause a shallower etching pattern and a reduction in enamel demineralization leading to shorter resin tags. (Cal-Neto & Miguel, 2006; Holzmeier *et al*, 2008; Paschos *et al*, 2008)

Recent advances in bonding technology have led to the use of nanotechnology in dentistry. Some new self-etching primers now consist of nano-reinforced filler particles where the manufacturers claim the nanofillers are small enough to penetrate into the enamel increasing the bond strength by acting as reinforced cross-links at the hybrid layer. A recent study by Başaran *et al* (2009) looked at one such nano-reinforced self-etching primer called Futurabond NR (Voco). The study compared the bond strengths of three self-etching primers against conventional etching. The three self-etching primers were: Adper Prompt L-pop (3M ESPE), Transbond Plus SEP (3M Unitek) and Futurabond (Voco). Comparing shear bond strengths at 12 and 24 hours they found no statistical difference among the three self-etching primers studied ($p>0.05$). Thus, the nano-reinforced Futurabond NR did not achieve a higher bond strength as expected. Further studies are needed in this new area of bonding technology and its relevance in orthodontics.

2.4.1 Transbond Plus Self Etching Primer

Transbond Plus Self Etching Primer (3M Unitek, Monrovia, CA) is a 6th generation light-cured bonding agent that requires mixing prior to application, however, no separate etching, rinsing and drying steps are required. Transbond Plus Self Etching Primer's main indication is to be used to bond orthodontic brackets with light-cured direct bonding composite resins.

Transbond Plus Self Etching Primer uses the lollipop system consisting of two separated compartments: one contains 2-HEMA, polyalkenoic acid, water and stabilizers and the other methyacrylated phosphoric acid esters, Bis-GMA, photo-initiator and stabilizers. (Trites *et al*, 2004; Holzmeier *et al*, 2008) With a pH of approximately 1.0, the methyacrylated phosphoric acid esters demineralize the enamel and dentin similar to traditional 30%-50% phosphoric acid. As the two solutions are mixed together, they are applied to the enamel where the acidic monomer becomes neutralized. (Arnold *et al*, 2002; Bishara *et al*, 2002)

Currently, Transbond Plus Self Etching Primers is widely accepted in its use in bonding orthodontic brackets and is now one of the most popular self-etching primers on the market today. The suitability of Transbond Plus Self Etching Primers for bonding orthodontic brackets was first described in the early 2000s. (Bishara *et al*, 1999a; Brosnihan & Safranek, 2000; Miller, 2001; White, 2001; Arnold *et al*, 2002; Buyukyilmaz *et al*, 2003) These early *in vitro* studies comparing Transbond Plus Self Etching Primers to conventional bonding has shown them to have lower shear bond strength values, however, they were still deemed acceptable for clinical use of bonding brackets to teeth.

Clinical studies published have found conflicting results on the failure rates of Transbond Plus Self Etching Primers compared to the conventional bonding system (see Table 2-1). The majority of these recent publications indicated that Transbond Plus Self Etching Primer has no significant difference in clinical failure rates as compared to conventional bonding systems and thus can be used for orthodontic purposes. (Aljubouri *et al*, 2004; Manning *et al*, 2006; Pandis *et al*, 2006; Banks & Thiruvenkatachari, 2007; Khalha, 2008; Reis *et al*, 2008; Cal-Neto *et al*, 2009) The most recent study by Cal-Neto *et al* (2009) was a randomized clinical controlled trial evaluating the performance of Transbond Plus Self Etching Primer against conventional Transbond XT bonding system over a 12 month period. Twenty-eight patients were randomly selected to be bonded with either Transbond Plus Self Etching Primer or the conventional Transbond XT bonding system. After a 12 month period, the failure rates of the Transbond XT bonding system and Transbond Plus Self Etching Primer groups were 4.78% and 6.88%, respectively. There was no significant difference in the survival rates that were observed between the two bonding procedures (*P* = 0.311). In conclusion, both systems had low bond failure rates and both were adequate for orthodontic bonding.

Only two studies found Transbond Plus Self Etching Primer to have a lower failure rate (Asgari *et al*, 2002; dos Santos *et al*, 2006) and three studies found it to have a higher failure rate. (Ireland *et al*, 2003; Murfitt *et al*, 2006; Elekdag-Turk *et al*, 2008a)

Table 2-1: Clinical Trials - Transbond Plus SEP vs. Conventional Bonding system

Author	Year	Publication	Failure Rate	
			Transbond Plus SEP	**Conventional Bonding**
No difference in clinical failure rates				
Cal-Neto	2009	American Journal of Dentofacial Orthopedics & Orthodontics	6.88%	4.78%
Khalha	2008	Evidence Based Dentistry	4.8%	3.5%
Reis	2008	European Journal of Orthodontics	15.6%	17.6%
Banks	2007	Journal of Orthodontics	4.8%	3.5%
Manning	2006	Journal of Orthodontics	7.0%	7.4%
Aljubouri	2004	European Journal of Orthodontics	1.6%	3.1%
Transbond Plus SEP: lower failure rate				
Dos Santos	2006	Angle Orthodontics	7.4%	10.6%
Asgari	2002	Journal of Clinical Orthodontics	0.57%	4.60%
Transbond Plus SEP: higher failure rate				
Elekdag-Turk	2008	Angle Orthodontics	4.7%	1.7%
Murfitt	2006	European Journal of Orthodontics	11.2%	3.9%
Ireland	2003	American Journal of Dentofacial Orthopedics & Orthodontics	10.99%	4.95%

2.4.2 iBOND Self Etch Primer

Heraeus Kulzer (Hanau, Germany) manufactures iBOND Self Etch Primer a 7th generation, an all-in-one bonding agent that etches, primes, bonds and desensitizes with a single application. In contrast to Transbond Plus Self Etching Primer, iBOND does not require any mixing and needs only a single application. Currently, iBOND is only indicated for bonding direct composite restorations, bonding of indirect restorations in combination with a light-curing luting cement, and for sealing of hypersensitive teeth.

iBOND is composed of an acetone/water base with light-activated resin urethane dimethacrylate (UDMA), 4-methacryloyloxyethyl trimellitate anhydride (4-META), stabilizers, a photo-initiator, and glutaraldehyde with <1% filler. (iBOND Self Etch: Scientific Information, 2007; Holzmeier *et al*, 2008) The 4-META is the acidic monomer responsible for etching and polymerizing into the retentive resin tags. (Minick *et al*, 2009)

In the orthodontic literature, studies evaluating the bond strength of iBOND on orthodontic brackets are limited. The results from these studies vary, but it must be kept in mind that each study used different methodologies including incubation times, the use of thermocycling, and cross-head speeds. Holzmeier *et al* (2008) used bovine teeth to look at several self-etching primers used primarily in restorative dentistry including iBOND. These new self-etching primers were compared to Transbond Plus Self Etching Primer and conventional Transbond XT bonding system. They found iBOND to have a mean shear bond strength of 8.1 MPa which was considerably lower than Transbond Plus Self Etching Primer (20.7 MPa) and Transbond XT bonding system (21.0 MPa). (Holzmeier *et al*, 2008) Paschos *et al* (2008) using human teeth with thermocycling found iBOND to have a shear bond strength 11.3 MPa. (Paschos *et al*, 2008) Both studies concluded that iBOND may be clinically acceptable to use for orthodontic bonding of brackets to teeth due to achieving the minimal bond strength.

On the other hand, Minick *et al* (2009) using bovine teeth looked at several new bonding systems and primers including iBOND. The teeth were bonded with a stainless steel bracket and were incubated for 30 minutes and 24 hours at 37°C. The bond strengths for iBOND (30 minutes: 3.91 MPa; 24 hours: 3.86 MPa) were found to be significantly lower than Transbond XT bonding system (30 minutes: 10.05 MPa; 24 hours: 10.11 MPa) at both the 30 minutes and 24 hours time points. At both incubation times, the shear bond strength of iBOND were below the minimal amount of 6-8 MPa as recommended by Reynolds (1975). Thus, based on Reynolds minimum bond strength recommendations, the authors concluded that iBOND may not have adequate strength for clinical orthodontic use. (Reynolds, 1975; Minick *et al*, 2009) However, Wiltshire & Noble (2010) recently suggested that the shear bond strength may be as low as 3-4 MPa and still can be clinically successful.

2.4.3 G-Bond: One-component self-etching light-cured adhesive

G-Bond (GC Corporation, Tokyo, Japan) is an all-in-one 7th generation bonding system. G-Bond is indicated for bonding light-cured composites, compomers, dual-cured cements and core build-up materials to tooth structure. (G-Bond: Advanced 7th Generation Single Component Adhesive, 2006) A review of the literature has found that there are currently no published studies on the use of G-Bond to bond orthodontic brackets to enamel. The publications found on G-Bond were from a restorative perspective looking at the shear bond strength of composite resin to enamel and dentin. The restorative study by Burrow *et al* (2008) showed that the shear bond strength of composite resin to human enamel using G-Bond (27.1 MPa) was not statistically significantly different when compared to the control two-step self-etching priming system Clearfil SE Bond (30.2 MPa). (Burrow *et al*, 2008) Another restorative study by Söderholm *et al* (2008) compared several new single-bottle self-etching primers to a conventional two-bottle etch and rinse bonding system. After the bonding procedure, each tooth was store in 37°C for 24 hours prior to debonding. G-Bond was found to have a mean shear bond strength of 14.9 MPa. (Söderholm *et al*, 2008) Both these restorative studies provide a preliminary indication that the bond strength of G-Bond may be sufficient or even too high for orthodontic use. Accordingly, future orthodontic studies will be needed to provide clearer evidence of G-Bond's ability to be used in clinical orthodontics.

3 Purpose

The purpose of this study was to evaluate and compare the shear bond strength of two new 7th generation bonding systems used in restorative dentistry: iBOND Self Etch and G-Bond: One-component self-etching light-cured adhesive.

4 Null Hypotheses

1. There is no difference in shear bond strength to enamel between brackets bonded with Transbond XT primer (control), Transbond Plus Self Etching Primer, iBOND Self Etch and G-Bond at each time interval of immediate (<5 minutes), 24 hours and 3 months.
2. There is no difference in shear bond strengths of Transbond XT primer (control), Transbond Plus Self Etching Primer, iBOND Self Etch and G-Bond over the three time points.
3. There is no difference in the scores of the Adhesive Remnant Index between Transbond XT primer (control), Transbond Plus Self Etching Primer, iBOND Self Etch and G-Bond.

5 Materials & Methods

5.1 Materials used in the study

In this study, the shear bond strength of four different bonding systems were tested when debonding lingual buttons from human tooth enamel *in vitro*. The following are descriptions of the materials used in this study.

5.1.1 Transbond XT Light Cure Adhesive System

The Transbond XT Light Cure Adhesive system (3M Unitek, Monrovia, CA) (Figure 5-1) is used in orthodontics to bond metal or ceramic brackets to teeth. The entire system consists of several components. They are as follows:

> **Transbond XT adhesive paste** is a light-cured, composite resin consisting of 10-20%wt Bisphenol A diglycidylether methacrylate (Bis-GMA), 5-10%wt Bisphenol A bis (2-hydroxyethyl ether) dimethacrylate (Bis-EMA), 70-80%wt silane treated quartz and <2%wt silane treated silica. (Material Safety Data Sheet: Transbond XT Light Cure Adhesive, 2008) The quartz and silica are filler particles that have been treated with silane which is a coupling-agent. Bis-EMA is used to alter viscosity and handling characteristics of the resin. (Mitchell, 2008)

Transbond XT primer is an unfilled light-cured resin consisting of Bis-GMA and Triethylene glycol dimethacrylate (TEGDMA) in a 1:1 ratio. A photoinitiator is also present. (Material Safety Data Sheet: Transbond XT Primer, 2005; Holzmeier *et al*, 2008)

Etching gel (Figure 5-2) is 35% phosphoric acid in water and amorphous silica. (Material Safety Data Sheet: Transbond XT Etching Gel System, 2008)

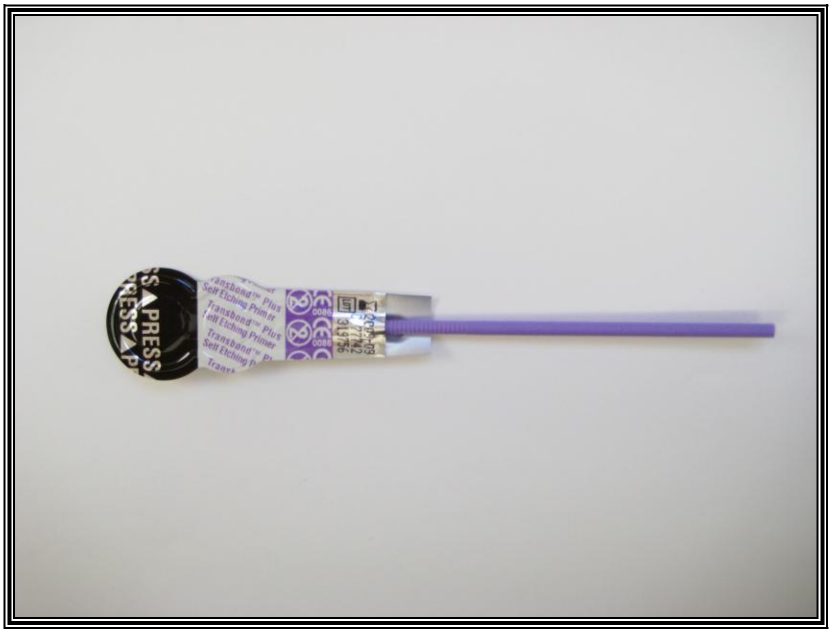
Figure 5-1: Transbond XT Light Cure Adhesive System

Figure 5-2: Transbond XT etching gel 35% phosphoric acid

5.1.2 Transbond Plus Self Etching Primer

Transbond Plus Self Etching Primer (SEP) (3M Unitek, Monrovia, CA) (Figure 5-3) is a 6th generation light-curing bonding agent that requires mixing prior to application. The main indication for the use of Transbond Plus SEP is to bond orthodontic brackets with light-cured direct bonding composite resins. One compartment composes of a methylacrylated ester derivative, Bis-GMA, a photo-initiator and stabilizers. The second compartment contains 2-Hydroxyethyl Methacrylate (HEMA), polyalkenoic acid, stabilizers in water. (Material Safety Data Sheet: Transbond Plus Self Etching Primer, 2005; Holzmeier *et al*, 2008)

Figure 5-3: Transbond Plus Self Etching Primer



5.1.3 iBOND Self Etch

iBOND Self Etch is a 7th generation light-curing self-etching 1-component bonding agent which requires no mixing (Figure 5-4). The main use is to bond composite resin restorations to enamel and dentine. iBOND Self Etch is able to prime, bond and desensitize in a single step. iBOND Self Etch is composed of an acetone/water base with light-activated resin urethane dimethacrylate resin (UDMA), 4-methacryloxyethyl trimellitate anhydride (META), stabilizers, photo-initiator, glutaraldehyde with <1% filler. (iBOND Self Etch: Scientific Information, 2007; Holzmeier *et al*, 2008)

Figure 5-4: iBOND Self Etch

5.1.4 G-Bond: One-component self-etching light-cured adhesive

G-Bond is a 7th generation light-curing self-etching system that is able to etch, bond and desensitize in a single step (Figure 5-5). G-Bond is a formulation of phosphoric ester monomer, 4-methacryloxyethyltrimellitic acid (MET) monomer, 5% nano-filled particles in an acetone/water base solvent. (G-Bond: Advanced 7th Generation Single Component Adhesive, 2006)

Figure 5-5: G-Bond: One- component self-etching light-cured adhesive

5.1.5 Bondable Stainless Steel Buttons

The bondable stainless steel buttons used in this study were (Figure 5-6) obtained from GAC International. Their mildly curved mesh base allowed for optimal seating on the molars. The average surface areas of the buttons were determined by measuring the diameter of 20 random buttons using a digital caliper and then calculating the surface area using the formula: Area = πr^2. The mean area measurement ($3.33mm^2$) was then used to determine the stress in megapascals (MPa) during the shear bond strength tests of the buttons.

Figure 5-6: Lingual Button Curved

A list of bonding systems used in this study including their manufacturer, reference number and lot number can be found in Table 5-1.

Table 5-1: Bonding systems used in this experiment

Group	Bonding System	Manufacturer	Reference Number	Lot Number
1	Transbond XT Primer	3M Unitek, Monrovia, California	712-034	8EX
2	Transbond Plus Self Etching Primer	3M Unitek, Monrovia, California	712-091	010033
3	iBOND Self Etch	Heraeus Kulzer, Hanau, Germany	66033607	0801111
4	G-Bond: One-component self-etching light-cured adhesive	GC Corporation, Tokyo, Japan	002277	

A comprehensive list of all materials and equipment used in this study with their corresponding reference number and/or lot number can be found in Table 5-2.

Table 5-2: Materials used in this experiment

Material	Manufacturer	Ref #	Lot #
Bonding Kit			
Transbond XT Light Cure Adhesive Kit in capsules	3M Unitek, Monrovia, California	712-030	BL/8EX
Transbond XT Adhesive paste		712-031	
Transbond XT 35% Etching Gel		712-039 9802	
Tooth Preparation Materials			
Diamond saw	Buehler, Lake Bluff, Ill		
Pumice Preppies	Whip Mix, Louisville, KY		
Cold cure acrylic - Monomer liquid	SR-Ivolen		C13504
Cold cure acrylic - Polymer powder			C14783
Incubator at 37°C	Thelco/Canlab Model 2, Precision Scientific, Chicago, IL		
Bonding Materials			
Curved stainless steel lingual buttons	GAC International, Central Islip, NY	30-000-01	A597
Loading apparatus gauge	Federal: Miracle Movement 0.001″ C81S, Providence, RI		
Light curing unit - Mini LED Blue Ray	American Orthodontist		
Debonding Materials			
Universal Testing Machine	Zwick GmBH, Ulm, Germany		
Bencor Multi-T testing apparatus	Danville Engineering, San Ramon, CA		
ARI Materials			
Light Microscope	Nikon SMZ800		
Coolpix Nikon digital camera	Nikon E990 3.34megapixels		
Chemicals			
Chloramine-T trihydrate 98%	Acros Organics, New Jersey		A0236347
Other			
Digital Caliper	Mastercraft		

5.2 Experimental Method

180 teeth were bonded with a stainless steel button using one of the four different bonding systems i.e. 15 teeth per group. The shear bond strength was then determined after three different time points:

T1: Immediate (<5 minutes)

T2: 24 hours

T3: 3 months

Table 5-3 provides a general summary of the experimental outline showing how the four test groups were sub-divided into three sub-groups to test each of the different bonding systems at the three time points.

Table 5-3: Summary of experiment

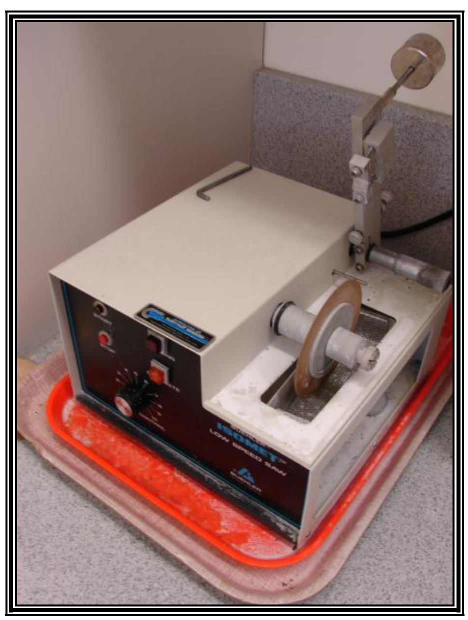
Group	Bonding Systems	Sub-groups	Time prior to debond	Number of molars
1	Transbond XT Primer (control)	1	Immediate (<5min)	15
		2	24 hours	15
		3	3 months	15
2	Transbond Plus Self Etching Primer	4	Immediate (<5min)	15
		5	24 hours	15
		6	3 months	15
3	iBOND Self Etch	7	Immediate (<5min)	15
		8	24 hours	15
		9	3 months	15
4	G-Bond	10	Immediate (<5min)	15
		11	24 hours	15
		12	3 months	15
			TOTAL	180

5.2.1 Tooth Preparation and Storage

Prior to the study, ethics approval was obtained through the Bannatyne campus research ethics board (University of Manitoba). Over a period several months, 180 extracted molars were collected from Oral surgery clinics throughout Winnipeg, Manitoba and were stored in distilled water with 0.5% Chloramine T (1g Chloramine T:200ml water). The teeth were thoroughly screened to exclude ones with visible caries, restorations, defects and/or anomalies.

The teeth were washed in distilled water and their roots were separated with a water-cooled diamond saw (Figure 5-7). The crowns were then stored in fresh distilled water with 0.5% Chloramine T and kept in the refrigerator at 4ºC until needed.

Figure 5-7: Diamond Saw



The crowns were embedded into cold-cure acrylic (SR-Ivolen) within stainless steel mounting rings. As the acrylic hardened, the facial surface of the crown at the height of contour was made parallel to the horizontal plane with a 90° T-bar instrument. This was to ensure that the line of force of the shearing blade would be consistent between samples. Each tooth embedded in acrylic was then removed from the steel rings and was allowed to fully cure for 24 hours in 100% humidity. This was done by placing all the samples on a tray and laying a damp paper towel covering the teeth. The tray was then tightly sealed with plastic wrap preventing moisture from escaping, thus preventing the teeth from desiccating. After 24 hours each tooth embedded in acrylic was ready for bonding of a button.

5.2.2 Bonding Procedure

A total of 180 molars were randomly divided into the four bonding system groups of 45 molars and then further separated into 12 sub-groups of 15 molars (Table 5-3). The teeth were then polished with a non-fluoridated pumice (Whip Mix, Louisville, KY) and water slurry for 10 seconds, washed and dried prior to bonding. (Nemeth *et al*, 2006) Each group consisting of 45 teeth were then bonded with only one of the following primers according to the manufacturer's instructions: Transbond XT primer, Transbond Plus SEP, iBOND and G-Bond.

Group 1: Transbond XT Primer + 35% etching gel (control)

Each tooth was etched with 35% phosphoric acid etching gel for 15 seconds. The etching gel was then rinsed with water spray for 15 seconds and air dried. A frosty white enamel appearance was observed to indicate adequate etching of the enamel. With an applicator a single thin layer of Transbond XT primer was applied to the etched enamel surface.

Group 2: Transbond Plus Self Etching Primer

To prepare the Transbond Plus SEP for application the contents within the black reservoir at then end was completely squeezed into the middle reservoir towards the applicator. The black reservoir was then folded over the top of the middle reservoir and the contents of the middle reservoir completely squeezed into the purple reservoir closest to the applicator stick. The applicator was then swirled around within the purple reservoir for 5 seconds to evenly apply the mixture onto the applicator tip. The applicator was then rubbed with light pressure on the enamel of each tooth for 5 seconds. Finally, a gentle air burst was applied over the primer for 1-2 seconds.

Group 3: iBOND Self Etch

Prior to dispensing the iBOND the bottle was vigorously shaken. A drop of iBOND was then dispensed into the mixing well. An applicator brush was then dipped into the iBOND solution and then rubbed on the enamel for 20 seconds per tooth. Gentle air drying was performed until a glossy enamel surface was observed. The tooth was then light cured for 20 seconds using a LED light curing unit (American Orthodontics).

Group 3: G-bond: One-component self-etching light-cured adhesive

A drop of G-bond was dispensed into the mixing well and with an applicator a single coat of G-Bond was applied to the tooth surface. After waiting for 5 seconds, a gentle air burst was applied to thin the primer. The tooth was then light cured for 10 seconds using a LED light curing unit (American Orthodontics).

After each tooth was primed with one of the four bonding systems, they were bonded with a single stainless steel button using Transbond XT adhesive composite. A uniform amount of Transbond XT adhesive composite was applied to the button base and then gently placed on the enamel surface. To ensure uniform and complete seating of the button to the tooth a 500g vertical loading apparatus was used. Any excess composite surrounding the button was carefully removed using a periodontal probe. Each tooth was then light cured for 20 seconds each using a LED light-curing unit (American Orthodontics) placed as close as possible to the button without contacting it in a direction directly above and perpendicular the button and enamel surface.

The approximate total amount of time to prepare each tooth for bonding the different bonding systems was calculated and can be found in Table 5-4.

Table 5-4: Summary of bonding systems protocols

Bond	Etch	Rub	Air dry	Light cure SEP	Light cure adhesive resin	Total Time
Transbond XT primer + etch	15s (rinse 15s)	-	5s	-	20s	40s
Transbond SEP	-	5s	1-2s	-	20s	27s
iBOND	-	20s	1-2s	20s	20s	62s
G-Bond	-	Sit 5s	5s (max)	10s	20s	40s

5.2.3 Storage Conditions

For the evaluation of immediate bond strength, brackets were debonded <5 minutes after bonding of the button to the tooth. For the 24 hours and 3 months groups, the teeth embedded in acrylic with their bonded button were stored in de-ionized water in a 37°C incubator in a covered glass jar. The teeth were regularly checked to make sure they were constantly submerged in water.

5.2.4 Shear bond strength testing

The immediate (<5 minutes), 24 hours and 3 months test groups were tested for their shear bond strength using a universal testing machine (Zwick, Germany) (Figure 5-8). Each sample were mounted horizontally and secured into the Bencor Multi-T loading apparatus by tightening the screws with hex key (Figure 5-9). The Bencor Multi-T loading apparatus with the mounted sample was then placed on the universal testing machine platform. The shearing blade was directed in an occluso-gingival direction at an application point as close as possible to the tooth-adhesive resin-button interface using a crosshead speed of 0.5mm/minute on a 1kN load cell. The buttons were loaded until they debonded and the stress in megapascals was recorded onto a computer connected to the universal testing machine.

Figure 5-8: Universal testing machine and computer

Figure 5-9: Bencor Multi-T loading apparatus

5.2.5 Evaluation of Fracture sites

Once the brackets were debonded each tooth was evaluated under a light microscope (10x magnification) in order to determine the amount of adhesive left on the tooth surface. Each tooth was evaluated using a modified Adhesive Remnant Index (ARI) score as described by Bishara *et al* (1999). With the same operator, 33% of the samples were randomly selected from each group and the ARI scores were re-evaluated (evaluator was blinded to the samples) 6 months later. (Årtun & Bergland, 1984; Bishara *et al*, 1999b)

Adhesive Remnant Index (ARI) score (1-5):

Score 1 = 100% left on tooth + bracket impression

Score 2 = >90% left on tooth

Score 3 = 10-90% left on tooth

Score 4 = <10% left on tooth

Score 5 = 0% left on tooth

5.3 Statistical Analysis

Descriptive analysis including mean, standard deviation, minimum, maximum, and range for each of the tested groups were recorded. An analysis of variance (ANOVA) was performed to determine whether there was a significant difference among the test groups over time as well as within each test group at a specific time point. If significant differences were present, a Bonferroni post-hoc multiple comparisons test was performed to identify which means were significantly different from each other. The Chi-squared test was used to determine the significant differences in the ARI scores among the different groups. The significance used for all the tests was predetermined at a probability value of 0.05 or less.

6 Results

6.1 Statistics of Shear Bond Strength at Specific times over 3 months

The descriptive statistics for each of the bonding agents tested: Transbond XT primer (control), Transbond Plus SEP, iBOND and G-Bond are presented at the immediate, 24 hours and 3 months time points. The coefficient of variation was also calculated and presented with the data. In orthodontic bonding studies, the goal should be to achieve a coefficient of variation (standard deviation/mean) in the range of 20% - 30%. (Powers *et al*, 1997) An ANOVA test was performed to determine whether there was a significant difference between the tested groups. If a significant difference was present, a Bonferroni post-hoc multiple comparisons test was used to identify exactly which mean shear bond strengths were significantly different from one another. All significant differences were pre-determined at a probability value of 0.05 or less. Recommendations of the minimum bond strengths provide clinical meaning to the bond strengths measured during *in vitro* studies. Reynolds (1975) first recommended the minimum value of 6-8 MPa. However, since then, there have been advances in materials, computer technology and testing systems. Recently, Wiltshire & Noble (2010) recommended that the minimal reliable clinical bond strength should be at least 3-4 MPa as measure during *in vitro* studies. (Reynolds, 1975; Wiltshire & Noble, 2010)

6.1.1 Immediate Shear Bond Strength

Table 6-1: Descriptive statistics of the Immediate shear bond strengths (MPa)

Groups	Sample size (N)	Mean (MPa)	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Transbond XT primer	15	11.22	1.98	7.91	14.92	7.01	17.73%
Transbond Plus SEP	15	5.32	1.81	2.78	8.83	6.05	34.00%
iBOND	15	6.69	1.78	2.84	9.64	6.80	26.59%
G-Bond	15	8.30	2.42	5.17	13.35	8.18	29.14%

Table 6-2: Bonferonni Post-hoc Multiple Comparisons Test of the Immediate shear bond strengths (MPa)

Groups	Groups in comparison	Mean Difference	Statistical Significance
Transbond XT primer	Transbond Plus SEP	5.90*	0.000
	iBOND	4.53*	0.000
	G-Bond	2.92*	0.001
Transbond Plus SEP	Transbond XT primer	-5.90*	0.000
	iBOND	-1.37	0.407
	G-Bond	-2.98*	0.001
iBOND	Transbond XT primer	-4.53*	0.000
	Transbond Plus SEP	1.37	0.407
	G-Bond	-1.61	0.196
G-Bond	Transbond XT primer	-2.92*	0.001
	Transbond Plus SEP	2.98*	0.001
	iBOND	1.61	0.196

* mean difference is significantly significant ($p<0.05$)

The ANOVA test showed that there was a statistically significant difference ($p<0.001$) between the mean shear bond strengths of the four groups tested at the immediate debond stage. The Bonferroni post-hoc multiple comparison test identified that Transbond XT primer (11.22±1.98 MPa) had a significantly higher mean shear bond strength when compared to the Transbond Plus SEP

(5.32±1.81 MPa; p<0.001), iBOND (6.69±1.78 MPa; p<0.001) and G-Bond (8.30±2.42 MPa; p<0.01). The tests revealed that G-Bond had a significantly higher shear bond strength compared to the Transbond Plus SEP (p<0.01). Comparison of the shear bond strength of G-Bond to iBOND yielded no significant difference. The coefficient of variations were within normal limits of 20% - 30% except for Transbond Plus SEP which was slightly high (cv=34%). Transbond Plus SEP was also the only bonding agent that exhibited a mean shear bond strength of 5.32±1.81 MPa which is just below the clinically acceptable minimum bond strength of 6 MPa as suggested by Reynolds (1975), but above the acceptable range as suggested by Wiltshire & Noble (2010).

6.1.2 Twenty-four hours Shear Bond Strengths

Table 6-3: Descriptive statistics of the 24hrs shear bond strengths (MPa)

Groups	Sample size (N)	Mean (MPa)	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Transbond XT primer	15	16.65	6.04	2.63	26.87	24.24	36.28%
Transbond Plus SEP	15	13.20	6.43	5.48	22.33	16.85	48.70%
iBOND	15	9.32	3.18	4.85	16.55	11.70	34.11%
G-Bond	14	13.18	4.35	7.18	25.18	18.00	33.00%

Table 6-4: Bonferonni Post-hoc Multiple Comparisons Test of the 24hrs shear bond strengths (MPa)

Groups	Groups in comparison	Mean Difference	Statistical Significance
Transbond XT primer	Transbond Plus SEP	3.44	0.441
	iBOND	7.32*	0.002
	G-Bond	3.46	0.430
Transbond Plus SEP	Transbond XT primer	-3.44	0.441
	iBOND	3.88	0.266
	G-Bond	0.02	1.000
iBOND	Transbond XT primer	-7.32*	0.002
	Transbond Plus SEP	-3.88	0.266
	G-Bond	-3.86	0.274
G-Bond	Transbond XT primer	-3.46	0.430
	Transbond Plus SEP	-0.02	1.000
	iBOND	3.86	0.274

* mean difference is significantly significant ($p<0.05$)

After 24 hours of incubation at 37°C, the ANOVA test revealed that there was a statistically significant difference between the mean shear bond strengths of the four groups tested ($p<0.05$). Specifically, there was a statistically significant difference ($p<0.005$) between Transbond XT primer (16.65±6.04 MPa) and iBOND (9.32±6.43 MPa). After 24 hours, the coefficient of variation increased in all four groups beyond the acceptable 20% - 30% range. However, the mean shear bond strength for all the bonding agents increased over the minimal bond strength of 6-8 MPa as suggested by Reynolds (1975) and 3-4 MPa as suggested by Wiltshire & Noble (2010). The final sample size for G-Bond after 24 hours was 14 due to one sample being accidentally debonded prior to the shear bond strength analysis.

6.1.3 Three month Shear Bond Strengths

Table 6-5: Descriptive statistics of the 3 month shear bond strengths (MPa)

Groups	Sample size (N)	Mean (MPa)	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Transbond XT primer	14	15.31	4.17	8.19	21.90	13.71	27.24%
Transbond Plus SEP	14	13.62	4.58	7.38	21.66	14.28	33.63%
iBOND	15	11.85	5.02	5.32	26.08	20.76	42.45%
G-Bond	15	12.24	3.46	4.25	16.91	12.66	42.45%

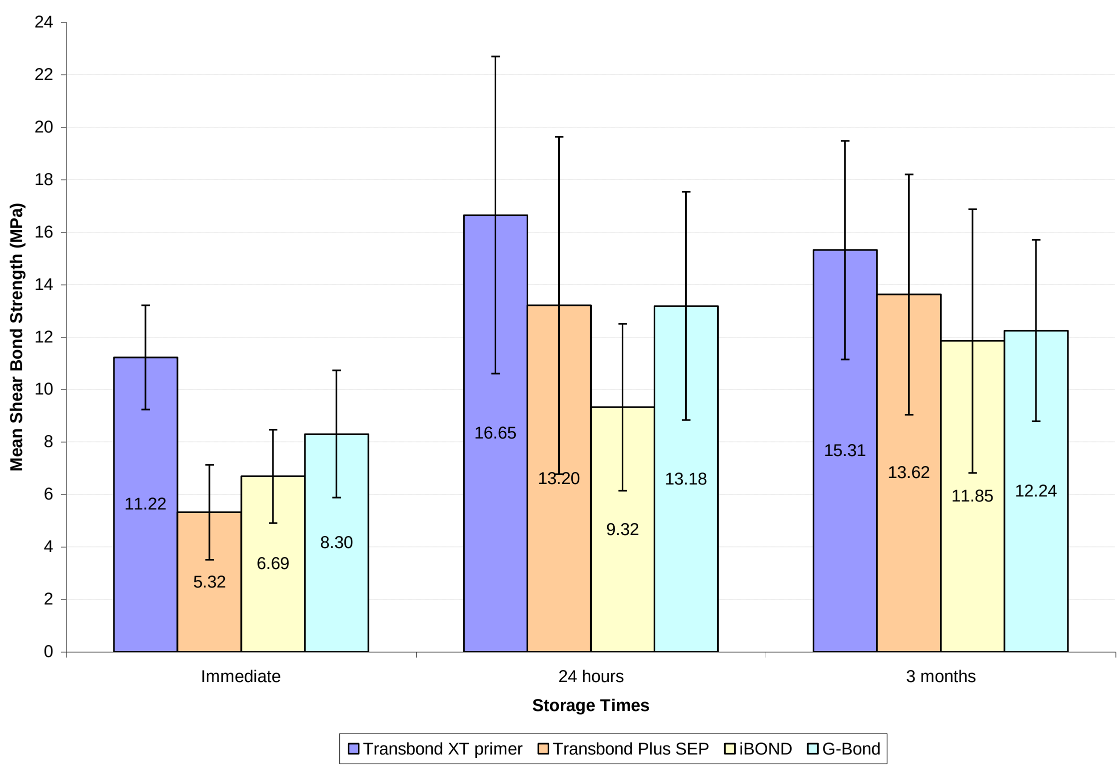
Table 6-6: Bonferonni Post-hoc Multiple Comparisons Test of the 3 months shear bond strengths (MPa)

Groups	Groups in comparison	Mean Difference	Statistical Significance
Transbond XT primer	Transbond Plus SEP	1.70	1.000
	iBOND	3.46	0.201
	G-Bond	3.07	0.351
Transbond Plus SEP	Transbond XT primer	-1.70	1.000
	iBOND	1.77	1.000
	G-Bond	1.37	1.000
iBOND	Transbond XT primer	-3.46	0.201
	Transbond Plus SEP	-1.77	1.000
	G-Bond	-0.40	1.000
G-Bond	Transbond XT primer	-3.07	0.351
	Transbond Plus SEP	-1.37	1.000
	iBOND	0.40	1.000

* mean difference is significantly significant ($p<0.05$)

After 3 months of incubation at 37°C, the ANOVA test found that there was no statistically significant difference between the mean shear bond strengths between the four groups ($p>0.05$). The coefficient of variation showed that only Transbond XT primer had an acceptable value of 27.24% whereas Transbond Plus SEP, iBOND and G-Bond were beyond the range of 20% - 30%. The mean shear bond strength for all the bonding agents were well above the minimal accepted 6-8 MPa as suggested by Reynolds (1975) and 3-4 MPa as suggested by Wiltshire & Noble (2010). The final sample size for Transbond XT primer and Transbond Plus SEP after 3 months is 14 due to one sample from each group being accidentally debonded prior to testing.

Figure 6-1 Mean shear bond strengths over different storage interval times



From
Figure 6-1, during the immediate debonding stage the mean shear bond strength of Transbond XT primer was significantly different from Transbond Plus SEP, iBOND and G-Bond and Transbond Plus SEP was significantly different from G-

Bond. After 24 hours, there was only a significant difference between Transbond XT primer and iBOND and after 3 months there were no significant differences among the four groups.

6.2 Statistics of Shear Bond Strengths over 3 months:

6.2.1 Transbond XT primer

Table 6-7: Descriptive statistics of the shear bond strengths (MPa) over 3 months

Groups	Sample size (N)	Mean	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Immediate	15	11.22	1.98	7.91	14.92	7.01	17.73%
24 hours	15	16.65	6.04	2.63	26.87	24.24	36.28%
3 months	14	15.31	4.17	8.19	21.90	13.71	27.24%

Table 6-8: Bonferonni Post-hoc Multiple Comparisons Test

Groups	Groups in comparison	Mean Difference	Statistical Significance
Immediate	24 hours	-5.42*	0.005
	3 months	-4.09*	0.043
24 hours	Immediate	5.42*	0.005
	3 months	1.33	1.000
3 months	Immediate	4.09*	0.043
	24 hours	-1.33	1.000

* mean difference is significantly significant ($p<0.05$)

The mean shear bond strength of Transbond XT primer changed significantly from immediate (11.22±1.98 MPa) to 24 hours (16.65±6.04 MPa; $p<0.05$) and from immediate to 3 months (15.31±4.17 MPa; $p<0.05$), however, there was no difference in bond strength from 24 hours to 3 months. It can also be seen that the bond strength of Transbond XT primer first increased significantly from immediate to 24 hours, then slightly decreased to after 3 months.

6.2.2 Transbond Plus Self Etch Primer

Table 6-9: Descriptive statistics of the shear bond strengths (MPa) over 3 months

Groups	Sample size (N)	Mean	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Immediate	15	5.32	1.81	2.78	8.83	6.05	34.00%
24 hours	15	13.20	6.43	5.48	22.33	16.85	48.70%
3 months	14	13.62	4.58	7.38	21.66	14.28	33.63%

Table 6-10: Bonferonni Post-hoc Multiple Comparisons Test

Groups	Groups in comparison	Mean Difference	Statistical Significance
Immediate	24 hours	-7.88*	0.000
	3 months	-8.29*	0.000
24 hours	Immediate	7.88*	0.000
	3 months	-0.41	1.000
3 months	Immediate	8.29*	0.000
	24 hours	0.41	1.000

* mean difference is significantly significant ($p<0.05$)

The mean shear bond strength of Transbond Plus SEP changed significantly from immediate (5.32±1.81 MPa), to 24 hours (13.20±6.43 MPa; $p<0.001$) and 3 months (13.62±4.58 MPa; $p<0.001$). However, from 24 hours to 3 months there was no difference in bond strength. At the immediate debond stage, the bond strength was 5.32 MPa which is below the recommended value by Reynolds (1975), but above the value deemed as acceptable by Wiltshire & Noble (2010) based on several clinical trials. The bond strength, however increased significantly after 24 hours ($p<0.005$) and then increased slightly again, although not significantly after 3 months ($p>0.5$).

6.2.3 iBOND Self Etch

Table 6-11: Descriptive statistics of the shear bond strengths (MPa) over 3 months

Groups	Sample size (N)	Mean	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Immediate	15	6.69	1.78	2.84	9.64	6.80	26.59%
24 hours	15	9.32	3.18	4.85	16.55	11.70	34.11%
3 months	15	11.85	5.02	5.32	26.08	20.76	42.45%

Table 6-12: Bonferonni Post-hoc Multiple Comparisons Test

Groups	Groups in comparison	Mean Difference	Statistical Significance
Immediate	24 hours	-2.63	0.153
	3 months	-5.16*	0.001
24 hours	Immediate	2.63	0.153
	3 months	-2.53	0.181
3 months	Immediate	5.16*	0.001
	24 hours	2.53	0.181

* mean difference is significantly significant ($p<0.05$)

The bond strength of iBOND gradually increased over time from immediate (6.69±1.78 MPa) to 24 hours (9.32±3.18 MPa; $p>0.05$) to 3 months (11.85±5.02 MPa; $p>0.05$). However, the change in bond strength from immediate to 24 hours and from 24 hours to 3 months was not significantly different. Only the overall increase from immediate bond strength to 3 months was highly significant ($p<0.001$). The bond strengths at all times were above the recommended minimum as suggested by both Reynolds (1975) and Wiltshire & Noble (2010).

6.2.4 G-Bond

Table 6-13: Descriptive statistics of the shear bond strengths (MPa) over 3 months

Groups	Sample size (N)	Mean	Standard Deviation	Minimum	Maximum	Range	Coefficient of Variation
Immediate	15	8.30	2.42	5.17	13.35	8.18	29.14%
G-Bond	14	13.18	4.35	7.18	25.18	18.00	33.00%
3 months	15	12.24	3.47	4.25	16.91	12.66	42.45%

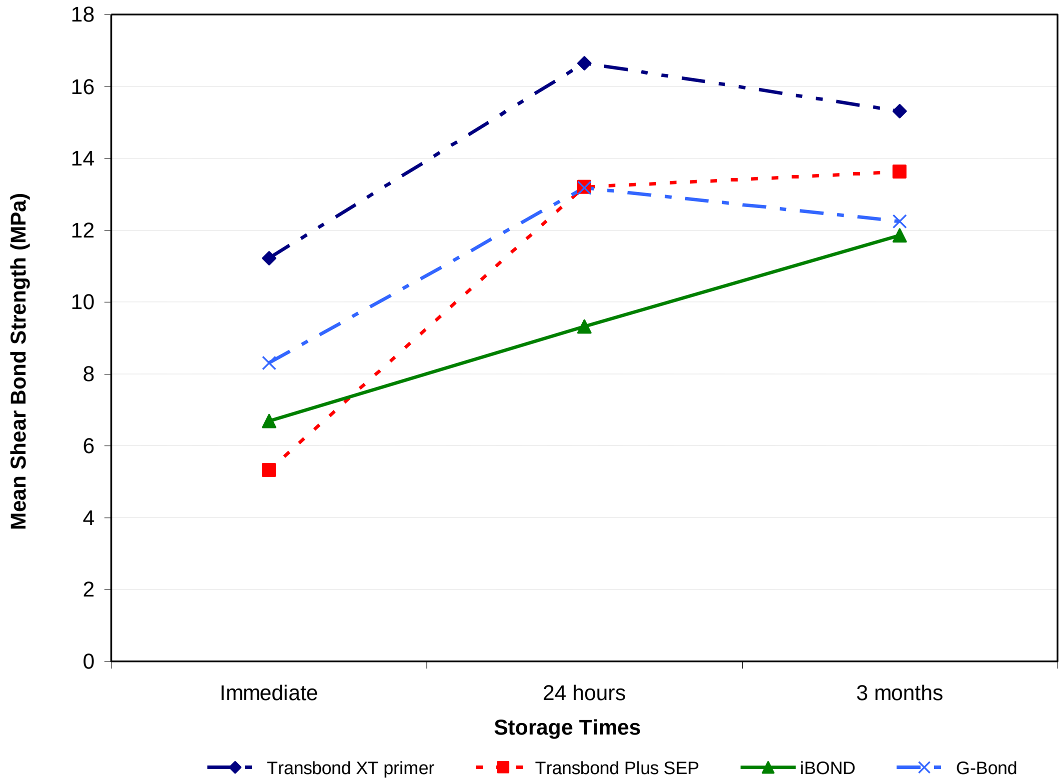
Table 6-14: Bonferonni Post-hoc Multiple Comparisons Test

Groups	Groups in comparison	Mean Difference	Statistical Significance
Immediate	24 hours	-2.63	0.153
	3 months	-5.16*	0.001
24 hours	Immediate	2.63	0.153
	3 months	-2.53	0.181
3 months	Immediate	5.16*	0.001
	24 hours	2.53	0.181

* mean difference is significantly significant (p<0.05)

The mean shear bond strength of G-Bond increased from immediate (8.30±2.42 MPa) to 24 hours (13.18±4.35 MPa; p>0.05), and then decreased slightly after 3 months (12.24±3.47 MPa; p>0.05). However, the changes between the times were not statistically significant. The only highly significant difference in mean shear bond strength was the overall increase bond strength from immediate to 3 months (p<0.001). The bond strengths at all times were above the recommended minimum as suggested by both Reynolds (1975) and Wiltshire & Noble (2010).

Figure 6-2: Mean shear bond strengths of different bonding agents over time



Generally as seen in Figure 6-2, both Transbond XT primer and G-Bond had an initial increase in shear bond strength from immediate debond to 24 hours with a final decrease in bond strength form 24 hours to 3 months. On the other hand, Transbond Plus SEP and iBOND both had a general increase in shear bond strength over time. It can also been seen that only Transbond Plus SEP at the immediate debond time (5.32 MPa) was the only bonding agent to go below the minimal bond strength as recommended by Reynolds (1975), but deemed acceptable according to Wiltshire & Noble (2010). Transbond XT primer always obtained the highest shear bond strength among the tested groups. After 3 months, Transbond XT primer remained with the highest bond strength (15.31 MPa) followed by Transbond Plus SEP (13.62 MPa), G-Bond (12.24 MPa) and iBOND (11.85 MPa), however, the differences among the groups were not statistically significant ($p>0.05$).

6.3 Adhesive Remnant Index:

Table 6-15: Frequency of ARI Scores at all storage intervals

		ARI Score					
Storage Interval	**Bonding Agent**	**1**	**2**	**3**	**4**	**5**	**Total**
Immediate	Transbond XT primer	1	4	5	5	0	15
	Transbond Plus SEP	0	0	0	7	8	15
	iBOND SEP	8	1	5	1	0	15
	G-Bond SEP	9	6	0	0	0	15
24 hours	Transbond XT primer	7	0	4	3	1	15
	Transbond Plus SEP	4	0	1	5	5	15
	iBOND SEP	1	0	2	4	8	15
	G-Bond SEP	4	0	4	3	4	15
3 months	Transbond XT primer	8	3	2	2	0	15
	Transbond Plus SEP	1	1	2	5	6	15
	iBOND SEP	2	0	3	3	7	15
	G-Bond SEP	2	0	1	5	7	15

Table 6-16: Chi-square Analysis at different time intervals

Storage Interval	Chi-square χ^2	Statistical Significance
Immediate	40.0	$p<0.001$
24 hours	10.95	$p<0.05$
3 months	18.92	$p<0.001$

ARI Scores:
1 – 100% left on tooth + bracket impression
2 – >90% left on tooth
3 – 10-90% left on tooth
4 – <10% left on tooth
5 – 0% left on tooth

The Chi-squared analysis at the three different time points revealed statistically significant differences in ARI scores among the four groups. Good debonding scores would be an ARI of 1 or 2 and poor debonding scores would be an ARI of 4 or 5. At the immediate debond stage, G-Bond had the most desirable ARI scores with a combined ARI score of 1 and 2 at 15/15 (100%). Low ARI scores indicate breakage at the adhesive-bracket base interface rather than at the unfavorable enamel-adhesive interface. Breakage at the enamel-adhesive interface increases the risk of enamel tears and fractures. Transbond Plus SEP had the highest ARI with a combined ARI score of 4 and 5 at 15/15 (100%).

After 24 hours, Transbond XT primer had lower debonding scores with 7 samples with an ARI of 1 (7/15). Both Transbond Plus SEP and iBOND had the higher scores of ARI 4 and 5 at 10/15 and 12/15 respectively. After 3 months, Transbond XT primer continued to provide lower scores with a combined ARI of 1 and 2 at 11/15. The remaining bonding agents Transbond Plus SEP, iBOND and G-Bond continued to have higher debonding scores with combine ARI 4 and 5 at 11/15, 10/15 and 12/15, respectively.

After 6 months, the same operator randomly re-evaluated 33% of the samples (operator was blinded to which samples were re-evaluated). The re-evaluation found that 90% of the groups had 100% confirmation of its ARI scores.

7 Discussion

This study evaluated the shear bond strength of two new self-etching primers over three different time points. Immediate shear bond strength was tested to evaluate whether the bonding agent may permit for immediate archwire insertion. Bond strength evaluation over 24 hours and 3 months would provide more long term information on the new self-etching primers. Measuring the shear bond strength over 3 months of time would provide valuable information on the ability of the bonding agent to withstand the forces of mastication and applied orthodontic forces over a longer duration of treatment, particularly the initial 3 month alignment phase.

7.1 Shear Bond Strength

The mean shear bond strength of the buttons tested immediately (<5 minutes) after bonding showed that the control Transbond XT primer (11.22 ± 1.98 MPa) had a significantly higher mean shear bond strength when compared to the Transbond Plus SEP (5.32 ± 1.81 MPa; $p<0.001$), iBOND (6.69 ± 1.78 MPa; $p<0.001$) and G-Bond (8.30 ± 2.42 MPa; $p<0.01$). The immediate shear bond strength for Transbond XT primer (11.22 MPa) was comparable to a study by Turk *et al* (2007) which found Transbond XT primer to have a shear bond strength of 9.50 MPa after 5 minutes. However, in their study he used human premolars using a crosshead speed of 1mm/minute. (Turk *et al*, 2007) Comparing the self-etching primers, G-Bond produced a statistically significantly higher shear bond strength compared to Transbond Plus SEP ($p<0.01$), however, G-Bond and iBOND did not differ significantly. Both of the two new 7th generation bonding systems iBOND and G-Bond had mean shear bond strengths above the minimum recommended 6-8 MPa as suggested by Reynolds (1975) as well as the 3-4 MPa as suggested by Wiltshire & Noble (2010). Thus, Transbond XT primer, iBOND and G-Bond may have adequate bond strength to allow for immediate archwire insertion. (Reynolds, 1975; Wiltshire & Noble, 2010)

Interestingly, Transbond Plus SEP (5.32±1.81 MPa) a self-etching primer marketed for orthodontic bonding did not perform well at the immediate debond time period. There have been several studies that have tested the immediate bond strength of Transbond Plus SEP. This bond strength was similar to that found by Bishara *et al* (2004) which found Transbond Plus SEP to have shear bond strength of 5.9 MPa that was debonded within 30 minutes of curing. (Bishara *et al*, 2004; Bishara *et al*, 2008) On the other hand, Turk *et al* (2007) found the shear bond strength of Transbond Plus SEP 5 minutes after bonding to be 8.97 MPa, which is approximately 4 MPa higher than the current study. (Turk *et al*, 2007) The mean shear bond strength was below the 6-8 MPa as recommended by Reynolds (1975), however, it was above the 3-4 MPa as suggested by Wiltshire & Noble (2010). Looking at the minimum value at 2.78 MPa the bond strength of Transbond Plus SEP may at times be compromised. Thus, Transbond Plus SEP may be able to withstand immediate archwire insertion, especially if the forces of the archwire are kept light, however bond strength may be compromised in some cases. (Reynolds, 1975; Wiltshire & Noble, 2010)

After 24 hours of incubation, the only significantly different bond strength was Transbond XT primer (16.65±6.04 MPa) which had a higher bond strength than iBOND (9.32±6.43 MPa; $p<0.005$). Even though, they were significantly different, the shear bond strength of iBOND was still considered adequate for orthodontic bonding. (Reynolds, 1975; Wiltshire & Noble, 2010) It is interesting to see that over 24 hours Transbond XT primer and Transbond Plus SEP had a statistically significant increase in their bond strengths. From immediate to 24 hours (Figure 2), the shear bond strength of Transbond XT primer increased approximately 5 MPa from 11.22±1.98 MPa to 16.65±6.04 MPa ($p<0.05$) and Transbond Plus SEP increased approximately 8 MPa from 5.32±1.81 MPa to 13.20±6.43 MPa ($p<0.001$). This significant increase in bond strength for the Transbond Plus SEP suggests that it may be prudent to tie-in very light archwires or wait 24 hours prior to archwire tie-in, however, this may prove challenging from a clinical standpoint. This result was similar to that found by Turk *et al* (2007) who also found a significant increase ($p<0.001$) from immediate (5 minutes) debond to 24 hours debond for both Transbond XT primer (5min: 9.50 MPa; 24h: 16.82 MPa) and Transbond Plus SEP (5min: 8.97 MPa; 24h: 19.11 MPa). (Turk *et al*, 2007) G-Bond and iBOND also had an increase in their shear bond strengths after 24 hours, however it was not a statistically significant increase.

A possible reason for this overall trend in increasing bond strength over 24 hours is that only about 75% of the polymerization takes place during the first 10 minutes and that the curing reaction continues for a period of 24 hours. This is most likely due to most of the free radicals are being produced at the periphery of the resin where the light is able to penetrate. After 24 hours most of the polymerization should have been completed as the resin further polymerizes under the bracket base. (Klocke *et al*, 2004; Powers *et al*, 2006; Turk *et al*, 2007) A study by Oesterle *et al* (2008) also supports our results of maximum shear bond strength after 24 hours. They looked at the effect of composite aging on the shear bond strength of orthodontic brackets. After testing two different types of bonding systems stored in distilled water at 37°C for 30 minutes, 24 hours, and 1, 6, 12, 18, and 24 months before shear-peel testing. Their results found that the maximum shear bond strength occurred after 24 hours (20.99 MPa) and began to decrease over the next 24 months. (Oesterle & Shellhart, 2008)

At 3 months, an ANOVA revealed that all four bonding agents had a shear bond strength that was not significantly different from one another. However, they all had mean shear bond strengths and minimum shear bond strength greater than those recommended by Reynolds and Wiltshire & Noble. (Reynolds, 1975; Wiltshire & Noble, 2010) It can also be seen that the change in shear bond strength from 24 hours to 3 months were also not statistically significant among all the four bonding agents. However, it is interesting to note that only iBOND and Transbond Plus SEP had a continual rise in shear bond strength, whereas with Transbond XT primer and G-Bond their shear bond strength slightly declined, although not statistically significant. A study by Trites *et al* (2004) showed similar results in which both Transbond XT primer and Transbond Plus SEP had no significant change in shear bond strength between 24 hours and 3 months. Similarly, there was also no significant difference between Transbond XT primer and Transbond Plus SEP at the 3 month debond time ($p>0.05$). (Trites *et al*, 2004)

Over all three time periods, Transbond XT primer had higher shear bond strength when compared to the other bonding agents. In particular, it had a statistically significantly higher shear bond strength during immediate debond ($p<0.005$), and statistically significantly higher than iBOND after 24 hours ($p<0.005$). A reason for this may be due to the increase depth of the etched enamel by the 35% phosphoric acid. A deeper penetration into the enamel may allow for larger and longer resin tags which may have contributed to a higher bond strength. Øgaard *et al* (2010) was able to look at the interface between the enamel and the adhesive using a scanning electron microscope. They found enamel that had conventional etching and bonding had long and thick resin tags varying from a few microns to 20μm. In contrast, the enamel treated with a self-etching primer showed thin and short resin tags ranging from 5 to 10μm. (Øgaard & Fjeld, 2010)

Previous studies on iBOND were limited and those that studied iBOND found mixed results on its possible use for orthodontic bonding. The results of this study were in line with those of Holzmeier *et al* (2008) and Paschos *et al* (2008) (see Table 7-1). However, it must be kept in mind that the methodologies used in these studies varied in terms of cross-head speeds, types of teeth used, type of bracket used, and thermocycling, thus, making direct comparisons would be difficult. Holzmeier used bovine teeth and found that iBOND had a mean shear bond strength of 8.1 MPa after 24 hours of incubation at 37°C and a cross-head speed of 1mm/min. Their results were comparable to this study where iBOND had 24 hour mean shear bond strength of 9.32 MPa. A study by Paschos (2008) used human premolars and a cross-head speed of 0.5mm/min and showed iBOND to have a mean shear bond strength of 11.3 MPa after 30 days of incubation at 37°C and thermocycled between 5°C and 55°C for 1300 cycles. There results were also in line with our shear bond strength of iBOND at 11.85 MPa over 3 months. Both of these studies concluded that iBOND may have adequate bond strength to be used to bond orthodontic brackets.

A more direct comparison can be made with a study by Wiltshire & Karaiskos (2005). Their experiment was performed at the same institution using the same universal testing machine and cross-head speed (0.5mm/min) as this study. They also tested the shear bond strength of iBOND after 24 hours of incubation. Their results showed higher shear bond strength (see Table 7-1) compared to this study as well as previous studies on iBOND after 24 hours incubation. Some possible reason for these differences may be due to operator technique, load cell weight and/or material batch number. (Wiltshire & Karaiskos, 2005)

Table 7-1: Comparison of shear bond strength of iBOND after 24 hours

		iBOND
Authors	**Year**	**Shear Bond Strength after 24 hours**
Wiltshire & Karaiskos	2005	16.8 MPa
Holzmeier	2008	8.1 MPa
Minick	2009	3.86 MPa
Ho (current study)	2010	9.32 MPa

Minick (2009) used bovine teeth to look at several new bonding systems and primers including iBOND. The teeth were incubated for 30 minutes and 24 hours at 37°C. The bond strengths for iBOND were found to be 3.91 MPa at 30 minutes and 3.86 MPa at 24 hours. At both incubation times, the shear bond strength of iBOND were below those found in this study (see Table 7-1) and were also below the minimal amount of 6-8 MPa as recommended by Reynolds, but above the 3-4 MPa as recommended by Wiltshire & Noble. Thus, at the time of publication, the authors concluded that iBOND may not have adequate strength for clinical orthodontic use. (Reynolds, 1975; Minick *et al*, 2009; Wiltshire & Noble, 2010)

At the time of this study there were no previously published studies on the use of G-Bond to bond orthodontic brackets to enamel. A search of G-Bond on the PubMed website only resulted in publications from a restorative perspective looking at the shear bond strength of composite resin to enamel and dentin. The restorative studies by Burrow (2008) and Söderholm (2008) showed that G-Bond had a mean shear bond strength of 27.1 MPa and 14.9 MPa to enamel, respectively. Because these studies investigated the shear bond strength of composite resin rather than metal brackets or buttons, it would be difficult hypothesize how G-Bond would perform when bonding orthodontic brackets to teeth. (Burrow *et al*, 2008; Söderholm *et al*, 2008)

The main purpose of this study was to determine whether these two new self-etching primers iBOND and G-Bond would have the ability to be used as bonding agents in orthodontics. From their mean shear bond strengths over the three time periods, it seems that they will be able to withstand the masticatory and orthodontic forces throughout treatment. However, from a clinical efficiency viewpoint the use of Transbond Plus SEP out performed both iBOND and G-Bond in terms of the amount of time it takes to bond a single bracket onto a tooth. Based on the manufacturer's recommended amount of time needed at each step, the total amount of time to bond a single tooth are as follows: iBOND: 62 seconds, G-Bond: 40 seconds and Transbond Plus SEP: 27 seconds (see Table 5-4). Thus, the time required to fully bond an arch would be much quicker with the use of Transbond Plus SEP as compared to the other two SEPs.

7.2 Adhesive Remnant Index Scores

The Adhesive Remnant Index (ARI) is a method to help determine the frequency of the location of where debond occurs. A low score would indicate an adhesive failure at the bracket/composite resin interface which is desirable during orthodontic debonding because it leaves the composite resin on the tooth and avoids enamel tearing and damage. Conversely, a high score would indicate an adhesive failure at the enamel/composite resin interface. The Chi-squared analysis at the three different time points revealed statistically significant differences in ARI scores among the four groups.

At the immediate debond stage, G-Bond had the most desirable ARI scores with a combined ARI score of 1 and 2 at 15/15 (100%) leaving >90% of the composite resin on the tooth. Transbond Plus SEP had the highest ARI score with a combined score of 4 and 5 (<10% of composite resin on the tooth) at 15/15 (100%). In contrast, previous studies by Bishara *et al* (2006, 2008) found Transbond Plus SEP to have less frequent high ARI scores which ranged from 5% to 35% of a combined score of 4 and 5. (Bishara *et al*, 2007; Bishara *et al*, 2008)

After 24 hours, Transbond XT primer had low debonding scores with 7 samples with a desirable ARI score of 1 (47%). This result was similar to Scougall Vilchis *et al* (2009) with 40% of the samples leaving 100% of the composite resin left on the tooth, but in contrast with Trites *et al* (2004) and Turk *et al* (2007) with low frequencies of 3.3% and 10%, respectively. (Trites *et al*, 2004; Turk *et al*, 2007; Scougall Vilchis *et al*, 2009) Both Transbond Plus SEP and iBOND had the highest scores with a combined ARI score of 4 and 5 at 10/15 (67%) and 12/15 (80%) respectively. In contrast to other studies, Transbond Plus SEP ARI scores all had a low frequency of ARI scores that would have indicated little to no composite resin on the tooth; ranging from 0% to 17.1%. (Trites *et al*, 2004; Turk *et al*, 2007; Scougall Vilchis *et al*, 2009) The ARI scores for iBOND was similar to that found by Holzmeier (2008) with a combined ARI score of 4 and 5 at 100% and Minick (2009) with 100% of the samples having <50% of the composite resin left on the tooth.

After 3 months, Transbond XT primer continued to reduce its ARI score with a combined ARI of 1 and 2 at 11/15 (73%). The remaining bonding agents Transbond Plus SEP, iBOND and G-Bond continued to have high ARI scores with a combined ARI score of 4 and 5 at 11/15 (73%), 10/15 (67%) and 12/15 (80%), respectively. Trite *et al* (2004) found that after 3 months Transbond XT primer had 83% of the samples had <50% composite resin left on the tooth after debond, which is in contrast to the current study where 73% of the samples had >90% composite resin left on the tooth after debond.

A possible reason that Transbond XT primer had a greater number of low ARI scores (>90% of composite resin left on tooth) after 3 months can possibly be linked to the greater bond strength due to a deeper penetration of its resin tags. (Holzmeier *et al*, 2008; Øgaard & Fjeld, 2010) A stronger bond between the enamel and adhesive resin would increase the likelihood of bond failure at the bracket/composite resin interface which would be desirable during orthodontic debonding. The shallow penetration of the resin tags achieved by self-etching primers may be the reason for an increased bond failure at the enamel/resin composite interface as seen by the ARI scores of Transbond Plus SEP, iBOND and G-Bond after 3 months. (Øgaard & Fjeld, 2010)

7.3 Limitations of and Recommendations from the present study

Due to the lack of standardization in bond testing protocols, it is difficult to make comparisons to other studies. There were several factors and protocols not performed in this study that would have improved it making it more clinically realistic. An example of this would be storing the brackets in artificial saliva and thermocycling between 5°C - 55°C throughout the incubation period. Although thermocycling in artificial saliva may provide a more realistic clinical environment, it may be argued how often do orthodontic patients have a change in oral temperature ranging between 5°C - 55°C. In 2008, Elekdag-Turk *et al* studied the effects of thermocycling on shear bond strength on both conventional bonding and self-etching primers. They discovered that no thermocycling, thermocycling at 2000 cycles or 5000 cycles had no effect on the shear bond strength for conventional bonding. However, for self-etching primers, there was significant decrease in shear bond strength with 2000 and 5000 thermocycles when compared to no thermocycling. (Elekdag-Turk *et al*, 2008b) Thus, for self-etching primers the decision to thermocycle or not will affect the resulting shear bond strength of the bonding agent. We are of the opinion, however, that thermocycling is too rigorous and does not mimic the clinical situation, it merely tests the materials at the limits of their performance.

Also, within this study the time periods evaluated were immediate, 24 hours and 3 months after bonding. It would have been interesting to evaluate the shear bond strength after 6, 12, 18, and 24 months. The average orthodontic treatment time is 2 years, thus it would be valuable to evaluate the shear bond strength at this time since it is when the brackets are debonded from the tooth. Bond strength not only plays an important role during treatment to withstand masticatory and orthodontic forces, but also at the time of debond. Too strong of a bond may result in enamel tearing and fracture which are undesirable.

8 Conclusions

Based on this *in vitro* study on the SBS of Transbond XT primer (control), Transbond Plus SEP and the two new 7th generation SEP iBOND and G-Bond, we can conclude that:

1. Transbond XT primer, iBOND or G-Bond potentially produce adequate bond strength of orthodontic buttons to human enamel for successful immediate archwire tie-in.
2. 24 hours and 3 months after bonding with Transbond XT primer, Transbond Plus SEP, iBOND and G-Bond all maintain sufficient bond strength to potentially withstand occlusal and archwire forces.
3. Transbond XT primer, Transbond Plus SEP, iBOND and G-Bond all had an increase in bond strength after the first 24 hours.
4. The ARI score after 3 months, Transbond XT primer had the most adhesive remnants remaining on the tooth and Transbond Plus SEP, iBOND and G-Bond had the least adhesive remnants remaining on the tooth. This may indicate that Transbond XT primer has the least chance of enamel fracture, tears, crazing during debonding compared to the other tested bonding agents.

9 Raw Data

Table 9-1: Raw data for immediate shear bond strengths

Sample	Transbond XT primer	Transbond Plus	iBOND	G-BOND
1	14.92	6.30	6.11	13.35
2	9.75	5.99	7.30	6.82
3	10.51	6.34	4.19	7.00
4	9.69	5.92	6.49	10.70
5	13.93	4.43	9.52	9.15
6	11.17	8.83	8.18	11.05
7	10.85	5.76	7.85	7.01
8	9.91	4.34	2.84	5.17
9	11.80	3.06	6.80	6.04
10	9.80	7.37	5.56	12.08
11	13.20	3.54	5.89	7.94
12	14.07	4.49	6.80	7.71
13	9.85	2.78	6.11	6.73
14	10.97	3.21	9.64	6.32
15	7.91	7.49	7.12	7.50

Table 9-2: Raw data for 24 hours shear bond strengths

Sample	Transbond XT primer	Transbond Plus	iBOND	G-BOND
1	17.21	18.37	10.77	0.00
2	16.88	20.93	5.04	10.36
3	17.58	6.53	13.48	13.74
4	17.94	16.24	11.20	16.46
5	19.14	5.76	16.55	12.91
6	22.38	9.61	4.85	7.18
7	22.56	5.48	10.35	13.87
8	2.63	18.68	10.39	8.25
9	17.89	15.23	9.15	13.33
10	15.71	22.33	4.99	10.31
11	21.11	18.80	7.80	11.90
12	26.87	8.13	9.50	17.34
13	10.81	5.84	7.31	9.56
14	10.41	6.67	9.92	25.18
15	10.57	19.46	8.55	13.33

Table 9-3: Raw data for 3 months shear bond strengths

Sample	Transbond XT primer	Transbond Plus	iBOND	G-BOND
1	0.00	9.00	11.57	16.84
2	16.47	12.29	14.06	14.63
3	15.36	7.38	7.63	4.25
4	10.81	18.38	9.60	12.82
5	13.55	0.00	10.12	7.93
6	20.18	11.61	9.21	13.99
7	20.60	12.39	15.49	11.55
8	9.75	11.14	15.32	16.91
9	18.22	17.20	7.20	9.20
10	8.19	13.06	14.75	10.94
11	19.22	8.06	26.08	10.67
12	15.23	9.35	10.54	10.36
13	21.90	21.66	8.25	14.95
14	13.92	17.89	5.32	14.38
15	11.27	20.83	12.60	14.25

10 References

Aljubouri YD, Millett DT, Gilmour WH. Six and 12 months' evaluation of a self-etching primer versus two-stage etch and prime for orthodontic bonding: a randomized clinical trial. Eur J Orthod 2004; 26 (6):565-71.

Arnold RW, Combe EC, Warford JH, Jr. Bonding of stainless steel brackets to enamel with a new self-etching primer. Am J Orthod Dentofacial Orthop 2002; 122 (3):274-6.

Årtun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984; 85 (4):333-40.

Asgari S, Salas A, English J et al. Clinical evaluation of bond failure rates with a new self-etching primer. J Clin Orthod 2002; 36 (12):687-9.

Banks P, Thiruvenkatachari B. Long-term clinical evaluation of bracket failure with a self-etching primer: a randomized controlled trial. J Orthod 2007; 34 (4):243-51.

Bishara SE, Ajlouni R, Laffoon JF et al. Effect of a fluoride-releasing self-etch acidic primer on the shear bond strength of orthodontic brackets. Angle Orthod 2002; 72 (3):199-202.

Bishara SE, Gordan VV, VonWald L et al. Shear bond strength of composite, glass ionomer, and acidic primer adhesive systems. Am J Orthod Dentofacial Orthop 1999a; 115 (1):24-8.

Bishara SE, Oonsombat C, Ajlouni R et al. Comparison of the shear bond strength of 2 self-etch primer/adhesive systems. Am J Orthod Dentofacial Orthop 2004; 125 (3):348-50.

Bishara SE, Ostby AW, Laffoon J et al. A self-conditioner for resin-modified glass ionomers in bonding orthodontic brackets. Angle Orthod 2007; 77 (4):711-5.

Bishara SE, Otsby AW, Ajlouni R et al. A new premixed self-etch adhesive for bonding orthodontic brackets. Angle Orthod 2008; 78 (6):1101-4.

Bishara SE, VonWald L, Laffoon JF et al. Effect of a self-etch primer/adhesive on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2001; 119 (6):621-4.

Bishara SE, VonWald L, Olsen ME et al. Effect of time on the shear bond strength of glass ionomer and composite orthodontic adhesives. Am J Orthod Dentofacial Orthop 1999b; 116 (6):616-20.

Brantley WA, Eliades T. Orthodontic materials : scientific and clinical aspects. Stuttgart ; New York, NY: Thieme; 2001. x, 310 p. p.

Brosnihan J, Safranek L. Orthodontic bonding: the next generation. J Clin Orthod 2000; 34 (10):614-6.

Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955; 34 (6):849-53.

Burrow MF, Kitasako Y, Thomas CD et al. Comparison of enamel and dentin microshear bond strengths of a two-step self-etching priming system with five all-in-one systems. Oper Dent 2008; 33 (4):456-60.

Buyukyilmaz T, Usumez S, Karaman AI. Effect of self-etching primers on bond strength--are they reliable? Angle Orthod 2003; 73 (1):64-70.

Cal-Neto JP, Miguel JA. Scanning electron microscopy evaluation of the bonding mechanism of a self-etching primer on enamel. Angle Orthod 2006; 76 (1):132-6.

Cal-Neto JP, Quintao CA, Almeida MA et al. Bond failure rates with a self-etching primer: a randomized controlled trial. Am J Orthod Dentofacial Orthop 2009; 135 (6):782-6.

Cehreli ZC, Kecik D, Kocadereli I. Effect of self-etching primer and adhesive formulations on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2005; 127 (5):573-9; quiz 625-6.

Craig RG, Powers JM. Restorative dental materials. 11th ed. St. Louis: Mosby; 2002. xvi, 704 p. p.

dos Santos JE, Quioca J, Loguercio AD et al. Six-month bracket survival with a self-etch adhesive. Angle Orthod 2006; 76 (5):863-8.

Elekdag-Turk S, Cakmak F, Isci D et al. 12-month self-ligating bracket failure rate with a self-etching primer. Angle Orthod 2008a; 78 (6):1095-100.

Elekdag-Turk S, Turk T, Isci D et al. Thermocycling effects on shear bond strength of a self-etching primer. Angle Orthod 2008b; 78 (2):351-6.

Eliades T, Brantley WA. The inappropriateness of conventional orthodontic bond strength assessment protocols. Eur J Orthod 2000; 22 (1):13-23.

Farah J, Powers J. The Dental Advisor: Self-etching bonding agents. J Can Dent Assoc 2004; 70 (6):446-7.

Fricker JP. A 12-month clinical evaluation of a light-activated glass polyalkenoate (ionomer) cement for the direct bonding of orthodontic brackets. Am J Orthod Dentofacial Orthop 1994; 105 (5):502-5.

Fritz UB, Diedrich P, Finger WJ. Self-etching primers--an alternative to the conventional acid etch technique? J Orofac Orthop 2001; 62 (3):238-45.

G-Bond: Advanced 7th Generation Single Component Adhesive. Alsip: GC America Inc.

Holzmeier M, Schaubmayr M, Dasch W et al. A new generation of self-etching adhesives: comparison with traditional acid etch technique. J Orofac Orthop 2008; 69 (2):78-93.

iBOND Self Etch: Scientific Information. Hanau: Heraeus Kulzer.

Ireland AJ, Knight H, Sherriff M. An in vivo investigation into bond failure rates with a new self-etching primer system. Am J Orthod Dentofacial Orthop 2003; 124 (3):323-6.

Katona TR. The effects of load location and misalignment on shear/peel testing of direct bonded orthodontic brackets--a finite element model. Am J Orthod Dentofacial Orthop 1994; 106 (4):395-402.

Khalha A. No difference in the failure rates of orthodontic brackets bonded with self-etching primer. Evid Based Dent 2008; 9 (3):79-80.

Klocke A, Shi J, Vaziri F et al. Effect of time on bond strength in indirect bonding. Angle Orthod 2004; 74 (2):245-50.

Kugel G, Ferrari M. The science of bonding: from first to sixth generation. J Am Dent Assoc 2000; 131 Suppl:20S-5S.

Manning N, Chadwick SM, Plunkett D et al. A randomized clinical trial comparing 'one-step' and 'two-step' orthodontic bonding systems. J Orthod 2006; 33 (4):276-83; discussion 56-7.

Material Safety Data Sheet: Transbond Plus Self Etching Primer. St. Paul: 3M, January 6, 2005 Report No.: 11-8348-2.

Material Safety Data Sheet: Transbond XT Etching Gel System. 3M, July 10, 2008 Report No.: 18-4979-3.

Material Safety Data Sheet: Transbond XT Light Cure Adhesive. 3M, July 10, 2008 Report No.: 07-6174-2.

Material Safety Data Sheet: Transbond XT Primer. 3M, January 6, 2005 Report No.: 11-9309-3.

Miller RA. Laboratory and clinical evaluation of a self-etching primer. J Clin Orthod 2001; 35 (1):42-5.

Minick GT, Oesterle LJ, Newman SM et al. Bracket bond strengths of new adhesive systems. Am J Orthod Dentofacial Orthop 2009; 135 (6):771-6.

Mitchell CA. Dental materials in operative dentistry. London ; Chicago: Quintessence Pub.; 2008. 128 p. p.

Murfitt PG, Quick AN, Swain MV et al. A randomised clinical trial to investigate bond failure rates using a self-etching primer. Eur J Orthod 2006; 28 (5):444-9.

Nemeth BR, Wiltshire WA, Lavelle CL. Shear/peel bond strength of orthodontic attachments to moist and dry enamel. Am J Orthod Dentofacial Orthop 2006; 129 (3):396-401.

Newman GV. Epoxy adhesives for orthodontic attachments: progress report. Am J Orthod 1965; 51 (12):901-12.

Oesterle LJ, Shellhart WC. Effect of aging on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2008; 133 (5):716-20.

Øgaard B, Fjeld M. The enamel surface and bonding in orthodontics. Semin Orthod 2010; 16 (1):37-48.

Pandis N, Polychronopoulou A, Eliades T. Failure rate of self-ligating and edgewise brackets bonded with conventional acid etching and a self-etching primer: a prospective in vivo study. Angle Orthod 2006; 76 (1):119-22.

Paschos E, Westphal JO, Ilie N et al. Artificial saliva contamination effects on bond strength of self-etching primers. Angle Orthod 2008; 78 (4):716-21.

Powers JM, Kim HB, Turner DS. Orthodontic adhesives and bond strength testing. Semin Orthod 1997; 3 (3):147-56.

Powers JM, Sakaguchi RL, Craig RGRdm. Craig's restorative dental materials. 12th ed. / John M. Powers and Ronald L. Sakaguchi. ed. St. Louis, Mo. ; [London]: Mosby Elsevier; 2006. xvii, 632 p. p.

Proffit WR, Fields HW, Sarver DM. Contemporary orthodontics. 4th ed. St. Louis, Mo.: Mosby Elsevier; 2007. xii, 751 p. p.

Reis A, dos Santos JE, Loguercio AD et al. Eighteen-month bracket survival rate: conventional versus self-etch adhesive. Eur J Orthod 2008; 30 (1):94-9.

Reynolds I. A review of direct orthodontic bonding. Br J Orthod 1975; 2:171-8.

Scougall Vilchis RJ, Yamamoto S, Kitai N et al. Shear bond strength of orthodontic brackets bonded with different self-etching adhesives. Am J Orthod Dentofacial Orthop 2009; 136 (3):425-30.

Söderholm KJ, Soares F, Argumosa M et al. Shear bond strength of one etch-and-rinse and five self-etching dental adhesives when used by six operators. Acta Odontol Scand 2008; 66 (4):243-9.

Stanford SK, Wozniak WT, Fan PL. The need for standardization of test protocols. Semin Orthod 1997; 3 (3):206-9.

Trites B, Foley TF, Banting D. Bond strength comparison of 2 self-etching primers over a 3-month storage period. Am J Orthod Dentofacial Orthop 2004; 126 (6):709-16.

Turk T, Elekdag-Turk S, Isci D. Effects of self-etching primer on shear bond strength of orthodontic brackets at different debond times. Angle Orthod 2007; 77 (1):108-12.

Van Noort R. Introduction to dental materials. 3rd ed. Edinburgh ; New York: Mosby/Elsevier; 2007. viii, 308 p. p.

Velo S, Carano A, Carano A. Self-etching vs. traditional bonding systems in orthodontics: an in vitro study. Orthod Craniofac Res 2002; 5 (3):166-9.

White LW. An expedited indirect bonding technique. J Clin Orthod 2001; 35 (1):36-41.

Wiltshire W, Karaiskos N. Comparison of shear bond strengths of three self-etching primers. World J Orthod 2005; 6 (suppl) (177):abstracts SL060.

Wiltshire WA, Noble J. Clinical and Laboratory Perspectives of Improved Orthodontic Bonding to Normal, Hypoplastic, and Fluorosed Enamel. Semin Orthod 2010; 16 (1):55-65.

11 Appendix

11.1 Ethics Approval

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Research Ethics Boards

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APPROVAL FORM

Principal Investigator: Dr. A. Ho
Supervisor: Dr. T. Bonstein

Protocol Reference Number: H2008:201
Date of Approval: July 14, 2008
Date of Expiry: July 14, 2009

Protocol Title: "Shear Bond Strength Comparisons of New Self-Etching Primers Over a 3 Month Period"

The following is/are approved for use:

- **Proposal submitted July 14, 2008**

The above underwent expedited review and was **approved as submitted** on July 14, by Dr. John Arnett, Ph.D., C. Psych., Health Research Ethics Board, Bannatyne Campus, University of Manitoba on behalf of the committee per your letter received July 14, 2008. The Research Ethics Board is organized and operates according to Health Canada/ICH Good Clinical Practices, Tri-Council Policy Statement, and the applicable laws and regulations of Manitoba. The membership of this Research Ethics Board complies with the membership requirements for Research Ethics Boards defined in Division 5 of the *Food and Drug Regulations.*

This approval is valid for one year only. A study status report must be submitted annually and must accompany your request for re-approval. Any significant changes of the protocol and informed consent form should be reported to the Chair for consideration in advance of implementation of such changes. The REB must be notified regarding discontinuation or study closure.

This approval is for the ethics of human use only. For the logistics of performing the study, approval must be sought form the relevant institution, if required.

Sincerely yours,

John Arnett, Ph.D., C. Psych.
Chair, Health Research Ethics Board
Bannatyne Campus

Please quote the above protocol reference number on all correspondence.
Inquiries should be directed to REB Secretary
Telephone: (204) 789-3883 / Fax: (204) 789-3414

11.2 Journal article

Shear bond strengths of two new self-etching primers

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Abstract

Introduction: The purpose of this study was to evaluate the *in vitro* shear bond strength (SBS) of orthodontic attachments using 2 self-etching primers (SEP): iBOND and G-Bond.

Methods: 180 human molars were randomly divided into 4 groups and again into 3 sub-groups with 15 teeth each. Teeth were bonded with a stainless steel button (GAC International) using Transbond XT adhesive composite. The bonding agents were 1.iBOND 2.G-Bond 3.Transbond Plus SEP and 4.Transbond XT primer (control). Shear Bond Strengths (SBS) immediately, and at 24 hours and 3 months were measured using a universal testing machine. Adhesive Remnant Index (ARI) scores were also evaluated.

Results: Transbond XT primer (11.22±1.99MPa) had a significantly higher immediate ($p<0.05$) SBS when compared to the Transbond Plus SEP (5.32±1.81MPa), iBOND (6.69±1.78MPa) and G-Bond (8.30±2.42MPa). After 24 hours, Transbond XT primer (16.65±6.04MPa) and Transbond Plus SEP (13.20±6.43MPa) had a significant increase ($p<0.05$) in their SBS. At 3 months, all four bonding agents had SBS that were not significantly different from one another. Chi-squared comparisons of ARI indicated a significant difference ($p<0.05$) between the groups at all time points.

Conclusions: iBOND and G-Bond yielded sufficient *in vitro* SBS's over the three time points that may well be sufficient to withstand the alignment and occlusal

forces imparted by light archwires during immediate arch wire tie-in and over the initial levelling and alignment phase.

Introduction

Buonocore introduced the use of an acid etch in dentistry in 1955. [1] Newman [2] used the acid etch technique to bond orthodontic brackets to teeth ultimately eliminating the need for banding of teeth, which allowed clinicians to more efficiently treat patients with greater patient comfort, elimination of pretreatment tooth separation, improving oral hygiene and esthetics, and reducing chair time.

Adequate orthodontic bond strength of a bracket is an important aspect of clinical orthodontics – without it practicing orthodontics can become frustrating for both the practitioner and the patient. Broken brackets will cause increased treatment time, added costs of materials and personnel time, and additional visits by the patient. The bond strengths of orthodontic attachments must be able to withstand the masticatory forces of the patient and any orthodontic forces generated by the archwires.

Self-etching primers (SEPs) are considered 6th and 7th generation bonding agents. The 6th generation self-etching primers still require mixing of the primer and adhesive components with no separate etching required. [3,4] However, the 7th generation self-etching primers are single component bottles that requiring no mixing or etching. [3] Both *in vitro* [5,6] and *in vivo* [7-15] studies suggest that the use of SEPs in orthodontics has been found to be effective when bonding brackets to tooth enamel. There are several advantages of using SEPs during orthodontic bonding which include allowance for primer and etchant to simultaneously penetrate into the enamel, reducing technical errors, eliminating cross-contamination and shortening clinical chair time due to the elimination of several steps such as enamel etching. [16]

iBOND Self Etch Primer (Heraeus Kulzer, Hanau, Germany) is a 7th generation all-in-one bonding agent. It does not require any mixing and needs only a single application. Holzmeier *et al* [17] studied several SEPs *in vitro* including iBOND and compared them to Transbond Plus SEP and the conventional Transbond XT bonding system (primer + 35% phosphoric acid). According to their results iBOND had a shear bond strength (SBS) (8.1 MPa) significantly lower than Transbond Plus SEP (20.7 MPa) and Transbond XT bonding system (21.0 MPa). [17] In a study by Paschos *et al* [18], iBOND had a mean SBS of 11.3 MPa. Both studies concluded that iBOND may be clinically acceptable to use for orthodontic bonding of brackets to teeth due to the satisfaction of the minimal bond strength requirement. [19,20] G-Bond (GC Corporation, Tokyo, Japan) is another all-in-one 7th generation bonding system. Currently, there were no known published studies on the use of G-Bond to bond orthodontic brackets to enamel.

The purpose of this study was to evaluate and compare the SBS of two 7th generation bonding systems: iBOND Self Etch and G-Bond compared to the 6th generation Transbond Plus SEP (3M Unitek, Monrovia, CA) and the conventional Transbond XT primer (control) (3M Unitek, Monrovia, CA).

Materials & Methods

One-hundred and eighty extracted human molars were collected and stored in distilled water with 0.5% Chloramine T to prevent bacterial growth. Exclusion criteria for the extracted molars included visible caries, restorations, defects and/or anomalies. The teeth were washed in distilled water and their roots were separated with a water-cooled diamond saw. The crowns were embedded into cold-cure acrylic within stainless steel mounting rings. Each tooth embedded in acrylic was then removed from the steel rings and was allowed to fully cure for 24 hours.

The teeth were then polished with a non-fluoridated pumice (Whip Mix, Louisville, KY) and water slurry for 10 seconds followed by washing and drying prior to bonding. [21] The molars were then randomly divided into four groups of 45 molars. Each group was bonded with only one of the following primers according to the manufacturer's instructions: Transbond XT primer, Transbond Plus SEP, iBOND and G-Bond. There were four adhesive groups, which each had three debonding times (n=15).

Group 1: Transbond XT Primer + 35% etching gel (control): Each tooth was etched with 35% phosphoric acid etching gel for 15 seconds. The etching gel was then rinsed with water spray for 15 seconds and air dried. A frosty white enamel appearance was observed to indicate adequate etching of the enamel. With an applicator a single thin layer of Transbond XT primer was applied to the etched enamel surface.

Group 2: Transbond Plus SEP: To prepare the Transbond Plus SEP for application the contents within the black reservoir at the end was completely squeezed into the middle reservoir towards the applicator. The black reservoir was then folded over the top of the middle reservoir and the contents of the middle reservoir completely squeezed into the purple reservoir closest to the applicator stick. The applicator was then swirled around within the purple reservoir for 5 seconds to evenly apply the mixture onto the applicator tip. The applicator was then rubbed with light pressure on the enamel of each tooth for 5 seconds. Finally, a gentle air burst was applied over the primer for 1-2 seconds.

Group 3: iBOND Self Etch: Prior to dispensing the iBOND the bottle was vigorously shaken. A drop of iBOND was then dispensed into the mixing well. An applicator brush was then dipped into the iBOND solution and then rubbed on the enamel for 20 seconds per tooth. Gentle air drying was performed until a glossy enamel surface was observed. The tooth was then light cured for 20 seconds using a LED light curing unit (American Orthodontics - Sheboygan, WI).

Group 3: G-bond: A drop of G-bond was dispensed into the mixing well and with an applicator a single coat of G-Bond was applied to the tooth surface. After waiting for 5 seconds, a gentle air burst was applied to thin the primer. The tooth was then light cured for 10 seconds using a LED light curing unit (American Orthodontics - Sheboygan, WI).

Following the primer application, the teeth were bonded with a single stainless steel button using Transbond XT adhesive composite. A uniform amount of Transbond XT adhesive composite was applied to the button base and then gently placed on the enamel surface. To ensure uniform and complete seating of the button to the tooth a 500g vertical loading apparatus was used. After the placement of the button and removal of excess resin, each tooth was then light cured for 20 seconds each using a LED light-curing unit (American Orthodontics - Sheboygan, WI) placed as close as possible to the button without contacting it in a direction directly above and perpendicular the button and enamel surface.

For the evaluation of immediate bond strength, brackets were debonded approximately 5 minutes after bonding of the button to the tooth. For the 24 hours and 3 month groups, the teeth were stored in de-ionized water in a 37°C incubator in a covered glass jar. To test the bond strength a universal testing machine (Zwick, Germany) was used. Each sample was mounted and secured into the Bencor Multi-T loading apparatus [22] with the knife-edged shearing blade directed in an occluso-gingival direction at the button base using a crosshead speed of 0.5mm/minute on a 1kN load cell. The buttons were loaded until they debonded and the stress in megapascals (MPa) was recorded.

After debonding, each tooth was evaluated under a light microscope (10x magnification) in order to determine the amount of adhesive left on the tooth surface. Each tooth was evaluated using a modified Adhesive Remnant Index (ARI) score as described by Bishara *et al* (1999).[23,24] Score 1 = 100% left on tooth + bracket impression; Score 2 = >90% left on tooth; Score 3 = 10-90% left on tooth; Score 4 = <10% left on tooth; Score 5 = 0% left on tooth.

Descriptive analysis as well as One-way analysis of variance (ANOVA) was performed to determine whether there was a significant difference among the test groups over time as well as within each test group at each specific time point. Levene's test of homogeneity of variances indicated that data set was well distributed. If significant differences were present, a Bonferroni post-hoc multiple comparisons test was performed to identify which means were significantly different from each other. Chi-square test was used to determine the significant differences in the ARI scores among the different groups. The significance used for all the tests was predetermined at a probability value of 0.05 or less.

Results

The descriptive statistics for each of the bonding agents tested can be found in Table I. The ANOVA test showed that there was a statistically significant difference ($p<0.001$) between the mean SBS between the four groups tested at the immediate debond stage (Figure 1). Transbond XT primer (11.22±1.98 MPa) had a significantly higher mean SBS when compared to the Transbond Plus SEP (5.32±1.81 MPa; $p<0.001$), iBOND (6.69±1.78 MPa; $p<0.001$) and G-Bond (8.30±2.42 MPa; $p<0.01$). The tests revealed that G-Bond had a significantly higher SBS compared to the Transbond Plus SEP ($p<0.01$). Comparison of the SBS of G-Bond to iBOND yielded no significant difference. Transbond Plus SEP was also the only bonding agent that exhibited a low mean SBS of 5.32±1.81 MPa.

After 24 hours (Figure 1), the ANOVA test revealed that there was a significant difference among the four groups tested ($p<0.05$). Specifically, there was only a significant difference ($p<0.005$) between Transbond XT primer (16.65±6.04 MPa) and iBOND (9.32±6.43 MPa). After 3 months (Figure 1), the ANOVA test found that there were no significant differences among the four groups ($p>0.05$).

The mean SBS of Transbond XT primer changed significantly from immediate (11.22±1.98 MPa) to 24 hours (16.65±6.04 MPa; $p<0.05$) and from immediate to 3 months (15.31±4.17 MPa; $p<0.05$). However, there was no difference in SBS from 24 hours to 3 months. It can also be seen that the bond strength of Transbond XT primer first increased significantly from immediate to 24 hours, and then slightly decreased after 3 months (Figure 2).

The mean SBS of Transbond Plus SEP changed significantly from immediate (5.32±1.81 MPa), to 24 hours (13.20±6.43 MPa; $p<0.001$) and to 3 months (13.62±4.58 MPa; $p<0.001$). However, from 24 hours to 3 months there was no difference in bond strength. Although not statistically significant, the mean SBS of iBOND gradually increased over time from immediate (6.69±1.78 MPa) to 24 hours (9.32±3.18 MPa; $p>0.05$) to 3 months (11.85±5.02 MPa; $p>0.05$). The mean SBS of G-Bond increased from immediate (8.30±2.42 MPa) to 24 hours (13.18±4.35 MPa; $p>0.05$), and then decreased slightly after 3 months (12.24±3.47 MPa; $p>0.05$). However, the changes between the times were not statistically significant. The coefficient of variation ranged between 17% to 48%, with the majority in the low to mid 30% range. Transbond XT primer (17%) tested immediately had the most consistent values and Transbond Plus SEP tested at 24 hours had the least consistent range of values (48.7%).

The Chi-squared analysis revealed that there was a statistically significant difference in the ARI scores among the four adhesive groups tested at each time point. At the immediate debond stage, G-Bond had the mostly low ARI scores and Transbond Plus SEP had the mostly high ARI scores. After 24 hours, Transbond XT primer had mostly low ARI scores, and both Transbond Plus SEP and iBOND had the mostly high scores. After 3 months, Transbond XT primer continued to have the mostly low ARI scores. (Table)

Discussion

This study evaluated the SBS of two new SEPs over a three months period. Immediate SBS was tested to evaluate whether the SEP would permit for immediate arch wire insertion. Bond strength evaluation over 24 hours and 3 months would provide more long term information on the new SEPs. Measuring the SBS over 3 months of time would provide valuable information on the ability of the bonding agent to withstand the forces of mastication and applied orthodontic forces during the initial alignment and levelling phase.

The mean immediate SBS for Transbond XT primer (11.22 MPa) was comparable to a study by Turk *et al* [26] which found Transbond XT primer to have a mean SBS of 9.50 MPa after 5 minutes. Both of the two new 7th generation bonding systems iBOND (6.69 MPa) and G-Bond (8.30 MPa) had mean SBS above the minimum 6-8 MPa as recommended by Reynolds [19]. Reynolds [19] first recommended a minimum SBS more than 30 years ago, however more recently, Wiltshire & Noble [20] recommended a minimum SBS of 3-4 MPa based on *in vitro* and clinical studies on glass ionomers used in bonding orthodontic brackets. Based on our results, Transbond XT primer, iBOND and G-Bond may potentially have adequate bond strength to allow for immediate archwire insertion. Interestingly, Transbond Plus SEP (5.32 MPa), a SEP marketed for orthodontic bonding, produced lower SBS's at the immediate debond time period. Similarly Bishara *et al* [27,28] found a mean bond strength of 5.9 MPa for Transbond Plus SEP. However, according to Turk *et al* [26] the SBS of Transbond Plus SEP 5 minutes after bonding was reported as 8.97 MPa, approximately 4 MPa higher than the current study. Accordingly, Transbond Plus SEP may be sufficiently strong to withstand immediate archwire insertion, especially if the initial forces of the archwire are kept light. [19,20]

After 24 hours of incubation, Transbond XT primer (16.65 MPa) had a mean SBS significantly higher than iBOND (9.32 MPa; $p<0.005$). However, the SBS for iBOND may still be considered adequate for orthodontic bonding. [19,20] From immediate to 24 hours (Figure 2), the SBS for both Transbond XT primer and Transbond Plus SEP increased significantly. This could suggest that for Transbond Plus SEP it may be prudent to tie-in very light arch wires or to wait 24 hours prior to arch wire tie-in to allow its bond strength to improve. However, waiting 24 hours prior may be challenging from a clinical standpoint. Our result is similar to Turk *et al* [26], where a significant increase ($p<0.001$) from immediate (5 minutes) to 24 hours was observed for both Transbond XT primer (5min: 9.50 MPa; 24h: 16.82 MPa) and Transbond Plus SEP (5min: 8.97 MPa; 24h: 19.11 MPa).

A possible reason for an increase in bond strength over 24 hours is that only about 75% of the polymerization takes place during the first 10 minutes and that the curing reaction continues under the bracket base for a period of 24 hours. After 24 hours most of the polymerization should have been completed with little change in bond strength. [29,4,26] A study by Oesterle *et al* [30] also supports our 24 hours results. Their results found that the maximum SBS occurred after 24 hours (20.99 MPa) and began to decrease over the next 24 months.

A study by Trites *et al* [31] showed similar results in which both Transbond XT primer and Transbond Plus SEP ad no significant change in shear bond strength between 24 hours and 3 months. Similarly, there was also no significant difference between Transbond XT primer and Transbond Plus SEP at the 3 month debond time ($p>0.05$).

Previous studies on iBOND are limited in the orthodontic literature. The mean SBS in the current study for iBOND (9.32 MPa) was similar to Holzmeier *et al* [17] (8.1 MPa) after 24 hours incubation. Paschos *et al* [18] reported a bond strength of 11.3 MPa after 30 days of incubation which is similar to the current study's SBS of iBOND (11.85 MPa) after 3 months. Both of these studies concluded that iBOND may have adequate bond strength to be used to bond orthodontic brackets. A study by Wiltshire & Karaiskos [32] with similar experimental conditions (same universal testing machine and cross-head speed of 0.5mm/min) indicated a higher mean SBS of 16.8 MPa compared to the present study of 9.32 MPa. Some possible reasons for these differences may be due to operator technique, load cell configuration, type of teeth, light curing unit and/or material batch number.

At the time of the present study there were no previously published studies on the use of G-Bond to bond orthodontic brackets to enamel. The only studies found were from a restorative perspective looking at the SBS of composite resin to enamel and dentin. The restorative studies by Burrow *et al* [33] and Söderholm *et al* [34] showed that G-Bond had mean SBSs of 27.1 MPa and 14.9 MPa to enamel, respectively. Because these studies investigated the SBS of composite resin rather than metal brackets or buttons, a direct comparison would be difficult to make and remains speculative at best.

The main purpose of our study was to determine whether these two new SEPs iBOND and G-Bond would have the utility to be used as bonding agents in orthodontics. From their mean SBS at each time point over the three month period, they produced SBS values which were all above the recommended minimal SBS [19,20] to potentially withstand the masticatory and orthodontic forces immediately during archwire insertion and tie-in and throughout the initial 3 month alignment phase.

The Chi-square analysis at the three different time points revealed statistically significant differences in ARI scores among the four groups. At the immediate debond stage, Transbond Plus SEP had high ARI scores (100%) indicating most of the composite was removed with the bracket, however, this was in contrast to previous studies by Bishara *et al* [35,28] who found Transbond Plus SEP to have high ARI scores ranging from only 5% to 35%.

After 24 hours, Transbond XT primer had mainly low ARI scores (47%). This result was similar to Scougall Vilchis *et al* [36] with 40% of the samples having low ARI scores, but in contrast with Trites *et al* [31] and Turk *et al* [26] with low ARI scores of only 3.3% and 10%, respectively. Both Transbond Plus SEP (67%) and iBOND (80%) had mainly high ARI scores after 24 hours indicating little to no composite resin left on the tooth. In contrast, other studies [31,26,36] found Transbond Plus SEP to have similar ARI scores ranging from 0% to 17.1%. The high ARI scores for iBOND (80%) was similar to that found by Holzmeier *et al* [17] and Minick *et al* [37] with 100% of their samples had <50% of the composite resin left on the tooth.

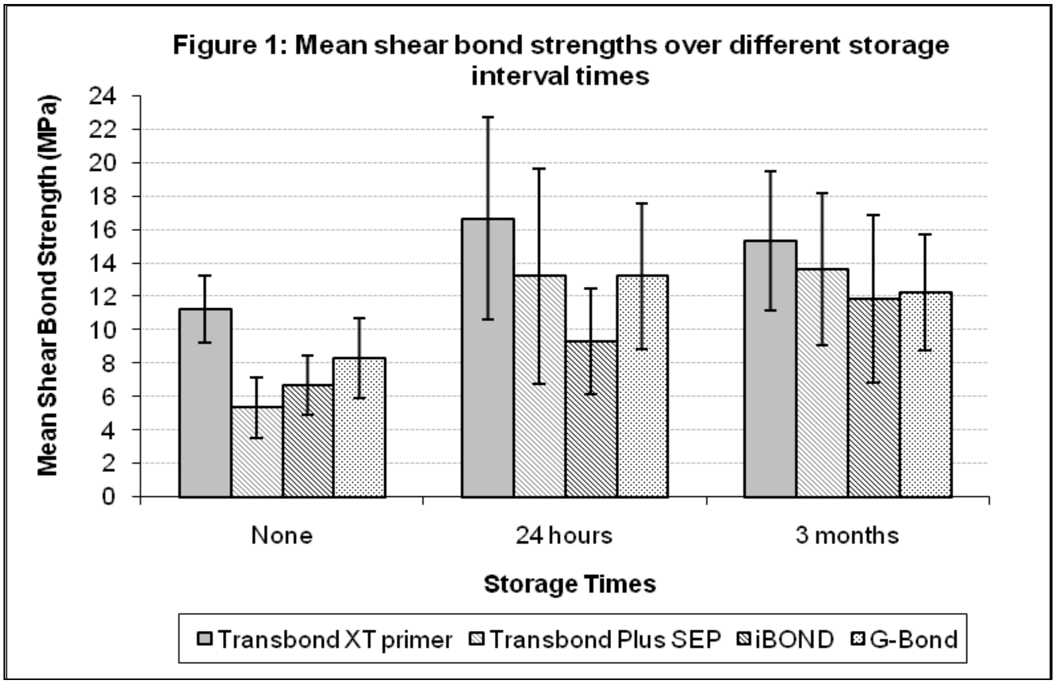
Trites *et al* [31] found that after 3 months Transbond XT primer had 83% of the samples to have <50% composite resin left on the tooth, which is in contrast to the current study where 73% of the samples had >90% composite resin left on the tooth after debonding.

Conclusions

Based on this *in vitro* study on the SBS of Transbond XT primer (control), Transbond Plus SEP and two new 7th generation SEP iBOND and G-Bond, we can conclude that:

1. Transbond XT primer, iBOND or G-Bond potentially produce adequate bond strength of orthodontic buttons to human enamel for successful immediate archwire tie-in.
2. 24 hours and 3 months after bonding with Transbond XT primer, Transbond Plus SEP, iBOND and G-Bond all maintain sufficient bond strength to potentially withstand occlusal and archwire forces.
3. Transbond XT primer, Transbond Plus SEP, iBOND and G-Bond all had an increase in bond strength after the first 24 hours.
4. The ARI score after 3 months, Transbond XT primer had the most adhesive remnants remaining on the tooth and Transbond Plus SEP, iBOND and G-Bond had the least adhesive remnants remaining on the tooth. This may indicate that Transbond XT primer has the least chance of enamel fracture, tears, crazing during debonding compared to the other tested bonding agents.

Tables & Figures



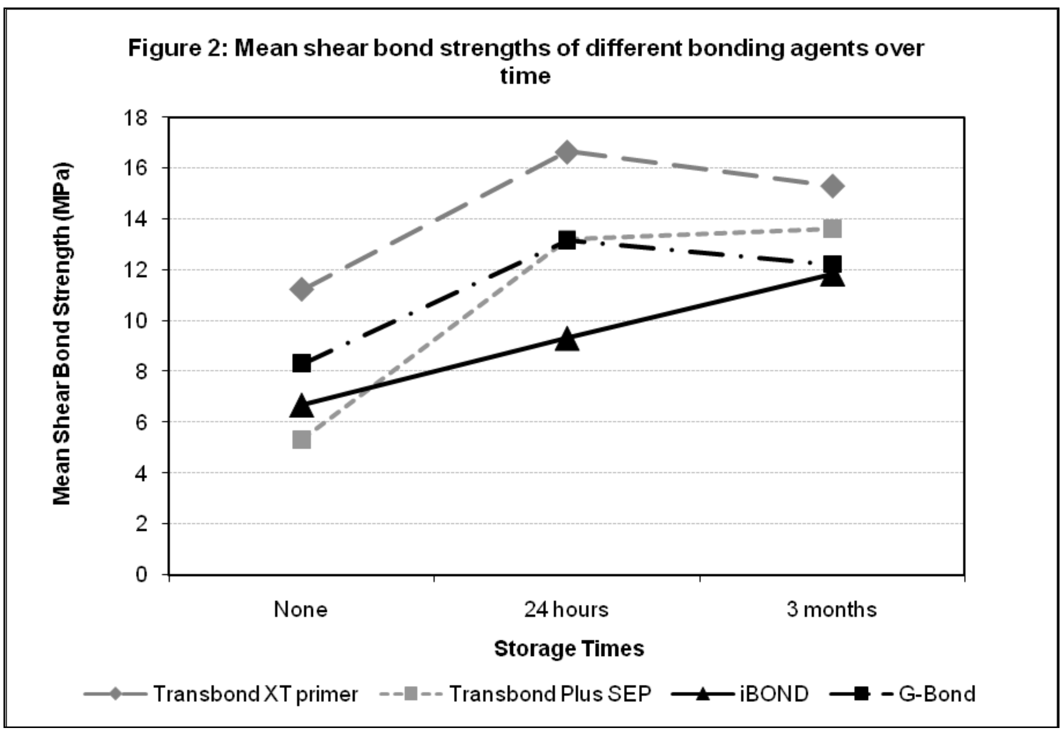


Table I: Descriptive statistics of the shear bond strengths (MPa) at immediate, 24 hours and 3 months

Groups	Time	Mean Shear bond strength (MPa)	SD (MPa)	Minimum (MPa)	Maximum (MPa)	Coefficient of Variation (%)
Transbond XT primer	Immediate	11.22	1.98	7.91	14.92	17.73
n=45	24h	16.65	6.04	2.63	26.87	36.28
n=15, each time groups	3mos	15.31	4.17	8.19	21.90	27.24
Transbond Plus SEP	Immediate	5.32	1.81	2.78	8.83	34.00
n=45	24h	13.20	6.43	5.48	22.33	48.70
n=15, each time groups	3mos	13.62	4.58	7.38	21.66	33.63
iBOND	Immediate	6.69	1.78	2.84	9.64	26.59
n=45	24h	9.32	3.18	4.85	16.55	34.11
n=15, each time groups	3mos	11.85	5.02	5.32	26.08	42.45
G-Bond	Immediate	8.30	2.42	5.17	13.35	29.14
n=45	24h	13.18	4.35	7.18	25.18	33.00
n=15, each time groups	3mos	12.24	3.46	4.25	16.91	42.45

Table II: Frequency of ARI Scores at all storage intervals

		ARI Score					
Storage Interval	**Bonding Agent**	**1**	**2**	**3**	**4**	**5**	**Total**
Immediate	Transbond XT primer	1	4	5	5	0	15
	Transbond Plus SEP	0	0	0	7	8	15
	iBOND	8	1	5	1	0	15
	G-Bond	9	6	0	0	0	15
24 hours	Transbond XT primer	7	0	4	3	1	15
	Transbond Plus SEP	4	0	1	5	5	15
	iBOND	1	0	2	4	8	15
	G-Bond	4	0	4	3	4	15
3 months	Transbond XT primer	8	3	2	2	0	15
	Transbond Plus SEP	1	1	2	5	6	15
	iBOND	2	0	3	3	7	15
	G-Bond	2	0	1	5	7	15

Chi-square χ^2: Immediate $p<0.001$; 24 hours $p<0.05$; 3 months $p<0.001$. ARI: Score 1 = 100% left on tooth + bracket impression; Score 2 = >90% left on tooth; Score 3 = 10-90% left on tooth; Score 4 = <10% left on tooth; Score 5 = 0% left on tooth

Reference List

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955;34:849-853.

2. Newman GV. Epoxy adhesives for orthodontic attachments: progress report. Am J Orthod 1965;51:901-912.

3. Farah J, Powers J. The Dental Advisor: Self-etching bonding agents. J Can Dent Assoc 2004;70:446-447.

4. Powers JM, Sakaguchi RL, Craig RGRdm. Craig's restorative dental materials. St. Louis, Mo. ; [London]: Mosby Elsevier; 2006.

5. Bishara SE, VonWald L, Laffoon JF, Warren JJ. Effect of a self-etch primer/adhesive on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2001;119:621-624.

6. Arnold RW, Combe EC, Warford JH, Jr. Bonding of stainless steel brackets to enamel with a new self-etching primer. Am J Orthod Dentofacial Orthop 2002;122:274-276.

7. Asgari S, Salas A, English J, Powers J. Clinical evaluation of bond failure rates with a new self-etching primer. J Clin Orthod 2002;36:687-689.

8. Aljubouri YD, Millett DT, Gilmour WH. Six and 12 months' evaluation of a self-etching primer versus two-stage etch and prime for orthodontic bonding: a randomized clinical trial. Eur J Orthod 2004;26:565-571.

9. dos Santos JE, Quioca J, Loguercio AD, Reis A. Six-month bracket survival with a self-etch adhesive. Angle Orthod 2006;76:863-868.

10. Manning N, Chadwick SM, Plunkett D, Macfarlane TV. A randomized clinical trial comparing 'one-step' and 'two-step' orthodontic bonding systems. J Orthod 2006;33:276-283; discussion 256-277.

11. Pandis N, Polychronopoulou A, Eliades T. Failure rate of self-ligating and edgewise brackets bonded with conventional acid etching and a self-etching primer: a prospective in vivo study. Angle Orthod 2006;76:119-122.

12. Banks P, Thiruvenkatachari B. Long-term clinical evaluation of bracket failure with a self-etching primer: a randomized controlled trial. J Orthod 2007;34:243-251.

13. Khalha A. No difference in the failure rates of orthodontic brackets bonded with self-etching primer. Evid Based Dent 2008;9:79-80.

14. Reis A, dos Santos JE, Loguercio AD, de Oliveira Bauer JR. Eighteen-month bracket survival rate: conventional versus self-etch adhesive. Eur J Orthod 2008;30:94-99.

15. Cal-Neto JP, Quintao CA, Almeida MA, Miguel JA. Bond failure rates with a self-etching primer: a randomized controlled trial. Am J Orthod Dentofacial Orthop 2009;135:782-786.

16. Fritz UB, Diedrich P, Finger WJ. Self-etching primers--an alternative to the conventional acid etch technique? J Orofac Orthop 2001;62:238-245.

17. Holzmeier M, Schaubmayr M, Dasch W, Hirschfelder U. A new generation of self-etching adhesives: comparison with traditional acid etch technique. J Orofac Orthop 2008;69:78-93.

18. Paschos E, Westphal JO, Ilie N, Huth KC, Hickel R, Rudzki-Janson I. Artificial saliva contamination effects on bond strength of self-etching primers. Angle Orthod 2008;78:716-721.

19. Reynolds I. A review of direct orthodontic bonding. Br J Orthod 1975;2:171-178.

20. Wiltshire WA, Noble J. Clinical and Laboratory Perspectives of Improved Orthodontic Bonding to Normal, Hypoplastic, and Fluorosed Enamel. Semin Orthod 2010;16:55-65.

21. Nemeth BR, Wiltshire WA, Lavelle CL. Shear/peel bond strength of orthodontic attachments to moist and dry enamel. Am J Orthod Dentofacial Orthop 2006;129:396-401.

22. Driessen C, Coetzee W, Biljon Bv. Results of the Bencor multi T system for testing shear and tensile bond strength. J Dent Res 1989;68:726.

23. Årtun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85:333-340.

24. Bishara SE, VonWald L, Olsen ME, Laffoon JF. Effect of time on the shear bond strength of glass ionomer and composite orthodontic adhesives. Am J Orthod Dentofacial Orthop 1999;116:616-620.

25. Powers JM, Kim HB, Turner DS. Orthodontic adhesives and bond strength testing. Semin Orthod 1997;3:147-156.

26. Turk T, Elekdag-Turk S, Isci D. Effects of self-etching primer on shear bond strength of orthodontic brackets at different debond times. Angle Orthod 2007;77:108-112.

27. Bishara SE, Oonsombat C, Ajlouni R, Laffoon JF. Comparison of the shear bond strength of 2 self-etch primer/adhesive systems. Am J Orthod Dentofacial Orthop 2004;125:348-350.

28. Bishara SE, Otsby AW, Ajlouni R, Laffoon J, Warren JJ. A new premixed self-etch adhesive for bonding orthodontic brackets. Angle Orthod 2008;78:1101-1104.

29. Klocke A, Shi J, Vaziri F, Kahl-Nieke B, Bismayer U. Effect of time on bond strength in indirect bonding. Angle Orthod 2004;74:245-250.

30. Oesterle LJ, Shellhart WC. Effect of aging on the shear bond strength of orthodontic brackets. Am J Orthod Dentofacial Orthop 2008;133:716-720.

31. Trites B, Foley TF, Banting D. Bond strength comparison of 2 self-etching primers over a 3-month storage period. Am J Orthod Dentofacial Orthop 2004;126:709-716.

32. Wiltshire W, Karaiskos N. Comparison of shear bond strengths of three self-etching primers. World J Orthod 2005;6 (suppl):abstracts SL060.

33. Burrow MF, Kitasako Y, Thomas CD, Tagami J. Comparison of enamel and dentin microshear bond strengths of a two-step self-etching priming system with five all-in-one systems. Oper Dent 2008;33:456-460.

34. Söderholm KJ, Soares F, Argumosa M, Loveland C, Bimstein E, Guelmann M. Shear bond strength of one etch-and-rinse and five self-etching dental adhesives when used by six operators. Acta Odontol Scand 2008;66:243-249.

35. Bishara SE, Ostby AW, Laffoon J, Warren JJ. A self-conditioner for resin-modified glass ionomers in bonding orthodontic brackets. Angle Orthod 2007;77:711-715.

36. Scougall Vilchis RJ, Yamamoto S, Kitai N, Yamamoto K. Shear bond strength of orthodontic brackets bonded with different self-etching adhesives. Am J Orthod Dentofacial Orthop 2009;136:425-430.

37. Minick GT, Oesterle LJ, Newman SM, Shellhart WC. Bracket bond strengths of new adhesive systems. Am J Orthod Dentofacial Orthop 2009;135:771-776.