

**Shear bond strength of rebonded orthodontic
attachments using self-etching primers**

By

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Abstract

Title: Shear bond strength of rebonded orthodontic attachments using self-etching primers

Objective: The purpose of this study was to evaluate the difference in shear bond strength (SBS) of bonding and rebonding orthodontic attachments when using a conventional etching primer (CEP) compared to a self-etching primer (SEP), as well as to evaluate if re-etching is necessary prior to rebonding a bracket with either a CEP or a SEP.

Materials and Methods: Orthodontic metal buttons were bonded to extracted human third molars according to 4 protocols: In Group A, teeth were bonded with the CEP technique for the first and second bondings. In Group B, teeth were bonded with the CEP technique for the first bonding but re-etching was omitted for the second bonding. In Group C, teeth were bonded with the SEP technique for both bondings. In Group D, teeth were bonded with the SEP technique for the first bonding but re-etching was omitted for the second bonding.

Results: The shear bond strengths when the SEP was used for the first bonding were significantly lower ($P < 0.001$; 10.65 ± 4.33 MPa) than when the CEP was used (14.04 ± 3.51 MPa). The second bonding omitting re-etching but still using the primer and adhesive generated significantly lower bond strengths ($P < 0.0001$; 9.07 ± 4.53 MPa) than when re-etching was used (14.66 ± 3.70 MPa), but still above the “clinically acceptable” standard of 6-8 MPa for *in vitro* shear bond strength testing. The second bonding SBS omitting re-etching and not using any primer or adhesive was very low (2.61 ± 2.34 MPa) and would definitely negatively effect the outcome of orthodontic bonding clinically.

Conclusion: When rebonding a bracket, re-etching may be omitted but then a primer and adhesive must be applied to obtain a “clinically acceptable” shear bond strength.

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Dedication

I dedicate this thesis to my girlfriend, parents, brother and friends. Your love and constant support has helped me completing this thesis.

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1. INTRODUCTION

In 1964, Newman introduced a direct-bonding technique for bonding orthodontic brackets (Newman 1964). This practice, however, only became a routine clinical procedure in the 1980s (Proffit 2013). Today, the most used bonding system in orthodontics is commonly referred to as the total-etch technique, and it consists of three components combined in two products: a 35-37% phosphoric acid etchant in the first, and a combined primer and adhesive in the second (Katona and Long 2006). Currently, the newer generations of adhesives (sixth and seventh) are referred to as self-etching primers and the three components are combined in one product thus eliminating the need for use of a syringe distributing the etch and subsequent rinsing. One of the important advantages of SEPs is a shallower etch creating less damage to the enamel structure.

When referring to orthodontic bonding, bond strength should be high enough to resist the forces of accidental debonding during the course of orthodontic treatment, but low enough to prevent the risk of enamel damage should debonding occur (Ozcan, Finnema et al. 2008). Bonding failure of an orthodontic attachment is possibly one of the most common emergencies encountered in orthodontics. Failure of bonded brackets is undesirable and can be costly in terms of clinical time and materials, can prolong treatment time, and can be frustrating for both the patient and the clinician. Beckwith *et al.* found that after missed appointments, replacing brackets was the second most important factor affecting the treatment duration (Beckwith, Ackerman et al. 1999). Similarly, Shia mentioned that appliance breakage was on his top three list of factors contributing to lengthening treatment (Shia 1986). Skidmore also observed that if 2 or more brackets failed, the mean addition to treatment time was between 2.2-4.6 months

(Skidmore, Brook et al. 2006). This might be a reason why many investigators have conducted research on the bonding of orthodontic attachments.

The *in vitro* method is often regarded as the gold standard in testing different bonding agents before clinical trials. Several articles have been published following the introduction of new adhesive systems and prior to its use in a clinical setting (Arnold, Combe et al. 2002, Asgari, Salas et al. 2002, Ireland, Knight et al. 2003, Cal-Neto, Carvalho et al. 2006, Ho, Akyalcin et al. 2011, Yadala, Gaddam et al. 2015).

To date, many studies have compared the mean shear bond strength of orthodontic brackets between the conventional etch and priming and the self-etching primers. Others have compared the average shear bond strength of a second and even third bonding of the same tooth. Most of the time after a bonding failure, new orthodontic attachments are rebonded using the same procedure as the first bonding. However, it must be noted that only one study has addressed the difference between the CEP and SEP on a second orthodontic bonding and whether re-etching was performed or not (Zhang, Yao et al. 2014).

2. LITERATURE REVIEW

2.1 Evolution of bonding

Since the beginnings of adhesive dentistry in 1955, and with the development of the acid etch technique (Buonocore 1955), dental bonding agents have evolved tremendously. In the mid 1950s, it was reported that bonding to acid-etched dentin surfaces could be achieved with the use of a resin containing glycerophosphoric acid dimethacrylate (GPDM) (Brudevold, Buonocore et al. 1956). Unfortunately, the technique was severely affected by immersion in water.

The first generation of dental adhesives to the tooth was hypothesized as being accomplished through the ability of the co-monomer N-phenylglycine glycidyl methacrylate (NPG-GMA) to chelate to the calcium in the smear layer. The bond strength was low, ranging from 2 to 3 MPa, therefore displaying poor clinical performance without mechanical retention in the tooth preparation (Heymann, Swift et al. 2013). The second generation appeared along with the chemical 2-Hydroxyethyl methacrylate (HEMA) in the primer in the late 1970's. The mechanism with this bonding agent was through ionic bonds between the negatively charged phosphate groups in the resin and the positively charged calcium in the smear layer of the tooth (Retief and Denys 1989, Kugel and Ferrari 2000). Microleakage was also an important issue. In 1979, Fusayama initiated the concept of acid-etching prior to the application of phosphate ester bonding agents (Fusayama, Nakamura et al. 1979). Five years later, the third generation emerged with the use of an acid etchant, with the introduction of Clearfil New Bond (Kurakay Co, Osaka, Japan). The two components, a hydrophilic primer and an unfilled resin adhesive, were dispensed separately (Kugel and Ferrari 2000). The fourth-generation, also known as the total

etch technique, emerged in the early 1990's with the introduction of the concept of "wet bonding" leaving the tooth moist so that the hydrophilic monomer found in the primer could properly penetrate the collagen fibres in the dentin, leading to what is known as the hybrid layer. The bond strength of this generation could reach up to 27 MPa (Freedman and Leinfelder 2002). In the mid 1990's, the primer and adhesive were incorporated in the same bottle in the fifth generation, simplifying the technique and considerably reducing post-operative sensitivity.

The sixth and seventh generations introduced the concept of self-etch primers (or SEP) in the late 1990's and early 2000's (Kugel and Ferrari 2000). The first sixth-generation adhesive released on the dental market was Prompt L-Pop (Turk, Elekdag-Turk et al. 2007). No separate step for etching was required. The sixth generation required mixing of two components prior to application on the tooth, and the seventh-generation was a one step, one component system. Bond strengths ranging from 18 to 25 MPa were attained with the seventh generation (Freedman and Leinfelder 2002). The newest generation of adhesive, the eighth-generation or the universal adhesive system, incorporates the bonding agent within the flowable resin composite. It is considered a self-etching, self-adhering flowable composite and virtually eliminates the need of a separate bonding application. Dentin bond strengths ranging from 38 to 47 MPa were reached with products like Single Bond (3M ESPE) and Adper Single Bond 2 Adhesive (3M ESPE) (Chandra 2008). Research evaluating the bond strength of the 8th generation adhesive with orthodontic attachments has non-concordant conclusions with products like Vertise Flow Self-adhesive (Kerr) and Maxcem Elite, Self-etch, self-adhesive (Kerr) (Bishara, Ajlouni et al. 2006, Isman, Karaarslan et al. 2012, Bernas, Wiltshire et al. 2013, Goracci, Margvelashvili et al. 2013).

Table 2.1 Evolution of bonding from first to eight-generation

Generation	Characteristics	Bond strength	Components	Brand names
1st	Very weak bond to dentin	2 MPa	1	Cervident (SS White Co), Cosmic Bond (Amalgamated Dental)
2nd	Weak adhesives, requires retentive preparations, prone to water degradation	2-8 MPa	2	Bondlite (Kerr), Scotchbond (3M)
3rd	Two-component primer and adhesive system, reduced sensitivity	8-15 MPa	2-3	Scotchbond II (3M), Prisma Universal Bond (Johnson and Johnson)
4th	Hybridization, total-etch	17-25 MPa	2-5	All Bond II (Bisco)
5th	Single component, moist bonding, no mixing, little sensitivity	20-24 MPa	1	Single Bond (3M), One-Step (Bisco)
6th	Multicomponent, self-etching, self-priming, very little sensitivity	18-23 MPa	2-3	Transbond Plus SEP (3M), Clearfil SE Bond (Kuraray)
7th	Single component, desensitizing, self-etching, self-priming, no mixing, bonds to metal, very little/no sensitivity	18-25 MPa	1	iBOND (Heraeus Kulzer), G-Bond (GC Corporation)
8th	Single component, self-etching, self-adhering flowable composite		1	Vertise Flow (Kerr), Maxcem Elite (Kerr)

Inspired from (Freedman and Leinfelder 2002)

2.2 Enamel structure

Tooth enamel is the hardest tissue of the human body. It has a highly mineralized crystalline structure, which gives it a rigid structure that is both strong and brittle. Its main component is hydroxyapatite which constitutes for 90-92% by volume and the other constituents include organic matrix proteins (1-2%) and water (4-12%) (Heymann, Swift et al. 2013). The average values for calcium and phosphorus are 37.5% and 17.5% respectively in a Ca:P ratio of 2.1/1 (Robinson, Weatherell et al. 1971). The largest structural constituent is the enamel rod, or enamel prism, which is surrounded by a rod sheath, composed of an organically rich interspace and a cementing inter-rod substance in specific areas (Heymann, Swift et al. 2013). The enamel rods have a diameter of about 4 μm near the dentino-enamel junction and 8 μm on the outer enamel surface (Heymann, Swift et al. 2013). The enamel permeability decreases with time because of reduction in the enamel matrix. Enamel is soluble in an acid environment and its solubility is variable and dependant on its fluoride content.

2.3 Bonding mechanism to enamel

Successful bonding in orthodontics involves attention to the preparation of the tooth surface, the surface of attachments and the bonding material joining the two (Proffit 2013). The tooth surface is typically cleaned with a non-fluorated pumice to remove the acquired enamel pellicle prior to etch application. Acid-etching changes the normally smooth enamel into a roughened surface through dissolution of the interprismatic substance in enamel. (Nanjannawar and Nanjannawar 2012). A fluid resin primer-adhesive penetrates within the porous enamel surface and adhesion results through the formation of micro-mechanical interlocking or tags within the tooth surface (Heymann, Swift et al. 2013).

2.4 Enamel etching

The role of etching is the dissolution of the hydroxyapatite in the superficial layer of the enamel and the creation of irregularities providing interlocked tags or micro-mechanical retention with the primer-adhesive (Brantley and Eliades 2000). The depth of etching usually varies between 5 and 50 μm^2 (Nanjannawar and Nanjannawar 2012). Five different etch patterns can be simultaneously observed on the enamel surface under scanning electron microscopy (Silverstone, Saxton et al. 1975).

Table 2.2 Enamel etching patterns

Types	
I	Dissolution of prism cores Honey comb appearance
II	Dissolution of interprismatic areas Cobblestone appearance
III	Mix of type II and III
IV	Pitted enamel Unfinished puzzles/maps/networks appearance
V	Flat smooth surface

Inspired from (Silverstone, Saxton et al. 1975)

The pattern most frequently observed for the conventional etching is the Type II with 53.3% as opposed to the Type IV, which is the usual etch pattern for the self-etching primers (Nanjannawar and Nanjannawar 2012).

2.5 Conventional bonding to enamel

The fifth generation adhesive systems (e.g. Transbond XT Light Cure Adhesive Primer) have been reported to be the gold standard for enamel bonding of orthodontic attachments (Turgut, Attar et al. 2011). The first product is an etchant (i.e. conditioner), usually a phosphoric acid (H_3PO_4) solution or gel at a concentration of 37%, which is used to produce a reliable etching pattern (Craig, Powers et al. 2006). The second product has 2 components; a primer and an adhesive. Primers are hydrophilic monomers carried in a solvent (e.g. acetone, ethanol/water, water or solvent-free) while adhesives are hydrophobic dimethacrylate oligomers (e.g. Bis-GMA) usually diluted with a lower molecular weight monomer (e.g. Triethylene glycol dimethacrylate – TEGDMA) (Craig, Powers et al. 2006).

Conventional bonding systems are usually used as a control when evaluating the bond strengths of alternative adhesive methods such as self-etch primers or 8th generation self-adhering flowable resin composite (Bishara, Oonsombat et al. 2004, Bishara, Ajlouni et al. 2006, Turk, Elekdag-Turk et al. 2007, Scougall Vilchis, Yamamoto et al. 2009, Scougall-Vilchis, Ohashi et al. 2009, Ho, Akyalcin et al. 2011).

2.6 Self-etch primers bonding to enamel

The self-etch primers or (SEPs) include the sixth-, seventh- and eighth-generation of adhesives. Both of these generations of products don't require the use of a separate acid etchant. The difference between the two generations is that the sixth-generation necessitates the mixing of the primer and the adhesive prior application on the tooth, but the seventh-generation does not require mixing of the primer and adhesive. These seventh-generation are sometimes referred to

as no-mix or premixed self-etching primers (Bishara, Oonsombat et al. 2004, Bishara, Otsby et al. 2008), and are often described as an “all-in-one” products (Farah 2005). The principal component in a SEP is methacrylated phosphoric acid ester, which comprises both the acidic constituent for etching and a monomer constituent for priming (Bishara, VonWald et al. 2001, Grubisa, Heo et al. 2004). Prompt L-Pop was the first sixth generation adhesive to be released on the market (Miller 2001). As of 2002, 20% of orthodontists were routinely using self-etch primers in the United States (Keim, Gottlieb et al. 2002).

Some of the main advantages of the SEP products are adequate bond strengths, simpler technique, as well as diminished depth of enamel etching and reduced enamel loss (Cal-Neto, Miguel et al. 2006, Vilchis, Hotta et al. 2007, Paschos, Westphal et al. 2008, Turgut, Attar et al. 2011, Nanjannawar and Nanjannawar 2012). The single-step technique usually exhibits weaker bond strength when compared with traditional orthodontic adhesive systems but offers bond strengths adequate for clinical use in orthodontics (Katona and Long 2006, Ho, Akyalcin et al. 2011). The bond strength of self-etch primers was found to increase with time and there was a significant difference between SBS done under 60 minutes (13.13 ± 2.09 MPa) after bonding and 24 hours (19.11 ± 3.40 MPa) (Turk, Elekdag-Turk et al. 2007).

The use of self-etch primers has been found to be less technique sensitive than conventional acid etching with mean bond strengths not significantly different when compared to the significantly different values obtained with phosphoric acid-etching technique using 3 orthodontists who were asked to bond teeth with the 2 methods (Grubisa, Heo et al. 2004). These 3 orthodontists concluded that it is probably due to the fewer steps in that technique.

2.6.1 Transbond Plus Self Etching Primer

Transbond Plus Self Etching Primer is a sixth-generation light-cured bonding agent and its principal indication is for orthodontic bracket bonding. The two compartments requiring mixing contain 2-Hydroxyethyl methacrylate (HEMA), polyalkenoic acid, water and stabilizers in one and methacrylate phosphoric acid esters, bisphenol A-glycidyl methacrylate (Bis-GMA), photo-initiators and stabilizers in the other (Holzmeier, Schaubmayr et al. 2008). The acidic component for etching reaches a pH of approximately 1.0 to demineralize the enamel before being neutralized by the stabilizer agents (Arnold, Combe et al. 2002).

2.6.2 Transbond Plus Self Etching Primer *in vitro* testing

Transbond Plus Self Etching Primer performs well in laboratory experiments when compared to a conventional etching primer with Transbond XT Primer. One research project established significantly higher SBS for TB+SEP with 16.0 ± 4.5 compared to 11.5 ± 3.3 MPa (Buyukyilmaz, Usumez et al. 2003). Two experiments discovered lower bond strengths for the self-etch primer, of which one was immediate debonding (Grubisa, Heo et al. 2004, Ho, Akyalcin et al. 2011). The remaining *in vitro* tests exhibited no significant differences between the two bonding methods (Arnold, Combe et al. 2002, Cal-Neto, Miguel et al. 2006, Vicente, Bravo et al. 2006, Turk, Elekdag-Turk et al. 2007, Scougall Vilchis, Yamamoto et al. 2009, Scougall-Vilchis, Ohashi et al. 2009, Ho, Akyalcin et al. 2011).

Table 2.3 *In vitro* - Transbond Plus Self Etching Primer (TB+SEP) compared to Conventional etching primer (CEP)

Study	Time of SBS test after bonding	Cross head speed, Load cell	N	Average SBS (MPa)		Conclusion
				TB+SEP	CEP	
Arnold, 2002 AJODO	24h	1mm/min, N.R.	12	8.0±1.3	9.7±3.1	No significant difference
Buyukyilmaz, 2003 AO	24h	0.5mm/min	20	16.0±4.5	11.5±3.3	Significantly higher
Grubisa, 2004 AJODO	N.R. Thermocycling	2.5mm/min 0.5kN	66(S) 70(C)	7.5±4.2	9.8±4.2	Significantly lower
Cal-Neto, 2006 AO	> 30 days	0.5mm/min N.R.	23	10.89±2.60	11.35±2.36	No significant difference
Vicente, 2006 EJO	24h	1mm/min 1kN	25	12.2±4.27	12.27±5.01	No significant difference
Turk, 2007 AO	5 min	1mm/min N.R.	10	8.97±2.05	9.50±1.52	No significant difference
	15 min		10	10.61±1.34	10.75±2.26	
	30 min		10	10.15±1.97	11.24±2.16	
	60 min		10	13.13±2.09	12.32±2.26	
	24h		10	19.11±3.40	16.82±3.02	
Scougall-Vilchis, 2008 AJODO	24h	0.5mm/min	20	21.1±6.2	26.5±8.1	No significant difference
Scougall-Vilchis, 2009 AJODO	24h	0.5mm/min N.R.	35	16.6±7.3	19.0±6.7	No significant difference
Ho, 2011 JO	Immediate	0.5mm/min, 1kN	15	17.7±6.0 N	37.3±6.6 N	Significantly lower
	24h		15	43.9±21.4 N	55.4±20.1 N	No significant difference
	3 months		15	45.3±15.2 N	50.9±13.8 N	No significant difference

AJODO: American Journal of Orthodontics and Dentofacial Orthopedics

AO: Angle Orthodontist

EJO: European Journal of Orthodontics

JO: Journal of Orthodontics

Both Turk and Ho discovered that better shear bond strengths are obtained as the elapsed time before debond increases (Turk, Elekdag-Turk et al. 2007, Ho, Akyalcin et al. 2011). Cycling fatigue (Mansour, Drummond et al. 2011) and thermocycling (Elekdag-Turk, Turk et al. 2008) have been found to reduce significantly the SBS of Transbond Plus Self Etching Primer. Katona

evaluated TB+SEP and the traditional acid etching and reported that the self-etch primer performed inferiorly in shear-peel mode, superiorly in tension, and equally in torsion (Katona and Long 2006). Montasser determined that significantly higher bond strengths were obtained when a 300g sustained loading force was maintained throughout the 40 seconds of light curing with the same SEP (Montasser 2011). Although conventional etching primer and TB+SEP shear bond strengths were negatively influenced by saliva, many experiments demonstrated that TB+SEP performed superiorly in that condition and still exhibited clinically acceptable bond strengths (Rajagopal, Padmanabhan et al. 2004, Oztoprak, Isik et al. 2007, Prasad, Mohamed et al. 2014)

Transbond Plus Self Etching Primer is commonly used as a reference in SEP bonding of orthodontic attachments. The latter is frequently compared to new SEPs emerging on the market before accepting these in clinical practice (Buyukyilmaz, Usumez et al. 2003, Bishara, Otsby et al. 2008, Scougall-Vilchis, Ohashi et al. 2009, Ho, Akyalcin et al. 2011). Generally, TB+SEP performed better or equally to other self-etching primers (Buyukyilmaz, Usumez et al. 2003, Bishara, Oonsombat et al. 2004, Bishara, Otsby et al. 2008, Scougall Vilchis, Yamamoto et al. 2009, Scougall-Vilchis, Ohashi et al. 2009). However, TB+SEP obtained significantly lower bond strengths than G-Bond immediately after bonding (Ho, Akyalcin et al. 2011) and Clearfil SE Bond 48h after bonding (Arhun, Arman et al. 2006).

Table 2.4 *In vitro* - Transbond Plus Self Etching Primer (TB+SEP) compared to other self-etch primers

Study	Time of SBS test after bonding	Cross head speed, Load cell	N	Average SBS (MPa)		Conclusion
				TB+SEP	Other SEP	
Buyukyilmaz, 2003 AO	24h	0.5mm/min N.R.	20	16.0±4.5	11.5±3.3 Clearfil SE Bond	Significantly higher
					9.9±4.0 Etch&Prime 3.0	
Bishara, 2004 AJODO	30min	5.0mm/min N.R.	20	5.9±2.7	6.6±3.2 Ideal 1	No significant difference
Arhun, 2006 AJODO	48h	1.0mm/min	12	6.4±2.9	9.6±4.0 Adper Prompt L-Pop	No significant difference
					13.9±4.3 Clearfil SE Bond	Significantly Lower
Bishara, 2008 AO	30min	5.0mm/min N.R.	20	5.9±3.2	3.6±1.3 AdhesSE	Significantly Higher
Scougall-Vilchis, 2008 AJODO	24h	0.5mm/min N.R.	20	21.1±6.2	13.4±4.1 AdheSE	Significantly Higher
					8.8±2.6 Primers A and B	
					19.0±4.3 Clearfil Mega Bond FA	No significant difference
					19.6±5.1 Peak SE and Peak LC	
					18.3±4.4 Bond Force	
Scougall-Vilchis, 2009 AJODO	24h	0.5mm/min N.R.	35	16.6±7.3	11.0±3.9 Clearfil Mega Bond FA	Significantly Higher
					10.1±3.7 Primers A and B	
					11.8±3.5 AdheSE	
Ho, 2011 JO	Immediate	0.5mm\min 1kN	15	17.7±6.0 N	22.2±5.9N iBond	No significant difference
					27.6±8.0N G-Bond	Significantly lower
	24h		15	43.9±21.4 N	31.0±10.5N iBond	No significant difference
					43.8±14.4N G-Bond	
	3 months		15	45.3±15.2 N	39.4±16.7N iBond	No significant difference
					40.7±11.5N G-Bond	

AJODO: American Journal of Orthodontics and Dentofacial Orthopedics

AO: Angle Orthodontist

JO: Journal of Orthodontics

Transbond Plus Self Etching Primer has also been compared to eighth-generation bonding agents with different outcomes. Goracci has found that Vertise Flow yielded similar bond strengths to conventional etching primer (Transbond XT Primer) but exhibited superior SBS compared to TB+SEP (Goracci, Margvelashvili et al. 2013). Bishara concluded that Transbond Plus Self Etching Primer SBS was significantly higher compared to with Maxcem Elite (Bishara, Ajlouni et al. 2006). Both Goracci and Isman established that the two previously mentioned eighth-generation bonding agents demonstrated increased bond strength with prior acid etching (Isman, Karaarslan et al. 2012, Goracci, Margvelashvili et al. 2013)

2.6.3 Clinical trials - Transbond Plus Self Etching Primer

In vivo performances of bonding agents is determined by the bond failure rate, which is the number of brackets debonded over a period of time divided by the number of brackets bonded multiplied by one hundred percent (Powers, Kim et al. 1997).

$$\text{Bond failure rate} = \frac{\text{brackets debonded}}{\text{brackets bonded}} \times 100\%$$

Randomized controlled trials have compared the failure rates of Transbond Plus Self Etching Primer to conventional etching with most of these using the split-mouth method having one side bonded with the SEP and the other with the CEP. Three of the publications indicated that Transbond Plus Self Etching Primer had a higher clinical bond failure rate (Ireland, Knight et al. 2003, Murfitt, Quick et al. 2006, Elekdag-Turk, Cakmak et al. 2008) and only one showed a lower failure rate (dos Santos, Quioca et al. 2006). However, the majority of these studies concluded that Transbond Plus Self Etching Primer has no significant difference in clinical

failure rates as compared to conventional bonding systems (Aljubouri, Millett et al. 2004, Manning, Chadwick et al. 2006, Banks and Thiruvengkatachari 2007, Elekdag-Turk, Isci et al. 2008, Reis, dos Santos et al. 2008, Cal-Neto, Quintao et al. 2009, Dominguez, Tortamano et al. 2013). A common finding mentioned by most of these studies was that the one step bonding procedure was significantly less time consuming than the two step technique.

Table 2.5 Clinical trials - Transbond Plus Self Etching Primer (SEP) compared to Transbond XT Primer (CEP)

Study	Journal	Method	Observation period	Failure rate %		Failure rate of SEP vs. CEP
				SEP	CEP	
Ireland 2003	AJODO	Split-mouth RCT	6 months	10.99	4.95	Higher
Aljubouri, 2004	EJO	Split-mouth RCT	6 months	0.8	1.1	No difference
			12 months	1.6	3.1	
Manning, 2006	JO	Parallel-group RCT	6 months	1.7	2.0	No difference
			12 months	3.7	3.5	
			Treatment	7.0	7.4	
Dos Santos, 2006	AO	Split-mouth RCT	6 months	7.4	10.6	Lower
Murfitt, 2006	EJO	Split-mouth RCT	12 months	11.2	3.9	Higher
Banks, 2007	JO	Parallel-group RCT	Treatment	4.8	3.5	No difference
Elekdag-Turk, 2008	EJO	Split-mouth RCT	6 months	0.6	0.6	No difference
Elekdag-Turk, 2008	AO	Split-mouth RCT	12 months	4.7	1.7	Higher
Reis 2008	EJO	Split-mouth RCT	18 months	15.6	17.6	No difference
Cal-Neto, 2009	AJODO	Parallel-group RCT	12 months	6.88	4.78	No difference
Dominguez 2013	DPJO	Split-mouth	36-48 months	4.58	5.41	No difference

AJODO: American Journal of Orthodontics and Dentofacial Orthopedics

AO: Angle Orthodontist

DPJO: Dental Press Journal of Orthodontics

EJO: European Journal of Orthodontics

JO: Journal of Orthodontics

2.7 Bond strength testing in orthodontics

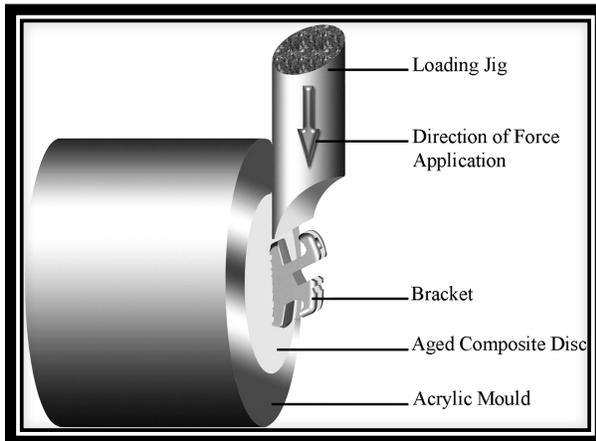
2.7.1 Bond strength testing

Although *in vitro* testing determines the accurate strength of a material, it cannot predict accurately clinical performances or substitute for *in vivo* experimentation (Finnema, Ozcan et al. 2010). Bond strength testing in orthodontics can be typically performed in shear, shear-peel, tension or torsion (Ostertag, Dhuru et al. 1991, Eliades and Brantley 2000). Both shear and tensile *in vitro* bond experiments are valid tests for studying bond strengths of orthodontic adhesives (Brantley and Eliades 2000). Some of the advantages of *in vitro* testing are the speed at which one can collect data, the simplicity of the methodology, the option to measure a specific variable while keeping the other parameters constant, and the ability to compare a new product with previously tested ones at a relative low cost of the materials and methods (Van Meerbeek, Peumans et al. 2010). Orthodontic materials that perform well in *in vitro* studies should always be evaluated with *in-vivo* randomized clinical trials (Finnema, Ozcan et al. 2010).

2.7.1.1 Shear bond strength

Shear bond strength testing is the most frequently used experiment for de-bonding brackets (Finnema, Ozcan et al. 2010). In shear bond testing, the bracket is loaded with a blade under stress of a load cell (kN) at a given speed measured in millimeters per minute (mm/min). The movement is achieved so that the shearing blade slides parallel (at 0°) to the enamel surface of the tooth. Clinically and even within a controlled environment of *in vitro* testing, pure shear loading is difficult to achieve, and most shear testing also includes components of peel, tension, and torsion. Due to the unavoidable element of bending during the test phase, it is impossible to apply a pure shear load to an attachment (Katona 1994).

Figure 2.1 Shear bond strength testing



(Bayram, Yesilyurt et al. 2011)

2.7.1.2 Shear-peel bond strength

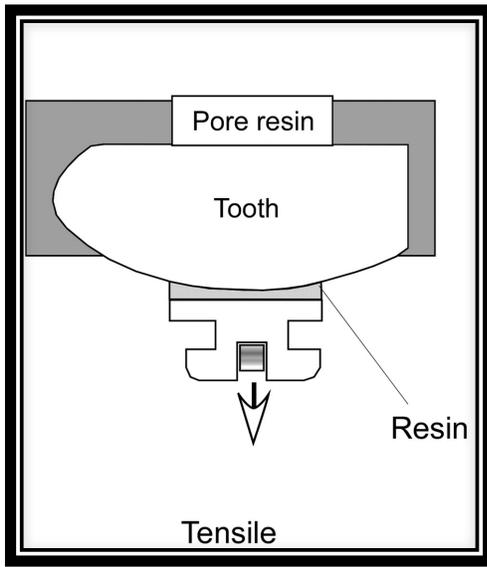
Very similar to the shear bond strength, the shear-peel bond strength is also recorded in $\text{N/mm}^2 = \text{MPa}$. However, to obtain a component of peel, the force must still be applied parallel to the enamel surface but away from the bracket-bonding agent interface. The extent at which an element of peel is incorporated is correlated to the distance of force application to that interface. The further a force is applied away from the tooth, the greater there will be a creation of a moment (Katona 1994). It is challenging to precisely quantify the magnitude of shear and peel forces but the articles reporting shear bond strengths are in reality shear-peel bond strengths (Katona 1994, Katona 1997, Eliades and Brantley 2000).

2.7.1.3 Tensile bond strength

Tensile bond strength is measured when a force is applied perpendicular to the tooth surface. The orthodontic attachment is pulled away from the tooth with a long loop harnessed around the

bracket (Katona and Chen 1994, Reicheneder, Gedrange et al. 2009). Pure tensile strength is also practically impossible and an element of peel is also distinguished during tensile bonding test.

Figure 2.2 Tensile bond strength testing



(Li 2011)

2.7.2 Testing machine

Laboratory dental materials research frequently involves the use of a mechanical testing machine. Based on the machine's mechanism of moving the crosshead and applying the loads on the samples, the testing machine can be screw-driven or use a servohydraulic system. The machines are also generally referred to as universal testing machines given the fact that they can be utilized for experiments on compression, bending, shear, torsion and tension. Some examples of Universal Testing Machines include the Instron (Norwood, MA) and the Zwick (Ulm, Germany). The forces recorded and the raw data are transferred to computer software. Some parameters can be precisely and controlled, e.g., the crosshead speed or the load cell weight.

2.7.3 *In vitro* bond strength testing parameters and standardization

In a meta-analysis by Finnema *et al.* from 2010, they identified a total of 27 experimental parameters worth mentioning for *in vitro* shear bond strength testing of orthodontic brackets to assess the validity of the results (Finnema, Ozcan et al. 2010). The parameters identified by Finnema *et al.* were the substrate origin, type of teeth, storage time, temperature and solution before bonding, cleaning method of the specimens, type of bracket and material, type of etchant, time of etching, adhesive type, amount of force at bracket placement, light device type, total polymerization time and light directions, sample storage time, solution and temperature, thermocycling or not, testing machine, type of bond strength method, crosshead speed, force location on bracket, blade design, Adhesive Remnant Index (ARI) or not, magnification used to determine the ARI and bond strength presented in MPa or not (Finnema, Ozcan et al. 2010).

Unfortunately, many studies omit to mention all the conditions in which the investigations have taken place and this makes comparison difficult among different shear bond strength experiments. As previously mentioned, the loading application methods can be either shear, tensile or torsion. The substrates most commonly used are enamel of extracted premolars or third molars (normal or fluorated), composite, porcelain or amalgam (Eliades and Brantley 2000). The storage time after bonding varies from no storage (immediate testing) to 5 years (Williams and Svare 1985), with 6 months as a normal method suggested by (Rueggeberg 1991, Soderholm 1991). However, some researchers have executed the shear bond strength rather earlier (2.5, 5, 10, 15, and 30 minutes) claiming that 24h does not simulate clinical practice (Turk, Elekdag-Turk et al. 2007). Many storage solutions, such as saline, thymol, aqueous chloramine, formalin

and tap or distilled water, have been used at different concentrations and temperatures (Eliades and Brantley 2000). The crosshead speed suggested for uniformity purposes is normally 0.5mm/min (Eliades, Viazis et al. 1991, Kao, Eliades et al. 1995). Thermocycling or cycling loading usually generates lower bond strengths in comparison to static testing (Elekdag-Turk, Turk et al. 2008, Mansour, Drummond et al. 2011). The most used control group, when comparing SEP to conventional etching systems, is composed of 37% phosphoric acid and Transbond primer and adhesive XT (Scougall Vilchis, Yamamoto et al. 2009).

Finnema *et al.* concluded that photopolymerization time, water storage and crosshead speed were the parameters shown to most affect the shear bond strength and that homogenous guidelines of *in-vitro* bond strength research are clearly indicated to assure standardization among different experiments (Finnema, Ozcan et al. 2010). On the other hand, Cheba *et al.* found no significant differences in the shear bond strengths measurements with variations in the photopolymerization time (20s vs 40s) with 17.95 ± 3.26 and 18.02 ± 3.43 MPa respectively and crosshead speeds (0.5mm/min vs 5.0mm/min) with 18.88 ± 2.94 and 17.09 ± 3.76 MPa respectively (Cheba 2012).

2.7.4 Bond strength and debonding force

Bond strength is defined as a measure of interfacial adhesion between a substrate and the bonded material, sometimes mediated by an adhesive agent (Eliades 2012). The data collected from the universal testing machines is recorded in Newtons (N) and corresponds to the stress recorded at the time of bonding failure, also known as debonding or fracture force. The formula to obtain the bond strength, which is largely expressed in Megapascals (MPa), is the debonding force divided by the surface area of the orthodontic attachment in mm^2 .

$$\text{Shear bond strength (MPa)} = \frac{\text{Stress at bond failure (N)}}{\text{Area of bracket base (mm}^2\text{)}}$$

The bond strength is also occasionally described in kilogram per square centimeter (kg/cm²) or pounds per square inch (lb/in²) (Powers, Kim et al. 1997).

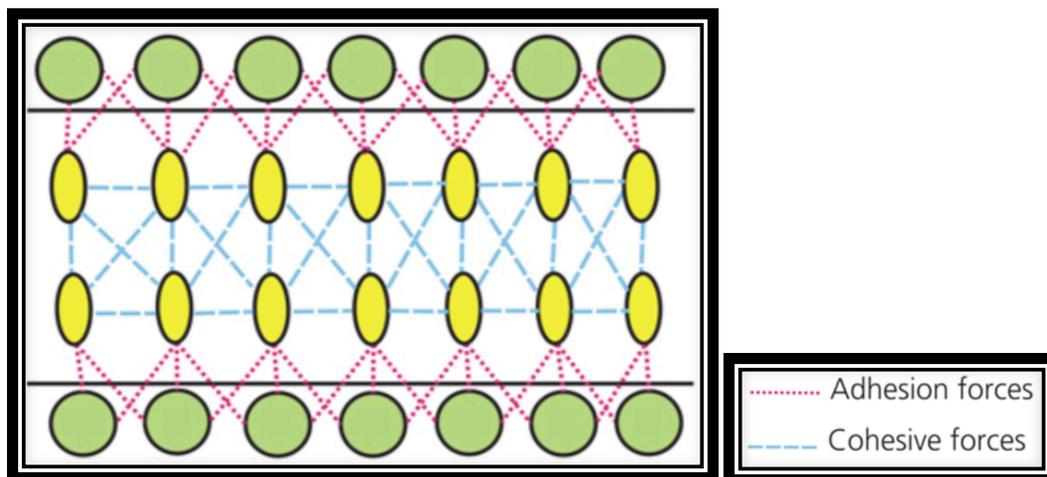
2.8 Optimum bond strength in orthodontics

The bond strength for an orthodontic attachment should be able to withstand the forces of mastication to retain the brackets for the desirable treatment duration, but it should be low enough to prevent enamel damage at the time of debonding. Average forces of mastication have been found to be ranging from 40 to 120 N (Brantley and Eliades 2000). With a force of 120N, Powers found that with a typical bracket that has a surface area of 16mm², the bond strength should be 7.5N/mm² or 7.5 MPa (Powers, Kim et al. 1997). In 1975, Reynolds reported adequate bond strength between 5.9 and 7.8 MPa (Reynolds 1975). Therefore, a bond strength ranging from 6 and 8 MPa has been found to be the “clinically acceptable” standard in the literature for *in vitro* shear bond strength testing (Reynolds 1975, Joseph and Rossouw 1990, Whitlock, Eick et al. 1994, Finnema, Ozcan et al. 2010, Wiltshire and Noble 2010). Nonetheless, *in vitro* SBSs testing with glass ionomer were found to perform inferiorly when compared to conventional bonding, with shear bond strength as low as 2.2 MPa, however this lower bond strength still performed satisfactorily intraorally (Fricker 1994, Wiltshire 1994, Wiltshire and Noble 2010). In 1974, Retief suggested that bond strength should be less than 14 MPa to prevent breakage within the enamel breaking strength but enamel fracture occurred in his experiment at as low as 9.7 MPa (Retief 1974). It is as important to observe the range of SBS values recorded to extrapolate whether a bonding product tested *in vitro* could perform consistently in clinical situations (Wiltshire and Noble 2010).

2.9 Bonding failure site and Adhesive Remnant Index

Cohesive forces are often referred to as intermolecular forces such as those from hydrogen bonding and Van der Waals forces which cause a tendency of like-molecules to resist separation (Chemwiki 2014). Adhesive forces are the attractive forces between two substances of different nature; such as mechanical or chemical forces (Chemwiki 2014). Cohesive bonding failures occur within the bonding agent, the enamel, or most unlikely, within the bracket itself. On the other hand, adhesive failures materialize at the resin-tooth or resin-bracket interface.

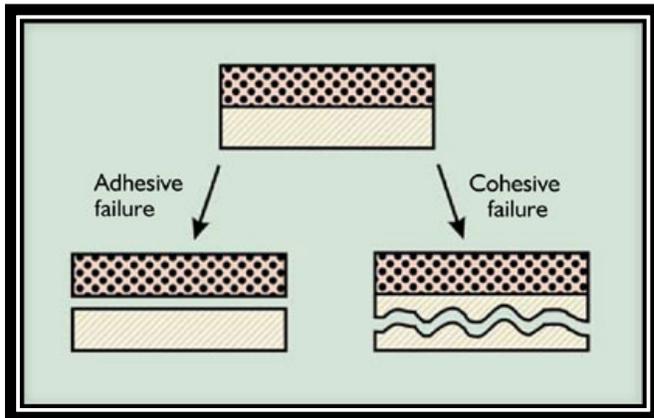
Figure 2.3 Adhesion and cohesive forces



(www.adhesion.org 2016)

Clinically, the ideal bonding failure location during orthodontic bracket removal would be an adhesive failure at the resin-tooth interface. However, de-bonding in orthodontics occurs mostly as a combination of adhesive-cohesive bond failures, whether it is accidental or provoked by the clinician.

Figure 2.4 Adhesive and cohesive failure



(Darvell and Clark 2000)

The type of bond failure can be qualified by using a gradation from 0 to 3, such as that found in the Adhesive Remnant Index developed by Årtun and Bergland in 1984 (Årtun and Bergland 1984). The tooth is typically observed under a microscope at certain magnification, 10X being the most commonly utilized. The index was modified by Bishara in 1999, with a score ranging from 1 to 5 (Bishara, VonWald et al. 1999).

Table 2.6 Adhesive Remnant Index (Årtun and Bergland)

Score	Criteria
0	No adhesive left on the tooth
1	Less than half of the adhesive left on the tooth
2	More than half of the adhesive left on the tooth
3	All adhesive left on the tooth, with distinct impression of the bracket mesh

(Årtun and Bergland 1984)

Table 2.7 Modified Adhesive Remnant Index (Bishara et al.)

Score	Criteria
1	All the composite left on tooth and impression of the bracket base
2	> 90% composite left on tooth
3	10-90% composite left on tooth
4	< 10% composite left on tooth
5	No composite left on enamel

(Bishara, VonWald et al. 1999)

The literature has shown multiple times that there is a significant difference in the ARI score between the teeth bonded either with a conventional etching versus a self-etching primer with more adhesive remaining on the enamel surface at the debond in the conventional etching group (Hosein, Sherriff et al. 2004, Scougall Vilchis, Yamamoto et al. 2009, Scougall-Vilchis, Ohashi et al. 2009, Øgaard and Fjeld 2010, Ho, Akyalcin et al. 2011).

2.10 Enamel loss

Enamel loss after acid etching has been found to vary significantly between studies. This is due to the type, concentration and length of acid contact time variation between studies (Hosein, Sherriff et al. 2004). Fitzpatrick and Way demonstrated a total enamel loss of 55µm after the process of etching, debonding and polishing (Fitzpatrick and Way 1977). Brown and Way showed very similar results with increased enamel loss using a highly filled resin adhesive as opposed to an unfilled polymethylmethacrylate resin (Brown and Way 1978). The same

investigators found considerable abrasion of the enamel at the polishing stage with a zirconium silicate polishing agent.

The enamel loss from prophylaxis with a bristle brush before acid etching is typically in the 10 μm range with a bristle and 7 μm with a rubber cup (Thompson and Way 1981). Most of the manufacturers recommend pumice prophylaxis before acid etching even though it has been proven to have no influence on bond failure rates whether a conventional acid etching is used with a composite resin bonding agent or with a resin modified glass ionomer cement (Barry 1995).

The amount of enamel loss during 15 to 30 seconds 37% phosphoric acid etching is normally between 8.8 μm and 16.4 μm (Legler, Retief et al. 1990). However, a more recent study has shown that the range of enamel loss was between 1.11 μm and 4.57 μm (Hosein, Sherriff et al. 2004). The advent of self-etch primers has considerably reduced the depth of enamel etching, with enamel loss ranging from 0.03 μm to 0.74 μm measured in the same experiment (Hosein, Sherriff et al. 2004). Therefore, these bonding systems produce less adverse effects to the enamel surface, and the clean up procedure is simpler for the orthodontist (Øgaard and Fjeld 2010).

The method of adhesive remnant removal after debonding has an influence on the quantity of enamel loss. Hosein *et al.* compared 4 different adhesive removal methods and found that the maximum amount of enamel abrasion occurred with the ultrasonic scaler, followed by the high-speed tungsten carbide bur, slow-speed tungsten bur and the debonding pliers (Hosein, Sherriff

et al. 2004). Irrespective of the technique employed, more enamel was lost in the conventional acid-etching group, than in the self-etch primer group (Hosein, Sherriff et al. 2004).

2.11 Rebonding

In vitro studies have demonstrated that, with a conventional adhesive system, higher values for shear bond strength are obtained at the initial bonding. Subsequent rebonding results in lower and inconsistent SBS values, and the discrepancies may be related to the modifications in the enamel morphology as a result of the presence of adhesive remnants (Bishara, VonWald et al. 2000, Bishara, Laffoon et al. 2002). Mui *et al.* (1999) suggested that the optimal procedure for rebonding orthodontic attachments is to resurface the enamel using a tungsten carbide bur, re-etching the enamel, and use a new or re-use an old bracket after microetching (Mui, Rossouw et al. 1999).

Transbond Plus Self Etching Primer showed comparable or higher bond strength and a marginally lower ARI score, when compared with a conventional phosphoric acid etch adhesive (Rely-a-Bond, Reliance) over three debonding experiments in laboratory testing (Montasser, Drummond et al. 2008). The same article demonstrated no difference in the clinical performance of the SEP and a CEP adhesive protocol (Montasser, Drummond et al. 2008). A scanning electron microscopy examination of the enamel surfaces with Transbond Plus Self Etching Primer, after the three debondings, indicated a less aggressive etching pattern and reduced adhesive left on the teeth (Montasser, Drummond et al. 2008).

A study in a laboratory determined that irrespective of the etching and priming technique used for the initial bonding, omitting etching for the rebonding manifested in reasonable shear bond strengths for clinical use. No difference between the CEP and SEP SBS was found. However, the mean SBS values were significantly different when acid etching was not performed. Also, more enamel damage was found with the CEP technique (Zhang, Yao et al. 2014).

3. PURPOSE

Primarily, the purpose of this study was to evaluate the difference in shear bond strength of bonding and rebonding orthodontic attachments to the enamel surface when using a conventional adhesive system compared to a self-etching primer-adhesive.

The second purpose was to evaluate if re-etching is necessary prior to rebonding a bracket with either a conventional etching primer or a self-etching primer.

4. NULL HYPOTHESES

The following are the primary hypotheses of this study:

1. A higher shear bond strength will be observed with the conventional bonding system consisting of the fifth generation adhesive Transbond XT Adhesive Primer as compared to the sixth generation adhesive self-etching primer Transbond Plus SEP.
2. A higher shear rebond strength will be observed with Transbond XT Adhesive Primer as compared to with Transbond Plus SEP.
3. A lower rebond strength will be observed when re-etching is not undertaken.
4. Less residual resin will be found on the enamel surface with Transbond Plus SEP as compared to with Transbond XT Adhesive Primer.

5. MATERIALS AND METHODS

The experiment consisted of bonding orthodontic buttons to enamel of extracted human third molar human teeth. Four different test groups were determined and two *in vitro* shear bond strength tests were performed on each tooth using different bonding protocols. The following are descriptions of the various materials and methods used in this research.

5.1 Ethics

Ethics approval was acquired through the Health Research Ethics Board of the University of Manitoba on November 30, 2015 (Appendix 11.1).

5.2 Teeth collection and storage

The sample size included 100 sound extracted human third molars. The teeth were collected from an Oral and Maxillofacial Surgery private practice in Quebec City, Canada (*Maxillo Québec*). The extracted third molars were kept in 0.5% Chloramine T until use in this experiment. The teeth were then examined and those with cavities, restorations and/or abnormalities were rejected, in order to use only teeth with healthy and similar buccal and/or lingual surfaces.

Table 5.1 Materials used in the experiment

Material	Manufacturer	Ref. number
Tooth preparation		
NSK 500 Ultimate Handpiece	NSK America Corp.	
Circular diamond separating disc		
Copper rings		
Bosworth Fastray	Bosworth, IL	0921375
Monomer liquid		
Polymer powder		
Prophy Paste	Ortho Technology, Tampa Bay, Florida	15486
Bonding agents		
Transbond XT Adhesive Paste	3M Unitek, Monrovia, California	712-036
Transbond XT Light Cure Adhesive Primer	3M Unitek, Monrovia, California	712-034
Transbond XT Etching Gel	3M Unitek, Monrovia, California	712-039
Transbond Plus Self Etching Primer	3M Unitek, Monrovia, California	712-091
Bonding materials		
Curved stainless steel lingual buttons	GAC International, Central Islip, NY	30-000-01
Loading apparatus gauge	Federal: Miracle Movement 0.001" C81S, Providence, RI	
Debonding materials		
Universal testing machine	Zwick GmBH, Ulm, Germany	
Bencor Multi-T testing apparatus	Danville Engineering, San Ramon, CA	
Leica EZ4 Stereo microscope	Ontario, Canada	2942700
Nikon D7000 16.2 MP	Nikon Corporation, Tokyo, Japan	
Chemicals		
Distilled water		
Chloramine-T trihydrate 98%	Acros Organics, NJ	
Other		
Digital caliper	Mastercraft, Canada	58-6800-4
Incubator 37°C	Thelco/Canlab Model 2, Precision Scientific, Chicago, IL	

5.3 Tooth preparation

The teeth were rinsed with water and cut with a circular diamond separating disc on a straight nose lab handpiece at the cemento-enamel junction. The roots were discarded. The crowns were embedded into Bosworth Fastray (Bosworth, IL), a self-curing acrylic, within copper cylindrical moulds. As the acrylic was setting, the flat buccal or lingual surfaces of the teeth were ensured to be parallel to the horizontal plane with a 90° T-bar instrument. The teeth were then removed from the copper rings and placed in plastic containers filled with distilled water and into an incubator at 37°C at 100% Relative Humidity for 24 hours to ensure complete polymerization of the acrylic at oral conditions, as well as adequate water sorption equilibration of the bonding.

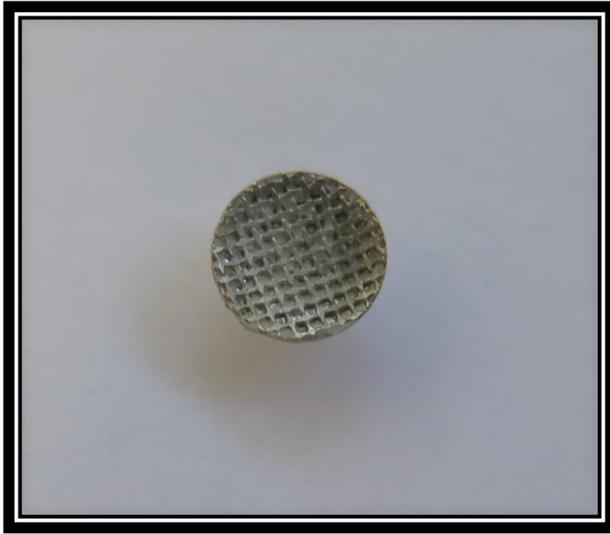
5.4 Bondable Stainless Steel Curved Base Lingual Buttons

The stainless steel buttons (30-000-01) used in this experiment were provided from GAC International. The curved base was chosen to allow optimal seating of the buttons to the curved surfaces of the third molars. The diameters of 10 randomly selected lingual buttons were measured using a digital caliper to obtain an average diameter (3.365mm). Then, the average surface area of the buttons was calculated using the formula:

$$Area = \pi r^2 = \pi \left(\frac{d}{2}\right)^2$$

The mean measurement was found to be 8.89mm². The different metal proportions of the buttons consist of 10-25%wt Chromium, 5-15%wt Nickel, 0-5%wt Manganese, 0-3%wt Molybdenum and the balance of Iron (MATERIAL COMPOSITION SHEET FOR LINGUAL AND WELDABLE LINGUAL ATTACHMENT).

Figure 5.1 Lingual button curved



5.5 Adhesive materials

Transbond XT Light Cure Adhesive System (3M Unitek)

Transbond XT Adhesive Paste (712-036)

The adhesive paste consisted of a light-cured resin composite used for direct and indirect bonding of orthodontic attachments such as metallic and ceramic brackets and metallic buttons. Its delivery is available in syringes and capsules. The components consists of 70-80%wt silane treated quartz, 10-20%wt Bisphenol A Diglycidylether Methacrylate (Bis-GMA), 5-10%wt Bisphenol A Bis (2-Hydroxyethyl Ether) Dimethacrylate (Bis-EMA), < 2%wt silane treated silica and < 0.2%wt Diphenyliodonium Hexafluorophosphate (MATERIAL SAFETY DATA SHEET 3M Unitek Transbond XT Light Cure Adhesive (712-036) 01/28/16).

Figure 5.2 Transbond XT Adhesive Paste



Transbond XT Light Cure Adhesive Primer (712-034)

The primer consisted of an unfilled resin of 45-55%wt Bisphenol A diglycidylether methacrylate (Bis-GMA) and 45-55%wt Triethylene glycol dimethacrylate (TEGDMA) dispensed in a 1:1 ratio. The other components are <1%wt Triphenylantimony, <0.5%wt 4-(Dimethylamino)-Benzeneethanol, <0.3%wt DL-Camphorquinone and <0.03%wt Hydroquinone (MATERIAL SAFETY DATA SHEET 3M Unitek Transbond XT Light Cure Adhesive Primer (712-034) 01/28/16).

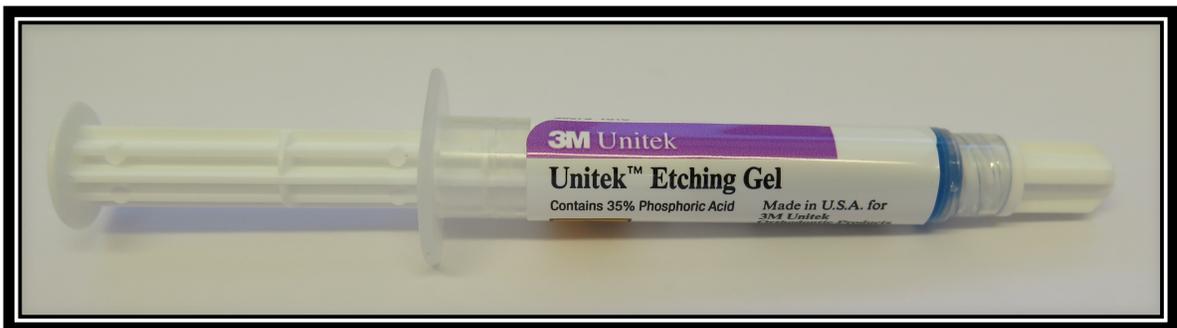
Transbond XT Etching Gel (712-039)

The etching gel consisted of composed of 35% phosphoric acid in water and amorphous silica.

Figure 5.3 Transbond XT Light Cure Adhesive Primer



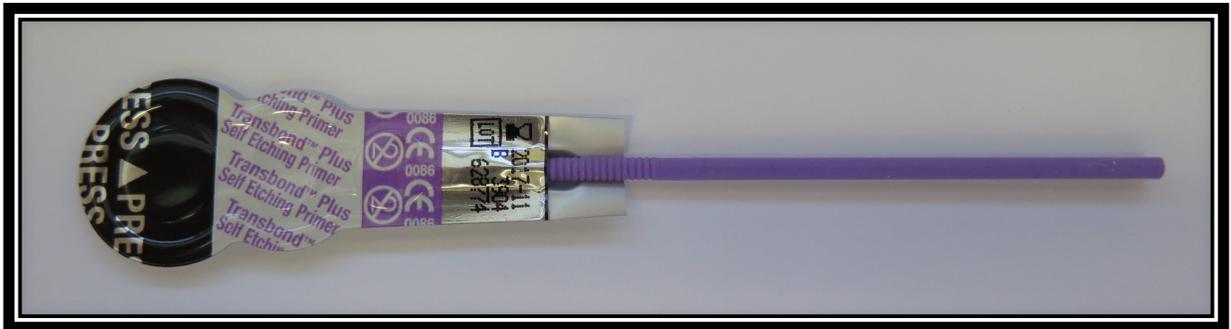
Figure 5.4 Transbond XT Etching Gel



Transbond Plus Self Etching Primer (712-091)

The Transbond Plus Self Etching Primer used, is a moisture resistant sixth generation light-curing bonding agent. This eliminates the need of using an etching gel prior to application of the primer. The product composes of two liquids embedded in two reservoirs that requires mixing before application on the teeth. The product comprises of 25-40 %wt 2-Propenoic Acid, 2-Methyl-Phosphinicobis (Oxy-2,1-Ethandiyl) Ester, 15-25%wt Water, 10-25%wt Mono HEMA Phosphate, 1-10%wt Tris[2-(Methacryloyloxy)Ethyl] Phosphate, <3%wt DL-Camphorquinone, <3%wt N,N-Dimethylbenzocaine and <3%wt Dipotassium Hexafluorotitanate (MATERIAL SAFETY DATA SHEET 3M Unitek Transbond Plus Self Etching Primer (712-090, 712-091) 02/25/16).

Figure 5.5 Transbond Plus Self Etching Primer



5.6 Bonding protocol

As previously mentioned, curved lingual buttons were used as the orthodontic attachments in this study. The one hundred teeth were randomly divided into 4 groups. Prior to bonding, the teeth were cleaned with a non-fluoridated and non-flavored prophylactic paste (Ortho Technology, Tampa Bay, FL) for 10 seconds, washed and dried. The bonding and rebonding protocols for each group were performed as follows:

Conventional etching primer (N=50)

Group A (control) (N=25)

Twenty-five teeth were bonded with the fifth-generation adhesive system consisting of Transbond XT Etching Gel (3M Unitek, Monrovia, California) and Transbond XT Light Cure Adhesive Primer (3M Unitek, Monrovia, California). The teeth were etched for 15 seconds, thoroughly rinsed with water spray for 20 seconds and air-dried for 20 seconds until a frosty appearance was observed to indicate sufficient etching of the enamel. Then, a thin uniform coat of Transbond XT Light Cure Adhesive Primer was applied for 5 seconds with a microbrush to the surface to be bonded, gently air-sprayed into a thin layer for 2 seconds and light-cured for 5 seconds. A uniform amount of Transbond XT Adhesive Paste was placed on the mesh pads and the buttons were placed on the surfaces of the teeth. A 500g vertical loading apparatus (Miracle Movement) was used to provide a homogeneous seating of the buttons to the teeth. The excess resin was removed using an explorer and the teeth were light-cured at an intensity of 1000 mW/cm² for 5 seconds mesial and 5 seconds distal to the metallic buttons using an Ortholux LED curing light lamp (3M Unitek, Monrovia, California) as per recommendation by the manufacturer. After the first shear bond strength test (details explained later) the remaining resin on the tooth was completely removed with a Brasseler USA (Savannah, GA) carbide bur H246M.31.009 #MW7901 Flame 5P. Resin removal was considered complete when the enamel surface appeared smooth and free of composite under an operatory lamp. The second bonding of this group (A2) was executed using the identical technique as the first bonding (A1).

Group B (N=25)

Twenty-five teeth were bonded with the fifth-generation adhesive system consisting of Transbond XT Etching Gel (3M Unitek, Monrovia, California) and Transbond XT Light Cure Adhesive Primer (3M Unitek, Monrovia, California). The teeth were etched for 15 seconds, thoroughly rinsed with water spray for 20 seconds and air-dried for 20 seconds until a frosty appearance was observed to indicate sufficient etching of the enamel. Then, a thin uniform coat of Transbond XT Light Cure Adhesive Primer was applied for 5 seconds with a microbrush to the surface to be bonded, gently air-sprayed into a thin layer for 2 seconds and light-cured for 5 seconds. A uniform amount of Transbond XT Adhesive Paste was placed on the mesh pads and the buttons were placed on the surfaces of the teeth. A 500g vertical loading apparatus (Miracle Movement) was used to provide a homogeneous seating of the buttons to the teeth. The excess resin was removed using an explorer and the teeth were light-cured at an intensity of 1000 mW/cm^2 for 5 seconds mesial and 5 seconds distal to the metallic buttons using an Ortholux LED curing light lamp (3M Unitek, Monrovia, California) as per recommendation by the manufacturer. After the first shear bond strength test (details explained later) the remaining resin on the tooth was completely removed with a Brasseler USA (Savannah, GA) carbide bur H246M.31.009 #MW7901 Flame 5P. The only difference between group A and B was that re-etching with the acid etch was not performed, before applying the primer/adhesive and the resin composite, for the second bonding.

Self-etching primer (N=50)

Group C (N=25)

Twenty five teeth were bonded with the sixth-generation adhesive system consisting of Transbond Plus Self Etching Primer (3M Unitek, Monrovia, California). Transbond Plus Self Etching Primer was mixed as per the manufacturer instructions, applied on the teeth with a saturated tip applicator for 5 seconds and gently air-sprayed into a thin layer for 2 seconds. A uniform amount of Transbond XT Adhesive Paste was placed on the mesh pads and the buttons were placed on the surfaces of the teeth. A 500g vertical loading apparatus (Miracle Movement) was used to provide a homogeneous seating of the buttons to the teeth. The excess resin was removed using an explorer and the teeth were light-cured at an intensity of 1000 mW/cm^2 for 5 seconds mesial and 5 seconds distal to the metallic buttons using an Ortholux LED curing light lamp (3M Unitek, Monrovia, California) as per recommendation by the manufacturer. After the first shear bond strength test (details explained later) the remaining resin on the tooth was completely removed with a Brasseler USA (Savannah, GA) carbide bur H246M.31.009 #MW7901 Flame 5P. The second bonding of this group (C2) was executed using the identical technique as the first bonding (C1).

Group D (N=25)

Twenty five teeth were bonded with the sixth-generation adhesive system consisting of Transbond Plus Self Etching Primer (3M Unitek, Monrovia, California). Transbond Plus Self Etching Primer was mixed as per the manufacturer instructions, applied on the teeth with a saturated tip applicator for 5 seconds and gently air-sprayed into a thin layer for 2

seconds. A uniform amount of Transbond XT Adhesive Paste was placed on the mesh pads and the buttons were placed on the surfaces of the teeth. A 500g vertical loading apparatus (Miracle Movement) was used to provide a homogeneous seating of the buttons to the teeth. The excess resin was removed using an explorer and the teeth were light-cured at an intensity of 1000 mW/cm² for 5 seconds mesial and 5 seconds distal to the metallic buttons using an Ortholux LED curing light lamp (3M Unitek, Monrovia, California) as per recommendation by the manufacturer. After the first shear bond strength test (details explained later) the remaining resin on the tooth was completely removed with a Brasseler USA (Savannah, GA) carbide bur H246M.31.009 #MW7901 Flame 5P. The only difference between group C and D was that re-etching and priming with the self-etching primer was not performed, before applying the resin composite, for the second bonding.

Table 5.2 Summary of the bonding protocols for the 4 groups

	Group	Number of teeth	First bonding	Second bonding
CEP	A	25	A1: Etch, TB XT primer and adhesive, TB XT adhesive paste	A2: Etch, TB XT primer and adhesive, TB XT adhesive paste
	B	25	B1: Etch, TB XT primer and adhesive, TB XT adhesive paste	B2: <u>No etch</u> , TB XT primer and adhesive, TB XT adhesive paste
SEP	C	25	C1: TB + SEP, TB XT adhesive paste	C2: TB + SEP, TB XT adhesive paste
	D	25	D1: TB + SEP, TB XT adhesive paste	D2: <u>No TB + SEP</u> , TB XT adhesive paste

CEP: conventional etching and priming

SEP: self-etch primer

TB: Transbond

Table 5.3 Summary of the experimental conditions

Experimental conditions	
Substrate origin	Enamel
Type of teeth	Extracted third molars (100)
Storage time before bonding	Variable
Storage temperature before bonding	4°C (refrigerator)
Storage solution before bonding	Distilled water with 0.5% Chloramine T
Cleaning of specimens	Prophy Paste (Ortho Technology) for 10 seconds
Bracket material	Stainless steel
Type of orthodontic attachment	Curved stainless steel lingual buttons (30-000-01) (GAC International)
Type of etchant	Group A+B: 35% phosphoric acid Group C+D: Transbond Plus SEP
Time of etching	15 seconds (35% phosphoric acid) 5 seconds (Transbond Plus SEP)
Adhesive type	Composite resin, Transbond XT Adhesive Paste
Amount of force at bracket placement	500g vertical loading apparatus (Miracle Movement)
Light device type	Ortholux LED curing light lamp (3M Unitek, Monrovia, California)
Total polymerization type	1000 mW/cm ²
Light directions	5 seconds mesial, 5 seconds distal
Sample storage time	1 week (168h)
Sample storage solution	Distilled water
Sample storage temperature	37°C, 100% humidity
Thermocycling	No
Testing machine	Zwick Universal testing machine (Ulm, Germany)
Shear testing as test method	Yes
Crosshead speed	0.5mm/min
Force location	Tooth-bracket interface
Blade design	Shearing blade, 10kN
ARI	Yes, score 1 to 5
Magnification used to determine ARI	10X magnification
Bond strength in MPa	Yes

Inspired from (Finnema, Ozcan et al. 2010)

5.7 Storage conditions

After each bonding, the teeth were stored in plastic containers filled with distilled water and in an incubator at 37°C at 100% Relative Humidity for 7 days. After the first shear bond strength test was executed, the teeth were bonded again according to their respective group bonding protocol and left in that same incubator for another week in the same storage conditions before the second debonding.

5.8 Debonding procedure

Two shear bond strength tests were completed as follows:

T1: First shear bond strength test, 1 week after initial bonding

T2: Second shear bond strength test, 1 week after second bonding

The teeth were mounted into the Bencor Multi-T testing castle (Danville Engineering, San Ramon, CA) and placed in the Zwick universal testing machine (Ulm, Germany) device, which was used to record the SBS with a crosshead speed of 0.5mm/min using a 10kN load cell. The measurements were recorded on a computer (Dell, Round Rock, TX) linked to the testing machine and the data were collected in megapascals (MPa). As previously mentioned, the surface area of the round buttons were measured using the formula:

$$\text{Surface area} = \pi r^2$$

The data were automatically converted by the computer in megapascals using the equation:

$$1 \frac{N}{mm^2} = 1 MPa$$

Figure 5.6 Tooth mounted onto the Bencor Multi-T Apparatus

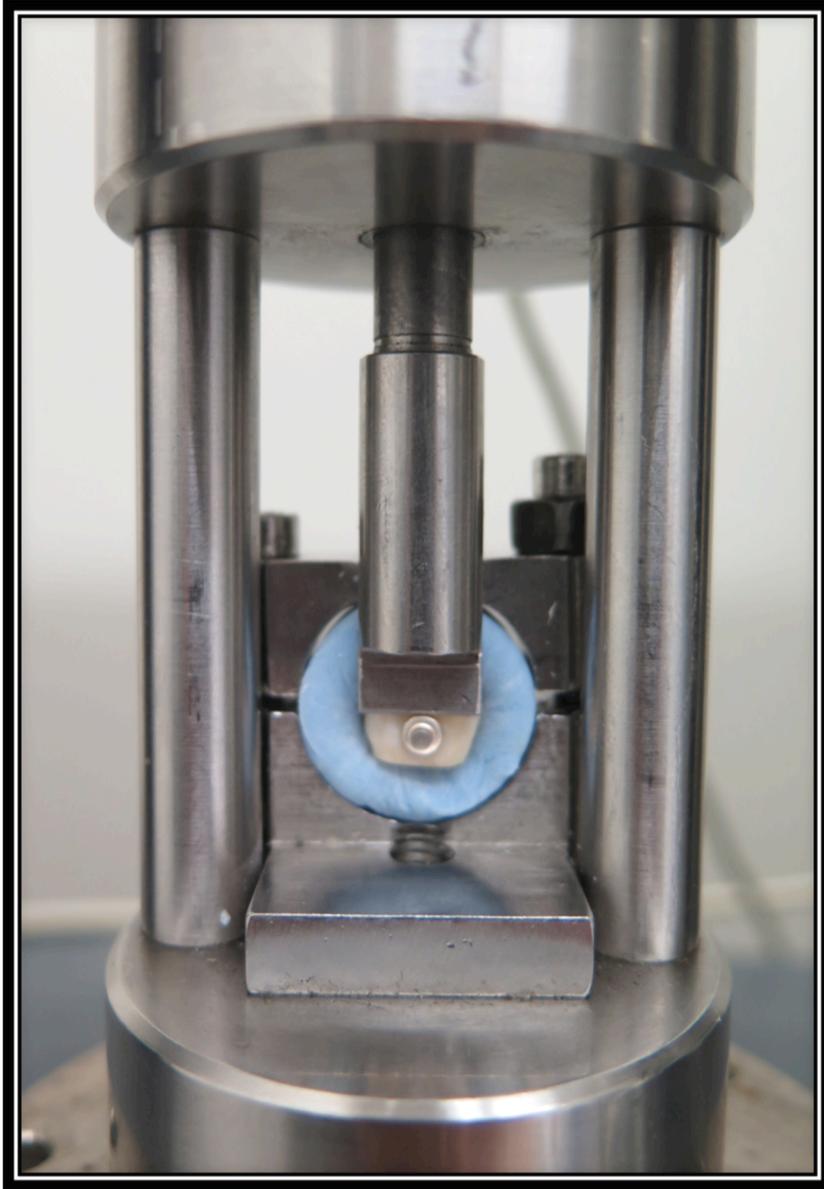


Figure 5.7 Zwick Universal testing machine

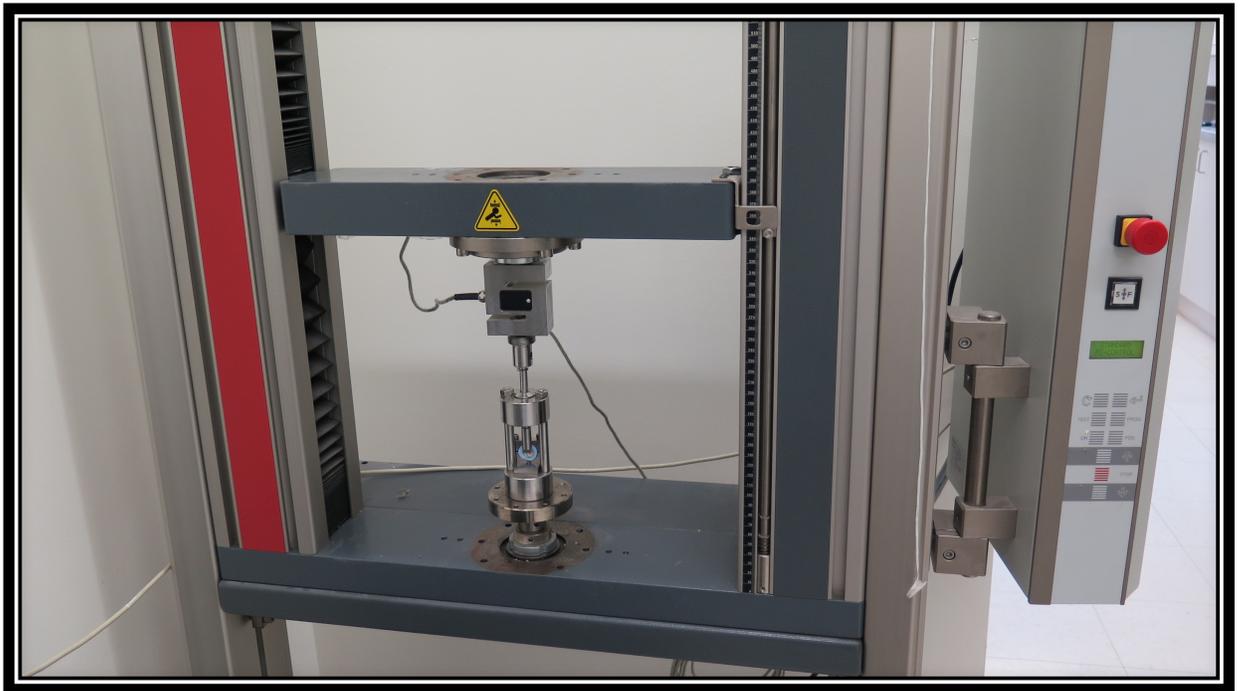
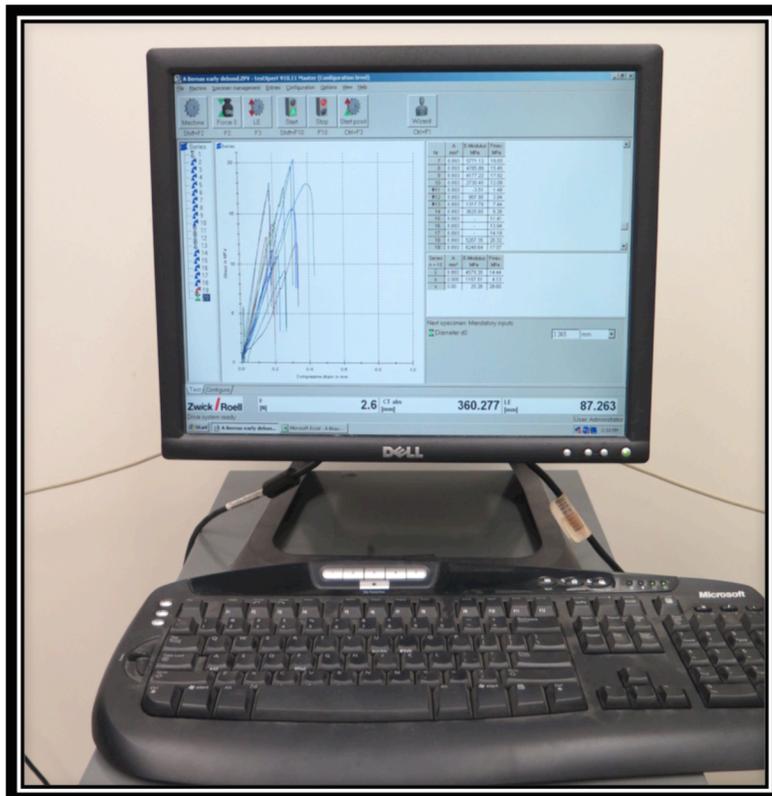


Figure 5.8 Computer linked to the Zwick Universal testing machine



5.9 Evaluation of the residual adhesive

Each tooth was observed under a Leica EZ4 Stereo microscope (Wetzlar, Germany) at 10X magnification after each debonding to assess the amount of resin composite left on the tooth. A score was given using the method described by Bishara *et al* (Bishara, VonWald et al. 1999), which was modified from the original Adhesive Remnant Index used by Årtun & Bergland (Årtun and Bergland 1984) .

Table 5.4 Adhesive Remnant Index (ARI) score (1-5)

Score	Criteria
1	All the composite left on tooth (100%) with the impression of the bracket base
2	> 90% composite left on tooth
3	10-90% composite left on tooth
4	< 10% composite left on tooth
5	No composite left on tooth

(Bishara, VonWald et al. 1999)

The ARI evaluation was completed twice on each of 100 teeth, after each debonding, for a total of 200 readings. 10 percent of the sample (i.e. 20 teeth) was randomly chosen and subjected to intra-rater and inter-rater reliability tests for the ARI evaluation to ensure results are reliable and valid. Intra-reliability testing included the principal investigator re-evaluating ten percent of the sample 2 weeks after the initial scoring. The results were statistically compared to original scores to ensure the results are reliable. Inter-reliability test included a qualified third party (a Graduate orthodontic resident) evaluating ten percent of the sample following proper training, for

consensus. The results were statistically compared to original results to ensure the results were valid. Variability equal or less than five percent was considered acceptable.

Figure 5.9 Adhesive Remnant Index (ARI) Score 1 through 5



5.10 Statistical analysis

All statistical analyses were performed using the OriginLab Software version 2016 (OriginLab Corp., Northampton, Massachusetts). A descriptive statistical analysis including the mean, standard deviation, coefficient of variation, median, minimum and maximum values and the range was calculated for each group at the two time points. The three-way ANOVA test and *Tukey's test* was utilized to compare the mean shear bond strength values among the different groups. The *Tukey's test* is used to identify where the difference is between factors and/or interactions. The *Fisher's exact* test was performed to determine any significant disparities in the *Adhesive Remnant Index* scores among the four groups. The inter- and intra-rater agreement used the *weighted kappa statistic* to calculate rater agreement. Significance level for all statistical tests of $p \leq 0.05$ was used.

6. RESULTS

6.1 Shear bond strength statistical analysis

The distribution of shear bond strength values was normally distributed and parametric tests were used for the statistical analysis. The three-way ANOVA test and *Tukey's test* was used to compare the mean shear bond strength values among the different groups. Significance was predetermined at a probability value of ≤ 0.05 . Table 6.1 is a recap summary of the bonding protocols used in this experiment.

Table 6.1 Summary of the bonding protocols for the 4 groups

	Group	Number of teeth	First bonding	Second bonding
CEP	A	25	A1: Etch, TB XT primer and adhesive, TB XT adhesive paste	A2: Etch, TB XT primer and adhesive, TB XT adhesive paste
	B	25	B1: Etch, TB XT primer and adhesive, TB XT adhesive paste	B2: <u>No etch</u> , TB XT primer and adhesive, TB XT adhesive paste
SEP	C	25	C1: TB + SEP, TB XT adhesive paste	C2: TB + SEP, TB XT adhesive paste
	D	25	D1: TB + SEP, TB XT adhesive paste	D2: <u>No TB + SEP</u> , TB XT adhesive paste

CEP: conventional etching and priming

SEP: self-etch primer

TB: Transbond

Since groups A1 and B1 as well as C1 and D1 had the same bonding protocols for the first bonding, they were grouped together for the statistical analysis (Table 6.2).

Table 6.2 Regrouping of the different bonding protocols for statistical analysis

	First bonding	Second bonding	Second bonding without re-etching
CEP	A1+B1 (N:50)	A2 (N:25)	B2 (N:25)
SEP	C1+D1 (N:50)	C2 (N:25)	D2 (N:25)

CEP: conventional etching and priming

SEP: self-etch primer

The number of teeth, mean shear bond strength, standard deviation, median, minimum, maximum, range and coefficient of variation for all groups are listed in Table 6.3.

Table 6.3 Descriptive data of the shear bond strength

Group	N	Mean (MPa)	SD	Median (MPa)	Minimum (MPa)	Maximum (MPa)	Range (MPa)	CV (%)
A1+B1	50	14.04	3.51	13.76	6.72	22.43	15.71	24.98
C1+D1	49	10.65	4.33	9.91	3.65	22.66	19.01	40.62
A2	25	14.66	3.70	13.76	9.38	22.29	12.91	25.21
B2	25	9.07	4.53	8.13	3.00	20.13	17.13	49.88
C2	24	12.72	3.72	12.19	5.47	21.45	15.98	29.20
D2	25	2.61	2.34	1.84	0.00	7.05	7.05	89.73

SD: standard deviation, CV: coefficient of variation

The mean shear bond strengths and standard deviations for all groups are illustrated in Figure 6.1. The comparisons between the groups and the significance of the mean differences are listed in Table 6.4.

Figure 6.1 Mean shear bond strength and standard deviations for all groups at first and second debonds

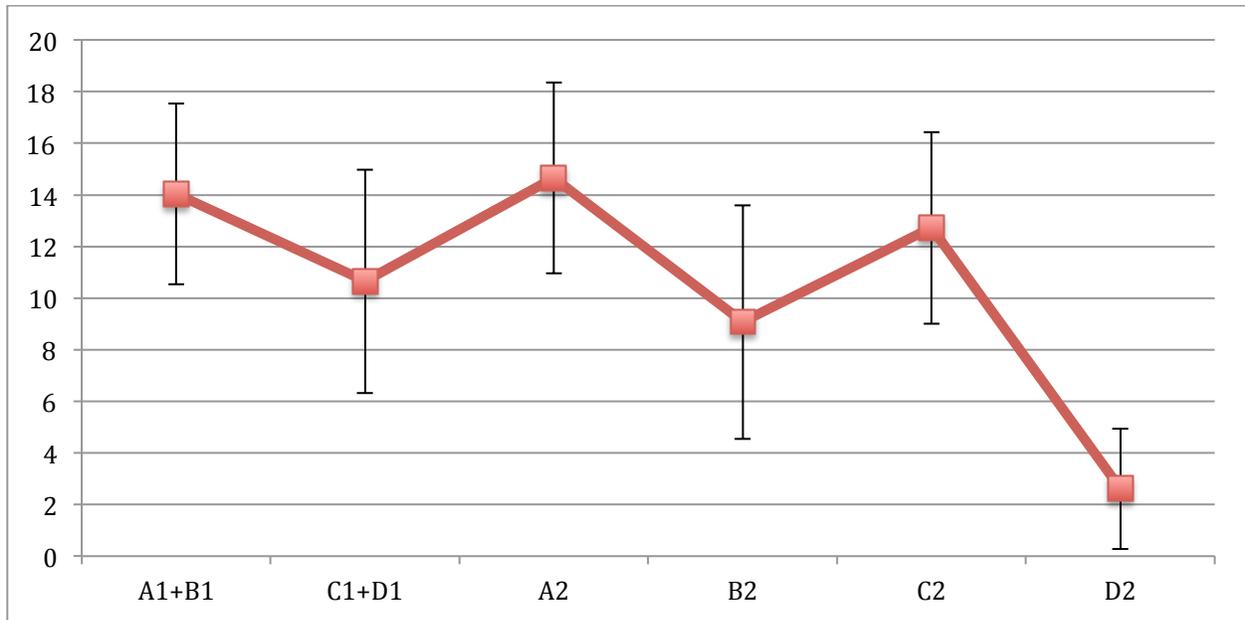


Table 6.4 Comparison of the mean shear bond strength between the groups, the statistical significance and the probability values

		Significance		p-value
A1+B1	C1+D1	Yes	$p < 0.001$	2.1849E-4
A1+B1	A2	No	$p > 0.05$	0.9856
A1+B1	B2	Yes	$p < 0.001$	3.8634E-6
C1+D1	C2	No	$p > 0.05$	0.2485
C1+D1	D2	Yes	$p < 0.001$	6.7248E-9
A2	B2	Yes	$p < 0.001$	7.4768E-6
C2	D2	Yes	$p < 0.001$	4.0583E-9
A2	C2	No	$p > 0.05$	0.4778
B2	C2	Yes	$p < 0.05$	0.0118

6.1.1 Comparison of the CEP and the SEP groups for the first debond (A1+B1 vs C1+D1)

As previously mentioned, groups A and B (conventional etching and primer) were combined for the first debond, as were groups C and D (self-etching primer), since they had the same bonding protocols and adding them together increases the statistical power. The highest mean shear bond strength of the two groups was obtained with group A1+B1 (14.04 ± 3.51 MPa). Group C1+D1 obtained a lower average shear bond strength of 10.65 ± 4.33 MPa. This mean difference of 3.39 MPa was considered highly statistically significant with a p-value of $2.19E-4$ ($p < 0.001$). The maximum values attained for both groups were in an almost precise order of magnitude whereas group C1+D1 showed the lowest minimum value of the two groups in the test series (3.65 MPa) for the first debond. The lowest coefficient of variation was calculated for group A1+B1 with 24.98% whereas group C1+D1 obtained 40.62%.

6.1.2 Comparison of first and second debonds within the CEP groups (A1+B1 vs A2)

The difference in the mean shear bond strength for groups A1+B1 and A2 within the CEP groups was calculated at 0.62 MPa and was not considered statistically significant with a probability value of 0.99 ($p > 0.05$). Group A2 obtained a mean shear bond strength of 14.66 ± 3.70 MPa, which is slightly higher than the mean value of A1+B1. The coefficient of variation and maxima were very analogous and interestingly the minimum value (9.38 MPa) was higher for the second debond by more than 2.5 MPa.

6.1.3 Comparison of first and second debonds within the CEP groups when omitting re-etching for the second debond (A1+B1 vs B2)

The mean difference between groups A1+B1 and B2 was 4.97 MPa and is highly statistically significant with a p-value of 3.86E-6 ($p < 0.001$). Group B2 obtained a mean shear bond strength of 9.07 ± 4.53 MPa. The coefficient of variation of group B2 was close to 50% as opposed to 25% for group A1+B1. The minimum value was 3.00 MPa, more than double the SBS for group A1+B1 (6.72 MPa).

6.1.4 Comparison of first and second debonds within the SEP groups (C1+D1 vs C2)

The difference in the average shear bond strength for groups C1+D1 and C2 was not statistically significant with a p-value of 0.25 ($p > 0.05$). The mean shear bond strength was 2.07 MPa higher for group C2, with 12.72 ± 3.72 MPa. Maxima and minima were almost identical, and the coefficient of variation was about 10% higher for C1+D1.

6.1.5 Comparison of first and second debonds within the SEP groups when omitting re-etching for the second debond (C1+D1 vs D2)

An important difference was evident in the mean shear bond strength of 8.04 MPa between groups C1+D1 and D2, and is highly statistically significant with a p-value of 6.72E-9 ($p < 0.001$). Group D2 obtained a mean shear bond strength of only 2.61 ± 2.34 MPa and a coefficient of variation of 89.73%. In group D2, five out of twenty-five (20%) SBS measurements were 0.00 MPa, because the attachments debonded while loading the teeth onto the Bencor Multi-T testing castle (Danville Engineering, San Ramon, CA).

6.1.6 Comparison of the second debonds with the CEP groups (A2 vs B2)

The highest mean shear bond strength of the two groups was measured for group A2 (14.66 ± 3.70 MPa). Group B2 attained a lower mean difference by 5.59 MPa, with a mean shear bond strength of 9.07 ± 4.53 MPa, and this was highly statistically significant with a p-value of $7.48E-6$ ($p < 0.001$). The minimum value for B2 decreased by over 6 MPa (3.00 MPa) compared to A2 (9.38 MPa). However, the maximum values were in a similar order of magnitude (20-22 MPa). The lowest coefficient of variation was calculated for group A2 with 25.21%, whereas group B2 obtained a high value of 49.88%.

6.1.7 Comparison of the second debonds with the SEP groups (C2 vs D2)

The highest mean shear bond strength of the two groups was generated for group C2 and averaged 12.72 ± 3.72 MPa. Group D2 obtained a mean shear bond strength of 2.61 ± 2.34 MPa, with a difference in mean of more than 10 MPa. This disparity was also highly statistically significant with a p value of $4.06E-9$ ($p < 0.001$). The minima and maxima of these two groups differed considerably with ranges of 5.47-21.45 MPa for group C2 and 0.00-7.05 MPa for group D2. The coefficient of variation also contrasted greatly with 29.20% and 89.73% respectively.

6.1.8 Comparison of groups A2 and C2 for the second debond (A2 vs C2)

The mean shear bond strength of the conventional etch and priming and the self-etch priming for the second debonding only differed by 1.94 MPa and this was not statistically significant with a p value of 0.48 ($p > 0.05$). Coefficients of variation were similar as were the maxima. However, the minimum for group C2 was inferior by almost 4 MPa.

6.1.9 Comparison of groups B2 and C2 for the second debond (B2 vs C2)

The mean shear bond strength of the conventional etch and priming omitting re-etching and the self-etch priming for the second debonding contrasted by 3.65 MPa and this was statistically significant with a p-value of 0.01 ($p < 0.05$). The coefficient of variation of group B2 was approximately 20% higher (49.88 vs 29.20%) than group C2 and the minimum value was 2.47 MPa higher for group C2 (5.47 vs 3.00 MPa).

6.2 Adhesive Remnant Index

Statistical comparison of the ARI scores distribution among the different groups was accomplished using the *Fisher's exact test*, which is the preferred analysis when some categories have low frequencies. Table 6.5 illustrates once again the criteria for the Adhesive Remnant Index scores and Table 6.6 displays the frequency and proportion of ARI scores 1 through 5 of all groups for the first and second debonds.

Table 6.5 Adhesive Remnant Index (ARI) score (1-5)

Score	Criteria
1	All the composite left on tooth (100%) with the impression of the bracket base
2	> 90% composite left on tooth
3	10-90% composite left on tooth
4	< 10% composite left on tooth
5	No composite left on tooth

(Bishara, VonWald et al. 1999)

At the first debond, group A1+B1 obtained ARI scores of 1 and 2 in 48/50 (96%) of the samples while ARI scores were more evenly distributed on the scale for group C1+D1 with ARI scores 1 and 2 and ARI scores 4 and 5 in 23/50 (46%) and 24/50 (48%) respectively. The difference in ARI scores between groups A1+B1 and C1+D1 is highly significant with a p-value of 1.81E-7 ($p < 0.001$).

Table 6.6 Frequency and percentage of ARI Scores for this experiment

Group	ARI Score					Total
	1	2	3	4	5	
A1+B1	32 (64%)	16 (32%)	0 (0%)	0 (0%)	2 (4%)	50
C1+D1	11 (22%)	12 (24%)	3 (6%)	6 (12%)	18 (36%)	50
A2	15 (60%)	2 (8%)	1 (4%)	2 (8%)	5 (20%)	25
B2	1 (4%)	1 (4%)	1 (4%)	0 (0%)	22 (88%)	25
C2	8 (32%)	0 (0%)	2 (8%)	0 (0%)	15 (60%)	25
D2	0 (0%)	0 (0%)	0 (0%)	0 (0%)	25 (100%)	25

For the second debond, the most frequent ARI score for group A2 was 1 in 15/25 (60%), 5 in 5/25 (20%) and either 2, 3 or 4 in 5/25 (20%) of the cases. ARI score 5 was found in the majority of the group B2 with 22/25 (88%). The difference between ARI scores of the two groups is highly significant with a p-value of 1.40E-6 ($p < 0.001$).

The second debond exhibited ARI scores of 1 in 8/25 (32%) and of 5 in 15/25 (60%) for group C2. All ARI scores (25/25, 100%) for the second debond of group D2 were 5. The difference between the two is also highly significant with a p-value of 6.36E-4 ($p < 0.001$).

6.2.1 Intra-rater and Inter-rater agreement for the Adhesive Remnant Index (ARI)

Of the total ARI score readings (200) completed in this study, 10 percent (20) were re-evaluated two weeks after the initial reading by the principal investigator to assess the intra-rater agreement. The re-assessment found that 100% of second ARI scores assigned to the debonded surfaces were the same as the initial evaluation with a *weighted kappa statistic* of 1.00, which indicates perfect agreement. The *weighted kappa statistic* takes into account the distance between rating categories, and penalizes large disagreements more than small ones.

The same sample (20 debonded teeth) was evaluated by a third party to verify the inter-rater agreement. Only one reading out of twenty (5%) differed between the principal investigator and the third party, which resulted in a *weighted kappa statistic* of 0.97 and is considered extremely high. Intra- and inter-rater agreement were accordingly considered highly valid.

7. DISCUSSION

Understandably, accidental detachment of brackets during the course of orthodontic treatment results in frustration for the patient, parent and practitioner, but also tends to increased treatment time. This is usually the consequence either of the patient's unintentionally applying inappropriate forces to the attachment or of a poor bonding procedure which disrupts the practice daily schedule (Bishara, VonWald et al. 2000). The present study explored the orthodontic shear bond strength of different adhesive protocols for a second bonding after accidental bonding breakage or the decision to reposition brackets.

7.1 Shear bond strength

Reynolds (1975) was one of the first authors to report that a bond strength ranging between 6 and 8 MPa was found to be the "clinically acceptable" standard for *in vitro* shear bond strength testing but many other researchers confirmed similar values (Reynolds 1975, Joseph and Rossouw 1990, Whitlock, Eick et al. 1994, Finnema, Ozcan et al. 2010, Wiltshire and Noble 2010). The minimum shear bond strength value should be considered as significant or of even more important to evaluate the performance of an adhesive, than the mean shear bond strength value (Wiltshire and Noble 2010). However, *in vitro* shear bond strength testing with glass ionomers was found to perform inferiorly when compared to composite resin, with shear bond strengths as low as 2.2 MPa. Nevertheless, this bonding agent still performed acceptably *in vivo* (Fricker 1994, Wiltshire 1994, Wiltshire and Noble 2010). Higher shear bond strengths may help to reduce undesirable bond failure of brackets during orthodontic treatment, but the risk of enamel damage and patient discomfort may arise when the brackets are detached at the end of

treatment (Zhang, Yao et al. 2014). Retief (1974) recommended that bond strength should be less than 14 MPa to prevent breakage within the enamel, but accidental fractures occurred at as low as 9.7 MPa in his investigation (Retief 1974). Kusy (1994) suggested that it is more important to consider the number of enamel fractures obtained with an adhesive rather than its actual mean shear bond strength (Kusy 1994). Powers *et al.* (1997) suggested that orthodontic bonding experiments should aim to attain a coefficient of variation, which is a measure of spread that describes the amount of variability relative to the mean, ranging from 20-30% (Powers, Kim et al. 1997).

Overall, the results from the current research project showed that the mean SBS of all groups but one (D2 with 2.61 MPa) were above the clinically accepted range of 6-8 MPa proposed by Reynolds (1975) to withstand typical orthodontic forces (Reynolds 1975). They are also above the 3-4 MPa mean range proposed by Wiltshire and Noble (2010) based on clinical studies on glass ionomers (Wiltshire and Noble 2010). Also, the control groups bonded with the conventional etch and primer (fifth generation) protocol obtained the highest mean shear bond strength values at each debond. The calculated coefficient of variation of groups C1+D1, B2 and D2 exceeded the range proposed by Powers (1997), which means that the amount of variability relative to the mean was high for these groups, which could be a concern, clinically, when assessing the clinical ramifications of the range of values at both the highest and lowest ends of the bond strength spectrum.

7.1.1 Comparison of the CEP and the SEP groups for the first debond (A1+B1 vs C1+D1)

The majority of *in vitro* studies have found no statistical difference in mean shear bond strengths between Transbond XT Light Cure Adhesive Primer and Transbond Plus Self Etching Primer (Arnold, Combe et al. 2002, Cal-Neto, Miguel et al. 2006, Vicente, Bravo et al. 2006, Turk, Elekdag-Turk et al. 2007, Scougall Vilchis, Yamamoto et al. 2009, Scougall-Vilchis, Ohashi et al. 2009, Ho, Akyalcin et al. 2011). Interestingly in this research, the CEP groups obtained a shear bond strength of 14.04 ± 3.51 MPa [6.72-22.43 MPa], while the SEP groups registered 10.65 ± 4.33 MPa [3.65-22.66 MPa], and this was found highly statistically significant ($p < 0.001$), as was found in two other studies, involving one with immediate debond and the other with thermocycling (Grubisa, Heo et al. 2004, Ho, Akyalcin et al. 2011). Both groups recorded maxima of approximately 22 MPa, however the SEP group (C1+D1) minimum fell under 6 MPa, which could be problematic under clinical circumstances according to Reynolds (1975) but not Wiltshire and Noble (2010). The difference in the mean shear bond strength between the two groups may be attributed to the shallower adhesive penetration when the self-etching primer is used compared to when phosphoric acid and the primer is employed separately with the conventional adhesive system (Cal-Neto and Miguel 2006).

It is important to note that groups C1 and D1 were combined into one group since they had the same bonding protocols and putting them together increases statistical power. However, when C1 and D1 were compared, they were considered to be statistically significantly different which in turn supposes that there has either been a repeated mistake during the bonding procedure, or that SEP bonding might have been less consistent in this research.

7.1.2 Comparison of first and second debonds within the CEP groups (A1+B1 vs A2)

Bishara and co-authors found that subsequent rebonding (3 total bondings) resulted in lower and inconsistent shear bond strengths with Transbond XT Light Cure Adhesive Primer (Bishara, VonWald et al. 2000, Bishara, Laffoon et al. 2002). The present study revealed consistent shear bond strength values for the CEP groups (A1+B1: 14.04 MPa, A2: 14.66 MPa) with a slightly higher mean value for the second debonding, ranges somewhat similar and minima above the clinically accepted minimum, and the differences between both groups' first and second debondings, were not statistically significant.

7.1.3 Comparison of first and second debonds within the CEP groups when omitting re-etching for the second debond (A1+B1 vs B2)

The mean shear bond strength obtained by group B2 that omitted re-etching prior to using the primer and adhesive was 9.07 ± 4.53 MPa. This average was almost 5 MPa less than group A1+B1 (14.04 ± 3.51 MPa), and this was considered highly statistically different. Zhang *et al.* (2014) obtained a lower shear bond strength with 6.57 ± 2.58 MPa [2.98-11.65 MPa] when the teeth were not re-etched, which is 2.50 MPa less than the current research (group B2) (Zhang, Yao et al. 2014).

7.1.4 Comparison of first and second debonds within the SEP groups (C1+D1 vs C2)

In a study by Montasser *et al.* (2008), Transbond Plus SEP demonstrated no difference in bond strength from rebonding twice, however the residual adhesive was removed with a scalpel unlike a carbide bur in the present study (Montasser, Drummond et al. 2008). The present study also discovered consistent shear bond strength values for the SEP groups (C1+D1: 10.65 MPa, C2:

12.72 MPa) with an approximately 2 MPa superior mean value for the second debonding, ranges somewhat similar and minima above the clinical accepted minimum, and the differences between the first and second debondings, were not statistically significant.

7.1.5 Comparison of first and second debonds within the SEP groups when omitting re-etching for the second debond (C1+D1 vs D2)

The difference between groups C1+D1 and D2 was determined highly statistically significant ($p < 0.001$) with a mean shear bond strength value for group D2 of 2.61 ± 2.34 MPa. This shear bond strength would definitely effect the outcome of orthodontic bonding clinically. Indeed, many orthodontic attachments (20%) shear bond strength in this group were not even recorded and given 0.00 MPa because the bond failed prior to being able to record it with the Zwick machine. Unfortunately, no group in their research, nor in the literature, tested the shear bond strength of teeth omitting any etch, primer and adhesive and using only composite resin on a second bonding, therefore no comparison can be made with the present study (group D2). From a clinical standpoint this does not seem to be a viable option in orthodontics.

7.1.6 Comparison of the second debonds with the CEP groups (A2 vs B2)

For the second debond, group B2 was not re-etched and obtained an average shear bond strength of 9.07 ± 4.53 MPa [3.00-20.13 MPa] while group A2's mean shear bond strength was measured at 14.66 ± 3.70 MPa [9.38-22.29 MPa]. Omitting the re-etching step lowered the mean shear bond strength by 5.59 MPa, which was considered highly statistically significant, but at the same time still superior to the 6-8 MPa range of Reynolds (1975) and 3-4 MPa of Wiltshire and Noble (2010). Similarly, Zhang *et al.* (2014) measured 14.18 ± 2.59 MPa [9.05-23.34 MPa] when re-

etching was performed with minima and maxima almost identical to the present study, but, as previously mentioned, obtained a lower shear bond strength with 6.57 ± 2.58 MPa [2.98-11.65 MPa] when the teeth were not re-etched, which is 2.50 MPa less than group B2 in the current research (9.07 ± 4.53 MPa) (Zhang, Yao et al. 2014). It is important to note that group B2's minimum value (3.00 MPa) fell at the Wiltshire and Noble (2010) threshold, same as for Zhang *et al.* (2014) with 2.98 MPa, and this might be insufficient to withstand the occlusal forces clinically. The generation of an adequate shear bond strength with group B2 may be attributed to the presence of shallow depressions on the enamel surface caused by the initial etching and still present after debonding and removal of all visible adhesive (Montasser, Drummond et al. 2008). It may be argued that the 9.07 MPa obtained in the present study without re-etching for a second time, could be considered “more biologic” or “safer” than a higher bond strength according to the work of Retief (1974) who found enamel fractures at magnitudes higher than 9 MPa (Retief 1974). Clinicians should be aware that using this bonding protocol may induce a higher incidence of debonds.

7.1.7 Comparison of the second debonds with the SEP groups (C2 vs D2)

The difference between using the self-etching primer for the second bonding resulted in a mean shear bond strength of 12.72 ± 3.72 MPa [5.47-21.45 MPa] while omitting use of the same product caused a mean shear bond strength of only 2.61 ± 2.34 MPa [0.00-7.05 MPa], which is highly significantly lower. Zhang *et al.* (2014) obtained a comparable 11.90 ± 2.70 MPa [8.89-18.02 MPa] for the second bonding using Transbond Plus Self Etching Primer and this is very similar to the current study but their minimum value (8.89 MPa) was slightly higher than the one

in the present study (5.47 MPa) (Zhang, Yao et al. 2014). As previously mentioned, no comparison can be made with group D2.

7.1.8 Comparison of groups A2 and C2 for the second debond (A2 vs C2)

Comparison of groups A2 (CEP: 14.66 ± 3.70 MPa) and C2 (SEP: 12.72 ± 3.72 MPa) was considered not significant in the present study as similarly demonstrated by Zhang *et al.* (2014) in their article with very similar magnitudes in mean shear bond strengths (CEP: 14.18 ± 2.59 MPa, SEP: 11.90 ± 2.70 MPa) (Zhang, Yao et al. 2014).

7.1.9 Comparison of groups B2 and C2 for the second debond (B2 vs C2)

A statistically significant difference between group B2 (9.07 ± 4.54 MPa) and C2 (12.72 ± 3.72 MPa) was found and this was due to the not re-etching before applying Transbond XT Light Cure Adhesive Primer within group B2.

7.2 Adhesive Remnant Index

An important concern at the time of bracket removal is to maintain a sound and undamaged enamel surface on the teeth. A low ARI score signifies a breakage of the bond at the attachment-adhesive interface. On the contrary, a higher ARI score indicates a bond failure at the adhesive-enamel interface, which implies less composite resin remnant left on the tooth but it increases the risks of superficial enamel fractures.

The *Fisher's exact test* revealed significant differences between the CEP and SEP groups at first debond and also whether or not re-etching was performed at bonding prior to second debonding. At first debond, group A1+B1 (CEP) obtained ARI scores 1 and 2 in 96% (48/50) of the samples leaving >90% of the composite on the tooth which is very similar to Buyukilmaz *et al.* (2003) with 95% (Buyukyilmaz, Usumez *et al.* 2003). ARI scores were more evenly distributed on the scale for group C1+D1 (SEP). Indeed, combined ARI score 1 and 2 and ARI score 4 and 5 were observed in 23/50 (46%) and 24/50 (48%) respectively. The difference between the CEP and SEP groups was established as highly significant. Cal-Neto *et al.* (2006) measured more than half of enamel bonding site covered with adhesive in 70% (14/20) of his sample bonded with the conventional etch and priming method (Cal-Neto, Miguel *et al.* 2006). The ARI scores for Transbond Plus SEP are similar to what Bishara *et al.* (2008) found with ARI scores spread along the scale (Bishara, Otsby *et al.* 2008). Interestingly, the same group of authors measured a ARI score of 1 and 2 in 85% (17/20) of their readings for the same product in a different study completed one year earlier as well as Buyukilmaz *et al.* with 90% (18/20) for the same ARI scores (Buyukyilmaz, Usumez *et al.* 2003, Bishara, Ostby *et al.* 2007). In an article by Scougall-Vilchis *et al.* (2009) comparing CEP and Transbond Plus SEP ARI scores, all interpretations were either no composite or less than 50% left on the enamel surface (Scougall-Vilchis, Ohashi *et al.* 2009). However, the same group found a more sparse distribution of ARI scores for Transbond Plus SEP compared to the study by Cal-Neto *et al.* (Cal-Neto, Miguel *et al.* 2006, Scougall Vilchis, Yamamoto *et al.* 2009). Ho *et al.* (2011) obtained ARI scores of 1 and 2 in 11/15 of their sample with etching and Transbond XT Light Cure Adhesive Primer and ARI scores of 4 and 5 in 11/15 of the teeth for Transbond Plus SEP at the 3 month debond time (Ho, Akyalcin *et al.* 2011). The shallower penetration of the resin tags achieved by Transbond Plus

SEP when compared to the conventional etching and priming method may be the reason for an increased bond failure at the resin-enamel interface as was observed in the present study (Øgaard and Fjeld 2010).

For the second debond, the general tendency was that less adhesive was left on the teeth for all groups. The most frequent ARI score for group A2 was 1 in 15/25 (60%), 5 in 5/25 (20%) and either 2, 3 or 4 in 5/25 (20%) of the cases. ARI score 5 was found in the majority of the group B2 second debonds with 22/25 (88%). This means that omitting to re-etch before the second bonding resulted of far more adhesive failures at the enamel-tooth interface. Sixty percent (15/25) of the ARI scores were 5 for the Transbond Plus SEP group at the second bonding compared to only 24% for the first bonding. All the debonds left no composite on the teeth for group D2. Zhang *et al.* (2014) used the Årtun and Bergland Adhesive Remnant Index for the assessment of the quality of debond so a direct comparison between this present study and theirs is difficult (Årtun and Bergland 1984, Zhang, Yao et al. 2014). However, the second bonding for the CEP group in their study revealed no composite left on the enamel in 30% (3/10) of the teeth and more than 90% composite left in the same proportion (3/10) (Zhang, Yao et al. 2014). When omitting re-etching, the entirety of their sample was left without composite on the enamel surface and this is similar to the current study (Zhang, Yao et al. 2014). As for the SEP protocol, 90% of the debond ended up with less than 50% of the resin left on the tooth, which could be interpreted as a different pattern from the present study (Zhang, Yao et al. 2014).

7.3 Evaluation of the null hypotheses

1. The null hypothesis that states that a higher shear bond strength will be observed with the conventional bonding system consisting of the fifth generation adhesive Transbond XT Adhesive Primer as compared to the sixth generation adhesive self-etching primer Transbond Plus SEP is accepted because there was a highly statistically significant difference ($p < 0.001$) between the conventional etch and priming and the self-etching primer groups at the first debond.
2. The null hypothesis that states that a higher shear rebond strength will be observed with Transbond XT Adhesive Primer as compared to with Transbond Plus SEP is rejected because there was no statistically significant difference ($p > 0.05$) between the conventional etch and priming and the self-etching primer groups at the second debond.
3. The null hypothesis that states that a lower rebond strength will be observed when re-etching is not undertaken is accepted because there was a highly statistically significant difference ($p < 0.001$) between the conventional etch and priming and self-etching primer groups whether re-etching was undertaken or not.
4. Finally, the null hypothesis that states that less residual resin will be found on the enamel surface with Transbond Plus SEP as compared to with Transbond XT Adhesive Primer is accepted because there was a statistically significant difference ($p < 0.05$) between the conventional etch and priming and the self-etching primer groups at the first and second debond.

7.4 Potential clinical application

Some of the potential clinical applications after analyzing the outcomes of the present study would be time, financial and iatrogenic considerations. Omitting to re-etch and using only a primer and adhesive would be similar to using a self-etch primer and could result in reduced chair time to rebond a new bracket. Although shown to be statistically significant by Fleming *et al.* (2012) when bonding time between a CEP technique versus a SEP system with a mean reduction of 23.2 seconds, this might not be clinically significant (Fleming, Johal *et al.* 2012). Furthermore, rebonding an orthodontic attachment with Transbond XT Light Cure Adhesive Primer instead of Transbond Plus Self Etching Primer could result in reduced adhesion expenses over the long term. Finally, and this could be the most important clinical application, skipping the use of etchant for a second time prior to the primer and adhesive application would possibly reduce additional iatrogenic damage to the enamel caused by repeated etching. On the other hand, the down side of not re-etching would be that there would be a possibility of increased probability of bonding failure with a reduced shear bond strength (mean: 9.07 MPa, minimum: 3.00 MPa) but it would be still considered “clinically acceptable”.

7.5 Limitations of the present research

It is challenging to make comparisons among orthodontic bonding experiments because of the absence of standardization in the study parameters. Indeed, Finnema *et al.* (2010) identified a total of 27 experimental parameters worth mentioning for *in vitro* shear bond strength testing of to assess the validity of the results (Finnema, Ozcan *et al.* 2010). Some of the sources of variability in the bonding protocol cited by Bishara *et al.* (1998) which can affect the shear bond strength may include premolar/molar crown contour variations, the quantitative aspects of

adhesive and force utilization during bonding, the method of adhesive removal, and interfacial characteristics of the bracket adhesive complex (Bishara, Gordan et al. 1998).

In the present study, the debonding procedures were completed one week after each of the initial or the second bondings. This does not necessarily reflect in fact the clinical situation where an archwire is normally tied to the brackets within minutes of placement. Also, immediate *in vitro* debonding has demonstrated inferior shear bond strengths in the literature (Turk, Elekdag-Turk et al. 2007, Ho, Akyalcin et al. 2011). At the same time, orthodontic brackets should be able to withstand masticatory forces throughout the duration of the treatment, so testing up to 24 months could be considered relevant. A solution of artificial saliva as the medium storage could have been used to mimic the oral cavity environment as much as possible within the boundaries of *in vitro* testing. Another consideration would be to have had the teeth undergo temperature change to simulate the variation caused by cold and hot food or beverages intake by the patient but the present study did not include it in its protocol as it may be considered too rigorous compared in fact to the clinical situation. Although precautions have been taken to ensure the testing with the shearing blade as parallel and as close as possible to the tooth surface, some variation in the angulation of the blade and/or distance could have included errors in the shear bond strength values. Finally, assessment of the enamel surface of the teeth after debond with scanning electron microscopy would have helped in quantifying the extent of enamel damage at the microscopic level but this was not part of the main purpose of this research.

8. CONCLUSIONS

Based on this *in vitro* study, which evaluated the effect of omitting re-etching on shear bond strength of rebonded orthodontic attachments, the following conclusions can be drawn:

1. The sixth generation adhesive (Transbond XT Light Cure Adhesive Primer) had higher shear bond strength than the self-etching primer (Transbond Plus Self Etching Primer) after the first debonding.
2. Transbond XT Light Cure Adhesive Primer and Transbond Plus Self Etching Primer provide equal and consistent rebondings.
3. Omitting re-etching with Transbond XT Light Cure Adhesive Primer may potentially provide adequate shear bond strength values for clinical use.
4. The adequate shear bond strengths obtained when omitting re-etching with Transbond XT Light Cure Adhesive Primer at the second debonding, may be due to the presence of adhesive remnants of the previously etched enamel, which continue to provide bond strength through cohesive forces.
5. Rebonding without re-etching with a self-etching primer is not a clinically viable option judging from this present study.
6. More resin composite remains on the enamel surface after the use of the conventional acid etching technique than after the use of the self-etching primer which takes more time to remove from the enamel surface and has the potential to cause enamel damage.

9. RECOMMENDATIONS

1. Future efforts should be directed towards stating all testing parameters utilized and also the standardization of *in vitro* bond strength testing to facilitate data interpretation among different studies.
2. Further research is needed to confirm the *in vitro* performance of rebonded orthodontic attachments without re-etching of the enamel surface with different bonding agents to verify if any may be adequate in a clinical situation.
3. Based on the results of the present study, a clinical trial investigating the bond failure rate of rebonded brackets with or without re-etching prior to applying the primer and adhesive would be helpful.
4. The results from this present study confirms that the following clinical bonding protocols for a rebonding are viable options in orthodontics:
 - Rebonding with etch, Transbond XT Light Cure Adhesive Primer followed by Transbond XT Adhesive Paste
 - Rebonding with Transbond Plus Self Etching Primer followed by Transbond XT Adhesive Paste
 - Rebonding with Transbond XT Light Cure Adhesive Primer followed by Transbond XT Adhesive Paste (no re-etching)

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11. APPENDIX

11.1 Ethics Approval

		P126-770 Bannatyne Avenue Winnipeg, Manitoba Canada, R3E 0W3 Telephone : 204-789-3255 Fax: 204-789-3414	
UNIVERSITY OF MANITOBA Research Ethics - Bannatyne Office of the Vice-President (Research and International)			
HEALTH RESEARCH ETHICS BOARD (HREB) CERTIFICATE OF FINAL APPROVAL FOR NEW STUDIES Delegated Review			
PRINCIPAL INVESTIGATOR:		INSTITUTION/DEPARTMENT:	ETHICS #:
Dr. Antoine Beaudet		U of M/College of Dentistry	HS19177 (H2015:447)
APPROVAL DATE:		EXPIRY DATE:	
November 30, 2015		November 30, 2016	
STUDENT PRINCIPAL INVESTIGATOR SUPERVISOR (If applicable):			
Dr. W. Wiltshire			
PROTOCOL NUMBER:	PROJECT OR PROTOCOL TITLE:		
N/A	Shear bond strength of re-bonded orthodontic attachments using self-etching primers		
SPONSORING AGENCIES AND/OR COORDINATING GROUPS:			
University of Manitoba - Division of Orthodontics			
Submission Date of Investigator Documents:		HREB Receipt Date of Documents:	
November 26, 2015		November 30, 2015	
THE FOLLOWING ARE APPROVED FOR USE:			
Document Name	Version(if applicable)	Date	
<u>Protocol:</u>	Protocol (undated)	submitted November 24, 2015	
<u>Consent and Assent Form(s):</u>			
<u>Other:</u>			
CERTIFICATION			
The above named research study/project has been reviewed in a delegated manner by the University of Manitoba (UM) Health Research Board (HREB) and was found to be acceptable on ethical grounds for research involving human participants. The study/project and documents listed above was granted final approval by the Chair or Acting Chair, UM HREB.			
HREB ATTESTATION			
The University of Manitoba (UM) Research Board (HREB) is organized and operates according to Health Canada/ICH Good Clinical Practices, Tri-Council Policy Statement 2, and the applicable laws and regulations of Manitoba. In respect to clinical trials, the HREB complies with the membership requirements for Research Ethics Boards defined in Division 5 of the Food and Drug Regulations of Canada and carries out its functions in a manner consistent with Good Clinical Practices.			
- 1 -			
umanitoba.ca/research			

QUALITY ASSURANCE

The University of Manitoba Research Quality Management Office may request to review research documentation from this research study/project to demonstrate compliance with this approved protocol and the University of Manitoba Policy on the Ethics of Research Involving Humans.

CONDITIONS OF APPROVAL:

1. The study is acceptable on scientific and ethical grounds for the ethics of human use only. **For logistics of performing the study, approval must be sought from the relevant institution(s).**
2. This research study/project is to be conducted by the local principal investigator listed on this certificate of approval.
3. The principal investigator has the responsibility for any other administrative or regulatory approvals that may pertain to the research study/project, and for ensuring that the authorized research is carried out according to governing law.
4. **This approval is valid until the expiry date noted on this certificate of approval. A Bannatyne Campus Annual Study Status Report** must be submitted to the HREB within 15-30 days of this expiry date.
5. Any changes of the protocol (including recruitment procedures, etc.), informed consent form(s) or documents must be reported to the HREB for consideration in advance of implementation of such changes on the **Bannatyne Campus Research Amendment Form**.
6. Adverse events and unanticipated problems must be reported to the HREB as per Bannatyne Campus Research Boards Standard Operating procedures.
7. The UM HREB must be notified regarding discontinuation or study/project closure on the **Bannatyne Campus Final Study Status Report**.

Sincerely,



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

PROTOCOL NUMBER: N/A
PROJECT OR PROTOCOL TITLE: Great bond strength of re-banded orthodontic attachments using self-curing primers
SPONSORING AGENCIES AND/OR COORDINATING GROUPS: University of Manitoba - Division of Orthodontics
Submission Date of Investigator Documents: November 25, 2018
HREB Receipt Date of Documents: November 30, 2018
THE FOLLOWING ARE APPROVED FOR USE:
Document Name: [Redacted]

CERTIFICATION
The above named research study/project has been reviewed in a delegated manner by the University of Manitoba (UM) Health Research Board (HREB) and was found to be acceptable on ethical grounds for research involving human participants. The study/project and documents listed above were granted final approval by the Chair or Acting Chair, UM HREB.

HREB ATTESTATION
The University of Manitoba (UM) Research Board (HREB) is organized and operates according to Health Canada/CHC Good Clinical Practice, Tri-Council Policy Statement 2, and the applicable laws and regulations of Manitoba. In respect to clinical trials, the HREB complies with the mandatory requirements for Research Ethics Boards defined in Division 2 of the Food and Drug Regulations of Canada and carries out its functions in a manner consistent with Good Clinical Practice.

Please quote the above Human Ethics Number on all correspondence.
Inquiries should be directed to the REB Secretary Telephone: (204) 789-3255/ Fax: (204) 789-3414

11.2 Journal Article and Submission Confirmation

Manuscript #	100417-669
Current Revision #	0
Submission Date	2017-10-04 09:28:36
Current Stage	Under Review
Title	Shear bond strength of rebonded orthodontic attachments using self-etching primers
Running Title	SBS of rebonded orthodontic attachments using SEP
Manuscript Type	Original Article
Special Section	N/A
Corresponding Author	Antoine Beaudet (University of Manitoba)
Contributing Authors	William Wiltshire , Rodrigo França , Bradley Klus
Financial Disclosure	I have no relevant financial interests in this manuscript.
Abstract	The purposes were to evaluate the difference in SBS of bonding and rebonding orthodontic attachments when using a CEP compared to a SEP and to evaluate if re-etching is necessary prior to rebonding with either a CEP or a SEP. Orthodontic metal buttons were bonded to extracted human molars according to 4 protocols: In Group A, teeth were bonded with the CEP for the first and second bondings. In Group B, teeth were bonded with the CEP for the first bonding but re-etching was omitted for the second bonding. In Group C, teeth were bonded with the SEP for both bondings. In Group D, teeth were bonded with the SEP for the first bonding but re-etching was omitted for the second bonding. The SBSs when the SEP was used for the first bonding were significantly lower ($P < 0.001$; 10.65 ± 4.33 MPa) than when the CEP was used (14.04 ± 3.51 MPa). The second bonding omitting re-etching but still using the primer and adhesive generated significantly lower SBSs ($P < 0.001$; 9.07 ± 4.53 MPa) than when re-etching was used (14.66 ± 3.70 MPa), but still above the "clinically acceptable" standard of 6-8 MPa for in vitro SBS testing. The second bonding SBS omitting re-etching and not using any primer or adhesive was very low (2.61 ± 2.34 MPa) and would definitely negatively effect the outcome of orthodontic bonding clinically. When rebonding a bracket, re-etching may be omitted but a primer and adhesive must be applied to obtain a "clinically acceptable" SBS.
Assistant Editor	Assigned
Key Words	orthodontic bonding, shear bond strength, self-etch, total etch, rebonding
Conflict of Interest	I have no conflict of interest that I should disclose.

Shear bond strength of rebonded orthodontic attachments using self-etching primers

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ABSTRACT

Objectives: The purposes were to evaluate the difference in SBS of bonding and rebonding orthodontic attachments when using a CEP compared to a SEP and to evaluate if re-etching is necessary prior to rebonding with either a CEP or a SEP.

Materials and Methods: Orthodontic metal buttons were bonded to extracted human molars according to 4 protocols: In Group A, teeth were bonded with the CEP for the first and second bondings. In Group B, teeth were bonded with the CEP for the first bonding but re-etching was omitted for the second bonding. In Group C, teeth were bonded with the SEP for both bondings. In Group D, teeth were bonded with the SEP for the first bonding but re-etching was omitted for the second bonding.

Results: The SBSs when the SEP was used for the first bonding were significantly lower ($P < 0.001$; 10.65 ± 4.33 MPa) than when the CEP was used (14.04 ± 3.51 MPa). The second bonding omitting re-etching but still using the primer and adhesive generated significantly lower SBSs ($P < 0.001$; 9.07 ± 4.53 MPa) than when re-etching was used (14.66 ± 3.70 MPa), but still above the "clinically acceptable" standard of 6-8 MPa for in vitro SBS testing. The second bonding SBS omitting re-etching and not using any primer or adhesive was very low (2.61 ± 2.34 MPa) and would definitely negatively effect the outcome of orthodontic bonding clinically.

Conclusions: When rebonding a bracket, re-etching may be omitted but a primer and adhesive must be applied to obtain a "clinically acceptable" SBS.

Keywords: Orthodontic Bonding, Shear Bond Strength, Self-Etch, Total Etch, Rebonding

INTRODUCTION

In 1964, Newman introduced a direct-bonding technique for bonding orthodontic brackets¹. This practice, however, only became a routine clinical procedure in the 1980s². Today, the most used bonding system in orthodontics is commonly referred to as the total-etch

technique, and it consists of three components combined in two products: a 35-37% phosphoric acid etchant in the first, and a combined primer and adhesive in the second³. Currently, the newer generations of adhesives (sixth and seventh) are referred to as self-etching primers and the three components are combined in one product thus eliminating the need for use of a syringe distributing the etch and subsequent rinsing. One of the important advantages of SEPs is a shallower etch creating less damage to the enamel structure.

When referring to orthodontic bonding, bond strength should be high enough to resist the forces of accidental debonding during the course of orthodontic treatment, but low enough to prevent the risk of enamel damage should debonding occur⁴. Bonding failure of an orthodontic attachment is possibly one of the most common emergencies encountered in orthodontics. Failure of bonded brackets is undesirable and can be costly in terms of clinical time and materials, can prolong treatment time, and can be frustrating for both the patient and the clinician. Beckwith *et al.* found that after missed appointments, replacing brackets was the second most important factor affecting the treatment duration⁵. Similarly, Shia mentioned that appliance breakage was on his top three list of factors contributing to lengthening treatment⁶. Skidmore also observed that if 2 or more brackets failed, the mean addition to treatment time was between 2.2-4.6 months⁷. This might be a reason why many investigators have conducted research on the bonding of orthodontic attachments.

To date, many studies have compared the mean shear bond strength of orthodontic brackets between the conventional etch and priming and the self-etching primers. Others have compared the average shear bond strength of a second and even third bonding of the same tooth. Most of the time after a bonding failure, new orthodontic attachments are rebonded using the same procedure as the first bonding. However, it must be noted that only one study has addressed

the difference between the CEP and SEP on a second orthodontic bonding and whether re-etching was performed or not⁸.

Primarily, the purpose of this study was to evaluate the difference in shear bond strength of bonding and rebonding orthodontic attachments to the enamel surface when using a conventional adhesive system compared to a self-etching primer-adhesive. The second purpose was to evaluate if re-etching is necessary prior to rebonding a bracket with either a conventional etching primer or a self-etching primer.

MATERIALS AND METHODS

Teeth

One hundred extracted sound human third molars with similar lateral surfaces were collected and kept in 0.5% Chloramine T solution until use.

Orthodontic buttons

Curved orthodontic stainless steel buttons (GAC International, Central Islip, NY) were used in this study. The average surface area of the button base was determined to be 8.89mm² by measuring 10 buttons' diameter with a digital caliper and using the formula $area = \pi \left(\frac{d}{2}\right)^2$.

Bonding procedures

The one hundred teeth were randomly divided into 4 groups. Prior to bonding, the teeth were cleaned with a non-fluoridated and non-flavored prophy paste (Ortho Technology, Tampa Bay, FL) for 10 seconds, washed and dried. The bonding and rebonding protocols for each group are summarized in Table 1 and were performed as follows:

Group A (control). Twenty-five teeth were bonded with the fifth-generation adhesive system consisting of Transbond XT Etching Gel (3M Unitek, Monrovia, CA) and Transbond XT Light Cure Adhesive Primer (3M Unitek, Monrovia, CA). The teeth were etched for 15 seconds, thoroughly rinsed with water spray for 20 seconds and air-dried for 20 seconds until a frosty appearance was observed to indicate sufficient etching of the enamel. Then, a thin uniform coat of Transbond XT Light Cure Adhesive Primer was applied for 5 seconds with a microbrush to the surface to be bonded, gently air-sprayed into a thin layer for 2 seconds and light-cured for 5 seconds. A uniform amount of Transbond XT Adhesive Paste was placed on the mesh pads and the buttons were placed on the surfaces of the teeth with a cotton plier applying a firm pressure. The excess resin was removed using an explorer and the teeth were light-cured at an intensity of 1000 mW/cm² for 5 seconds mesial and 5 seconds distal to the metallic buttons using an Ortholux LED curing light lamp (3M Unitek, Monrovia, CA) as per recommendation by the manufacturer. After the first shear bond strength test (details explained later) the remaining resin on the tooth was completely removed with a Brasseler USA (Savannah, GA) carbide bur H246M.31.009 #MW7901 Flame 5P. Resin removal was considered complete when the enamel surface appeared smooth and free of composite under an operatory lamp. The second bonding of this group A (A2) was executed using the identical technique as the first bonding (A1).

Group B. Twenty-five teeth were bonded exactly as groups A1 and A2 for the first bonding (B1). The only difference between group A and B was that re-etching with the acid etch was not performed, before applying the primer/adhesive and the resin composite, for the second bonding (B2).

Group C. Twenty-five teeth were bonded with the sixth-generation adhesive consisting of Transbond Plus Self Etching Primer (3M Unitek, Monrovia, CA). Transbond Plus Self Etching

Primer was mixed as per the manufacturer instructions, applied on the teeth with a saturated tip applicator for 5 seconds and gently air-sprayed into a thin layer for 2 seconds. A uniform amount of Transbond XT Adhesive Paste was placed on the mesh pads and the buttons were placed on the surfaces of the teeth with a cotton plier applying a firm pressure. The excess resin was removed using an explorer and the teeth were light-cured at an intensity of 1000 mW/cm² for 5 seconds mesial and 5 seconds distal to the metallic buttons using an Ortholux LED curing light lamp (3M Unitek, Monrovia, CA) as per recommendation by the manufacturer. After the first shear bond strength test (details explained later) the remaining resin on the tooth was completely removed with a Brasseler USA (Savannah, GA) carbide bur H246M.31.009 #MW7901 Flame 5P. The second bonding of this group (C2) was executed using the identical technique as the initial bonding (C1).

Group D. Twenty-five teeth were bonded exactly as groups C1 and C2 for the first bonding (D1). The only difference between group C and D was that re-etching and priming with the self-etching primer was not performed, before applying the resin composite, for the second bonding (D2).

Debonding procedure

Prior to each debonding, the teeth were kept in distilled water in an incubator at 37°C, 100% humidity for a week. A shearing blade attached to the Bencor Universal testing castle (Danville Engineering, San Ramon, CA) was loaded into the Zwick universal testing machine (Ulm, Germany) in a shear force mode at the enamel-bracket interface and using a cross-head speed of 0.5mm/min with a 10kN load cell was tested to failure. The shear bond strength was

measured for each tooth twice, one week after each bonding. A computer (Dell, Round Rock, TX) linked to the testing machine recorded the measurements in megapascals (MPa).

Adhesive Remnant Index

The enamel surface of each tooth was observed under a Leica EZ4 Stereo microscope (Wetzlar, Germany) at 10X magnification after each debonding to assess the amount of adhesive left on the tooth. A modified adhesive remnant index⁹ was used to quantify the amount of remaining resin composite using the following scale: 1 = All the composite left on tooth (100%) with the impression of the bracket base; 2 = > 90% composite left on tooth ; 3 = between 10-90% composite left on tooth; 4 = < 10% composite left on tooth; 5= No composite left on tooth.

Statistical analysis

A three-way ANOVA test and *Tukey's test* was utilized to compare the mean shear bond strength values among the different groups, and the *Fisher's exact* test was performed to determine any significant disparities in the *Adhesive Remnant Index* scores among the four groups. The inter- and intra-rater agreement used the *weighted kappa statistic* to calculate rater agreement. Significance level for all statistical tests of $p \leq 0.05$ was used.

RESULTS

Shear Bond Strength

Since groups A1 and B1 as well as C1 and D1 had the same bonding protocols for the first bonding, they were grouped together for the statistical analysis. The descriptive statistics for the shear bond strengths of all groups are given in Table 2 and the comparisons between the groups and the significance of the mean differences are listed in Table 3.

For the first debonding, the highest mean SBS was obtained for the CEP system (group A1+B1) with 14.04 ± 3.51 MPa. The SEP system (group C1+D1) obtained a lower average SBS of 10.65 ± 4.33 MPa. This mean difference of 3.39 MPa is highly statistically significant with a p-value of $2.19E-4$ ($p < 0.001$). The maximum values attained for both groups were in an almost precise order of magnitude (22 MPa) whereas group C1+D1 showed the lowest minimum value of the two groups in the test series (3.65 MPa).

For the second debonding, the highest mean SBS was once again obtained by the CEP system (group A2) with 14.66 ± 3.70 MPa. When re-etching was omitted but the primer and adhesive still used (group B2), the mean SBS was calculated at 9.07 ± 4.53 MPa. This mean difference of 5.59 MPa is highly statistically significant with a p-value of $7.48E-6$ ($p < 0.001$). The SEP system (C2) obtained a SBS of 12.72 ± 3.72 MPa and it is not statistically significant when compared to group A2 with a p-value of 0.48 ($p > 0.05$). An important reduction was evident in the mean shear bond strength of group D2 when neither re-etching nor priming a second time was accomplished with a mean SBS of only 2.61 ± 2.34 MPa. For group D2, five out of twenty-five (20%) SBS measurements were 0.00 MPa, because the attachments debonded while loading the teeth onto the Bencor Multi-T testing castle (Danville Engineering, San Ramon, CA).

No statistical difference ($p > 0.05$) was found between the first and second debondings with the CEP system (groups A1+B1 vs A2) and the SEP system (groups C1+D1 vs C2). Groups A2 and C2 even reached higher mean SBS than their counterpart for the first debonding, namely groups A1+B1 and C1+D1 (see Table 2).

Adhesive Remnant Index

At the first debond, group A1+B1 obtained ARI scores of 1 and 2 in 48/50 (96%) of the samples while ARI scores were more evenly distributed on the scale for group C1+D1 with ARI scores 1 and 2 and ARI scores 4 and 5 in 23/50 (46%) and 24/50 (48%) respectively. The difference in ARI scores between groups A1+B1 and C1+D1 is highly significant with a p-value of $1.81E-7$ ($p < 0.001$). Table 4 illustrates the frequency distribution of the modified ARI scores.

For the second debond, the most frequent ARI score for group A2 was 1 in 15/25 (60%), 5 in 5/25 (20%) and either 2, 3 or 4 in 5/25 (20%) of the cases. ARI score 5 was found in the majority of the group B2 with 22/25 (88%). The difference between ARI scores of the two groups is highly significant with a p-value of $1.40E-6$ ($p < 0.001$).

The second debond exhibited ARI scores of 1 in 8/25 (32%) and of 5 in 15/25 (60%) for group C2. All ARI scores (25/25, 100%) for the second debond of group D2 were 5. The difference between the two is also highly significant with a p-value of $6.36E-4$ ($p < 0.001$).

DISCUSSION

Understandably, accidental detachment of brackets during the course of orthodontic treatment results in frustration for the patient, parent and practitioner, but also tends to increased treatment time. This is usually the consequence either of the patient's unintentionally applying inappropriate forces to the attachment or of a poor bonding procedure which disrupts the practice daily schedule¹⁰.

This study explored the orthodontic shear bond strength of different adhesive protocols for a second bonding after accidental bonding breakage or the decision to reposition brackets. The present findings showed that the mean SBS of all groups but one (D2 with 2.61 MPa) were

above the clinically accepted range of 6-8 MPa proposed by Reynolds (1975) to withstand typical orthodontic forces¹¹. They are also above the 3-4 MPa mean range proposed by Wiltshire and Noble (2010) based on clinical studies on glass ionomers¹². Also, the control groups bonded with the conventional etch and primer (fifth generation) protocol obtained the highest mean shear bond strength values at each debond.

Some of the potential clinical applications after analyzing the outcomes of the present study would be time, financial and iatrogenic considerations. Omitting to re-etch and using only a primer and adhesive would be similar to using a self-etch primer and could result in reduced chair time to rebond a new bracket. Although shown to be statistically significant by Fleming *et al.* (2012) when bonding time between a CEP technique versus a SEP system with a mean reduction of 23.2 seconds, this might not be clinically significant¹³. Furthermore, rebonding an orthodontic attachment with Transbond XT Light Cure Adhesive Primer instead of Transbond Plus Self Etching Primer could result in reduced adhesion expenses over the long term. Finally, and this could be the most important clinical application, skipping the use of etchant for a second time prior to the primer and adhesive application would possibly reduce additional iatrogenic damage to the enamel caused by repeated etching. On the other hand, the down side of not re-etching would be that there would be a possibility of increased probability of bonding failure with a reduced shear bond strength (mean: 9.07 MPa, minimum: 3.00 MPa) but it would be still considered “clinically acceptable”.

This was an *in vitro* study and this does not necessarily reflect in fact the clinical situation where an archwire is normally tied to the brackets within minutes of placement. In addition, further research is needed to confirm the clinical *in vivo* performance of rebonded orthodontic attachments without re-etching of the enamel surface with different bonding agents to verify if

any may be adequate in a clinical situation prior to considering a clinical trial investigating the bond failure rate of rebonded brackets with or without re-etching prior to applying the primer and adhesive.

CONCLUSIONS

Based on this *in vitro* study, which evaluated the effect of omitting re-etching on shear bond strength of rebonded orthodontic attachments, the following conclusions can be drawn:

- The sixth generation adhesive (Transbond XT Light Cure Adhesive Primer) had higher shear bond strength than the self-etching primer (Transbond Plus Self Etching Primer) after the first debonding.
- Transbond XT Light Cure Adhesive Primer and Transbond Plus Self Etching Primer provide equal and consistent rebondings.
- Omitting re-etching with Transbond XT Light Cure Adhesive Primer may potentially provide adequate shear bond strength values for clinical use.
- The adequate shear bond strengths obtained when omitting re-etching with Transbond XT Light Cure Adhesive Primer at the second debonding, may be due to the presence of adhesive remnants of the previously etched enamel, which continue to provide bond strength through cohesive forces.
- Rebonding without re-etching with a self-etching primer is not a clinically viable option judging from this present study.
- More resin composite remains on the enamel surface after the use of the conventional acid etching technique than after the use of the self-etching primer which takes more time to remove from the enamel surface and has the potential to cause enamel damage.

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Table 1. Summary of the protocols for the first and second bondings

	Group	Number of teeth	First bonding	Second bonding
CEP	A	25	A1: Etch, TB XT primer and adhesive, TB XT adhesive paste	A2: Etch, TB XT primer and adhesive, TB XT adhesive paste
	B	25	B1: Etch, TB XT primer and adhesive, TB XT adhesive paste	B2: <u>No etch</u> , TB XT primer and adhesive, TB XT adhesive paste
SEP	C	25	C1: TB + SEP, TB XT adhesive paste	C2: TB + SEP, TB XT adhesive paste
	D	25	D1: TB + SEP, TB XT adhesive paste	D2: <u>No TB + SEP</u> , TB XT adhesive paste

CEP, conventional etching and priming; SEP: self-etch primer; TB: Transbond

Table 2. Descriptive statistics in Megapascals (MPa)

Group	N	\bar{x} (MPa)	SD	Range (MPa)	CV (%)
A1+B1	50	14.04	3.51	6.72-22.43	24.98
C1+D1	49	10.65	4.33	3.65-22.66	40.62
A2	25	14.66	3.70	9.38-22.29	25.21
B2	25	9.07	4.53	3.00-20.13	49.88
C2	24	12.72	3.72	5.47-21.45	29.20
D2	25	2.61	2.34	0.00-7.05	89.73

\bar{x} , mean, SD, standard deviation

Table 3. Comparison of the mean shear bond strength between the groups, the statistical significance and the probability values

		Significance		p-value
A1+B1	C1+D1	Yes	p < 0.001	2.1849E-4
A1+B1	A2	No	p > 0.05	0.9856
A1+B1	B2	Yes	p < 0.001	3.8634E-6
C1+D1	C2	No	p > 0.05	0.2485
C1+D1	D2	Yes	p < 0.001	6.7248E-9
A2	B2	Yes	p < 0.001	7.4768E-6
C2	D2	Yes	p < 0.001	4.0583E-9
A2	C2	No	p > 0.05	0.4778
B2	C2	Yes	p < 0.05	0.0118

Table 4. Frequency distribution of Modified Adhesive remnant Index Scores for the four groups

Group	ARI Score					Total
	1	2	3	4	5	
A1+B1	32 (64%)	16 (32%)	0 (0%)	0 (0%)	2 (4%)	50
C1+D1	11 (22%)	12 (24%)	3 (6%)	6 (12%)	18 (36%)	50
A2	15 (60%)	2 (8%)	1 (4%)	2 (8%)	5 (20%)	25
B2	1 (4%)	1 (4%)	1 (4%)	0 (0%)	22 (88%)	25
C2	8 (32%)	0 (0%)	2 (8%)	0 (0%)	15 (60%)	25
D2	0 (0%)	0 (0%)	0 (0%)	0 (0%)	25 (100%)	25

12. RAW DATA

Table 12.1 Raw data of the shear bond strength for the first debond

Sample	SBS
A1	7.55
A2	10.93
A3	15.39
A4	15.60
A5	12.97
A6	15.18
A7	20.28
A8	12.68
A9	11.12
A10	13.79
A11	16.91
A12	14.39
A13	15.67
A14	17.44
A15	11.49
A16	6.75
A17	10.42
A18	21.66
A19	13.72
A20	13.02
A21	19.07
A22	17.37
A23	11.82
A24	14.70
A25	12.35
B1	17.13
B2	12.58
B3	10.40
B4	11.16
B5	22.43
B6	12.03
B7	15.94
B8	14.15
B9	10.83
B10	17.97
B11	14.09
B12	11.65
B13	13.06
B14	11.13
B15	12.78
B16	9.46

B17	16.12
B18	19.92
B19	14.62
B20	12.92
B21	15.84
B22	12.59
B23	6.72
B24	18.17
B25	16.23
C1	6.96
C2	17.18
C3	10.51
C4	-
C5	7.08
C6	7.62
C7	10.97
C8	9.14
C9	15.32
C10	14.09
C11	22.64
C12	18.28
C13	10.38
C14	13.38
C15	16.27
C16	22.66
C17	17.93
C18	11.12
C19	5.13
C20	9.78
C21	11.24
C22	8.75
C23	6.47
C24	12.37
C25	10.65
D1	9.67
D2	7.13
D3	6.55
D4	3.65
D5	5.18
D6	8.86
D7	13.44
D8	8.92
D9	8.34
D10	12.58
D11	8.40
D12	6.18
D13	15.60
D14	12.72

D15	6.26
D16	7.19
D17	5.18
D18	9.21
D19	9.91
D20	8.05
D21	11.92
D22	11.57
D23	5.49
D24	12.72
D25	11.41

Table 12.2 Raw data of the shear bond strength for the second debond

Sample	SBS
A1	10.83
A2	18.05
A3	17.33
A4	12.69
A5	19.65
A6	12.09
A7	9.38
A8	11.41
A9	13.94
A10	14.19
A11	20.32
A12	17.57
A13	11.02
A14	20.64
A15	11.73
A16	13.76
A17	22.29
A18	16.36
A19	11.35
A20	17.14
A21	12.55
A22	11.61
A23	12.07
A24	17.62
A25	10.96
B1	14.02
B2	10.52
B3	6.59
B4	3.63
B5	7.57
B6	8.65
B7	10.74

B8	20.11
B9	7.61
B10	20.13
B11	8.78
B12	7.80
B13	11.52
B14	3.00
B15	5.26
B16	8.13
B17	6.21
B18	7.27
B19	4.02
B20	15.63
B21	10.69
B22	8.79
B23	4.19
B24	10.07
B25	5.89
C1	8.83
C2	10.77
C3	11.56
C4	18.31
C5	13.01
C6	8.45
C7	12.89
C8	8.97
C9	-
C10	5.47
C11	10.80
C12	10.30
C13	16.84
C14	16.10
C15	16.19
C16	15.29
C17	10.84
C18	14.90
C19	10.20
C20	21.45
C21	13.92
C22	10.75
C23	12.82
C24	17.07
C25	9.65
D1	0.00
D2	1.65
D3	6.60
D4	6.14
D5	1.84

D6	0.00
D7	2.26
D8	4.87
D9	6.71
D10	3.46
D11	1.09
D12	3.90
D13	0.00
D14	3.11
D15	4.71
D16	0.59
D17	0.00
D18	4.46
D19	0.00
D20	0.90
D21	0.78
D22	7.05
D23	2.41
D24	1.39
D25	1.39

Table 12.3 Raw data of the Adhesive Remnant Index for the first debond

Sample	ARI
A1	1
A2	1
A3	1
A4	1
A5	1
A6	1
A7	1
A8	5
A9	1
A10	1
A11	1
A12	1
A13	1
A14	2
A15	2
A16	1
A17	1
A18	2
A19	1
A20	1
A21	1
A22	2
A23	1

A24	2
A25	1
B1	2
B2	1
B3	2
B4	2
B5	2
B6	2
B7	1
B8	2
B9	1
B10	1
B11	2
B12	5
B13	1
B14	1
B15	2
B16	1
B17	1
B18	2
B19	2
B20	1
B21	2
B22	1
B23	1
B24	1
B25	1
C1	4
C2	2
C3	5
C4	5
C5	2
C6	5
C7	3
C8	4
C9	5
C10	4
C11	1
C12	2
C13	3
C14	2
C15	1
C16	2
C17	2
C18	1
C19	5
C20	5
C21	2

C22	1
C23	1
C24	1
C25	1
D1	3
D2	1
D3	5
D4	5
D5	5
D6	2
D7	1
D8	2
D9	5
D10	5
D11	1
D12	5
D13	5
D14	2
D15	4
D16	4
D17	5
D18	5
D19	5
D20	5
D21	2
D22	1
D23	5
D24	2
D25	4

Table 12.4 Raw data of the Adhesive Remnant Index for the second debond

Sample	ARI
A1	1
A2	2
A3	3
A4	1
A5	1
A6	4
A7	1
A8	5
A9	5
A10	5
A11	1
A12	1
A13	1
A14	2

A15	1
A16	1
A17	4
A18	1
A19	5
A20	1
A21	5
A22	1
A23	1
A24	1
A25	1
B1	5
B2	5
B3	5
B4	5
B5	5
B6	5
B7	5
B8	1
B9	5
B10	2
B11	3
B12	5
B13	5
B14	5
B15	5
B16	5
B17	5
B18	5
B19	5
B20	5
B21	5
B22	5
B23	5
B24	5
B25	5
C1	3
C2	5
C3	5
C4	5
C5	1
C6	1
C7	5
C8	5
C9	1
C10	5
C11	5
C12	5

C13	5
C14	5
C15	1
C16	1
C17	5
C18	5
C19	5
C20	1
C21	5
C22	1
C23	1
C24	5
C25	3
D1	5
D2	5
D3	5
D4	5
D5	5
D6	5
D7	5
D8	5
D9	5
D10	5
D11	5
D12	5
D13	5
D14	5
D15	5
D16	5
D17	5
D18	5
D19	5
D20	5
D21	5
D22	5
D23	5
D24	5
D25	5
