COMPOSITE BOND STRENGTH OF ONE AND TWO - STEP ADHESIVE SYSTEMS USED FOR RESTORATIONS ON PRIMARY TEETH USING VARYING ACID ETCH APPLICATION TIMES

By

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ABSTRACT

The objective of this study was to determine whether the composite bond strength acquired using a single step self-etching bonding system is as or more effective when compared to the two step etch-rinse-bond system at clinically relevant in-vitro standards.

A total of 80 extracted human deciduous with at least one intact smooth surface were collected with parental consent. Collected teeth were randomly assigned to one of four treatment protocols (n=20): single step self-etch (Adper Prompt L-Pop, 3M ESPE), two step etch (15s)-rinse-bond (Optibond Solo Plus, Kerr), single step self etch (Adper L Pop, 3M ESPE) with 30s pre-etch, and two step etch (30s)-rinse-bond (Optibond Solo Plus, Kerr). A composite cylinder of uniform shape was bonded to each tooth using the designated adhesive system and specimens were tested with a universal testing machine. Descriptive statistics and statistical analysis was performed.

Median shear bond strengths for all specimens, regardless of treatment group, ranged from 3.25 MPa to 43.13 MPa. These values are above suggested minimum clinical required values of 3MPa. A Kruskal-Wallis analysis between treatment groups found statistical significance ($p<0.05$) for shear bond strength between the groups and both one step system protocols, had higher mean and median shear bond strength values than the two step systems.

An adhesive remnant index (ARI) was analyzed to confirm uniform adhesive fracture between all four treatment groups. Both a Fisher's exact test, and a Hantel-Haenszel statistic found no significant difference ($p<0.05$) between the samples.

Based on these findings we concluded that:

1. The ARI score for all treatment groups was not statistically significant, suggesting that the type of bond breakage was consistent regardless of adhesive system used.

2. Both Optibond Solo Plus and Adper Prompt L-Pop systems achieve adequate shear bond strengths as described by current literature.

3. The use of a one-step system to save chair time and aid in patient behavior management may be clinically useful.

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1 INTRODUCTION

In the modern day of adhesive dentistry, the availability of new materials with properties such as simplified techniques, improved adhesively to tooth structure, reduced number of applications, and reduced chair time, have made it advantageous for pediatric dentists to consider the benefit of these properties to his or her practice (Fritz & Finger, 1999). Specifically, these advances may save restorative procedure time and ultimately aid in pediatric dental patient management (Gonzales, Rich, Finkelman, & Dufuria, 2012). As the American Academy of Pediatric Dentistry has recommended the use of additional sedation methods such as nitrous oxide for lengthy procedures (American Academy on Pediatric Dentistry Council on Clinical Affairs, 2008), the use of dental materials which reduce chair-time may help negate the risk of uncooperative behavior during chairside treatment (Krifka, Borzsonyi, Koch, Hiller, Schmalz, & Friedl, 2008) and possibly limit the need for advanced and potentially risky patient management techniques.

The success of adhesive systems in dentistry has been found to be influenced by factors such as mineral hardness of the bonding substrate, namely enamel or dentin (Krifka *et al*, 2008). There is limited literature on adhesive bonding to the deciduous dentition compared to the permanent dentition, and extending clinical trends from the permanent dentition to the primary dentition tooth structure may be inaccurate due to the differences in morphology, structural and chemical differences in tooth composition between primary and permanent teeth (Johnsen, 2002). These anatomical differences may change the clinical qualities of adhesivity.

The availability of virgin deciduous human teeth for in-vitro study has also limited the ability to extrapolate and compare dental materials data from the permanent to deciduous dentition. Many deciduous teeth that are available for laboratory studies are compromised (demineralized) and have been collected due to caries, periapical abcess or non-restorability. Studies exist on fluorosed permanent teeth and these studies have found difficulty in producing a reliable bond to enamel due to the difficulty in achieving an adequate etched enamel surface (Noble, Karaiskos, & Wiltshire, 2008).

Although it could be argued that because the longevity of the deciduous dentition is less than the permanent dentition, and therefore the length expectation of a restoration could be less, any premature restoration replacement could negatively affect behavioral patient management (Krifta et al, 2008) and is often met with both patient and parent disappointment.

Two general adhesive systems currently on the market are one-step and two step systems (Kugel & Ferrari, 2000). It has been suggested that one step systems would be advantageous to the pediatric dentist in order to reduce chairside time (Fritz & Finger, 1999) and prevent saliva contamination. This in-vitro study will investigate whether or not the composite shear bond strength acquired for a one-step self-etching system to enamel in the primary dentition produces equivalent bond strengths compared to a two step etch-rinse-bond system to enamel in the deciduous dentition. Specifically, the shear bond strengths to enamel of Adper Prompt L-Pop (3M ESPE) and Optibond Solo Plus (Kerr) will be evaluated in order to assess whether the one-step system has equivalent bond strengths when using manufacturers' directions for clinical use, in an in vitro situation.

Literature for Adper Prompt L-Pop on shear bond strength to dental hard tissues has shown varying results (Horiuchi et al, 2004; Re. et al, 2004). The two step system (Optibond Solo Plus) was chosen as it is a very well used system currently in clinical use at the largest pediatric dentistry practice in Manitoba.

2 LITERATURE REVIEW

2.1 Evolution of Bonding

Bonding was first introduced to the field of dentistry in 1955 by Buonocore through the introduction of the acid etch technique, thereby allowing the bonding of acrylic dental materials to dental hard tissues (Buonocore, 1955). This was shortly followed by Bowen in 1956 when the basic material of adhesive dentistry, Bis-GMA was introduced (Bowen, 1956). Since that time, there have been several generations of dental adhesive systems introduced, with today's clinical pediatric practice incorporating either the one-step or two-step adhesive systems. The main difference between the one-step and two-step adhesive systems is that the one step adhesive system combines the acid etch component into the other components of the adhesive material (Ozeo-Ishida, Endo & Shimooka, 2010; Rossouw, 2010).

2.2 Enamel Differences in Primary and Permanent Teeth

There are several differences in the enamel surface of primary versus permanent teeth. The enamel of primary teeth is mainly prismless as compared to permanent teeth (Ripa, Gwinnett & Buonocore, 1966). The ratio of organic to inorganic content is higher in the primary dentition. (Bird, French, Woodside, Morrison & Hodge, 1940). As well, the chemical content and mineralization of enamel differ between different teeth in the arch (Sabel, Robertson, Nietzsche & Noren, 2012). With respect to the primary dentition, the degree of enamel porosity may be important to the demineralization and tendency of dissolution in primary enamel (Shellis, 1984; Wang, Tang, Bonstein, Bush & Nancollas, 2006). It is these aspects of the primary dentition which result in poorly developed etching patterns and cause relatively lower bonding strength as compared to permanent teeth (Ozeo-Ishida et al, 2010).

2.3 Shear Bond Testing

Bond strength of a dental adhesive is crucial to the success of many restorative procedures. One method of evaluating the strength of an adhesive bond is to test the adhesive material *in vitro*. The samples can be subjected to either shear bond strength, tensile strength or torsional strength tests using a universal testing machine (Katona, 1994; Powers et al, 1997)

The testing for shear bond strength includes applying a load to the substrate parallel to the external tooth surface. This method is possible in theory; however, due to difficulties in laboratory protocol, the true force is often a combination of tension, torsion and peeling forces. In essence, the measurement is often a shear-peel bond strength. (Katona, 1994; Ho, Akyalcin, Bonstein, & Wiltshire, 2011)

Other factors that may influence the true shear bond strength include the *in vitro* placement of the dental adhesive. Often, the adhesive is applied over the entire tooth surface thereby increasing the bonding area and subsequently creating unequal forces which cause the shear stress to be mainly at the adhesive-composite interface rather than adhesive-tooth interface (Van Meerbeek, Peumans, Poitevin, Mine, Van Ende, Neves & DeMunck, 2010). This finding was reinforced by Van Meerbeek et al. who also found several studies which agreed that the shear stress is not equally distributed and not usually focused at the true interface (DeHoff, Anusavice, & Wang, 1995; Van Noort, Noroozi, Howard & Cardew, 1989; Pashley, Sano, Ciucchi, Yoshiyama, & Carvalho, 1995; Versluis, Tantbirojn, & Douglas, 1997; Sudsangiam & Van Noort, 1999). Regardless of these discrepancies, it has been suggested that shear bond strength testing remain a popular method to screen adhesive materials (Van Meerbeek et al, 2011).

It is important to note which type of bond fracture occurs for each specimen; namely, cohesive or adhesive. A cohesive fracture occurs within a material itself, for instance, the composite or the tooth structure. An adhesive fracture occurs at the interface of the adhesive-composite or adhesive-tooth. Krifka et al. suggested a dynamic classification of fracture types taking into account the degree of variability and possible combinations of fracture modes for any one specimen (Krifka, Borzonyi, Koch, Hiller, Schmalz & Friedl, 2008).

Due to this variability in fracture type of specimens, an Adhesive Remnant Index (ARI) can be used to evaluate the consistency of bond fracture between treatment groups. (Powers et al, 1997). This method has been commonly employed in orthodontic shear bond testing of adhesives to bonded brackets

2.3.1 Minimum Recommended Bond Strength

There is limited literature on the minimum acceptable shear bond strength of an adhesive to primary dental enamel. A minimum recommended bond strength for primary dental enamel would be useful to the clinical relevance when comparing different dental adhesive systems. Provided a material meets the clinical benchmark, it would be fair to extrapolate that the product is clinically acceptable. In orthodontics, there have been several studies suggesting the minimum shear bond strength of an adhesive for orthodontic bracket bonding. A minimum shear bond strength of 6-8 MPa was suggested by Reynolds to be clinically relevant

(Reynolds, 1975). Alternatively, an *in vitro* "ideal bond strength" of 3-4 MPa has been suggested as a clinical minimum when bonding orthodontic brackets (Fricker, 1994; Fricker, 1998; Wiltshire & Nobel, 2010). A recent study by Ozoe-Ishida et al, found shear bond strength of orthodontic brackets bonded on primary teeth using 3 different primers to be in the range of 5.38-13.15 MPa collectively for teeth that were tested without moisture contamination (Ozoe-Ishida, Endo, & Shimooka, 2010).

2.4 Adhesive Systems

2.4.1 Two Step Adhesive Systems versus One Step Adhesive Systems

The two step adhesive systems combine an acid etch enamel conditioner followed with primer adhesive. The enamel conditioner, commonly 37% phosphoric acid, is applied to the tooth surface to create microscopic spaces in the enamel surface and increasing surface roughness, thereby allowing greater penetration of the dental bonding agent or primer. This conditioning step is followed by placement of the adhesive primer which flows freely into the newly roughened enamel surface, increasing surface area, and aiding in micromechanical retention through impregnation of resin tags into the enamel. In 2010, Van Meerbeek et al., described the micro-mechanical interlocking of resin tags to phosphoric acid-etched enamel as the best achievable bond to enamel (Van Meerbeek et al., 2010). Conversely, the one step adhesive systems utilize a self-etching primer. This product combines the acid etching, primer and adhesive into one clinical step (Ozoe-Ishida et al, 2010). Due to the incorporation of all materials into a single system, there has been a concern that these newer systems do not provide the same degree of enamel porosity and resin penetration as traditional systems; although varying results of significance exist between the systems in the literature (Ozoe-Ishida et al, 2010; Van Meerbeek et al, 2010).

The advantages to the one step system include reduced restorative chairtime, reduced post-op sensitivity, and ease of use (Kimmens, Barkmeier, Erickson & Latta, 2010). The reduced chairside time may be of utmost importance to the Pediatric Dentist with respect to behavior management of the pediatric dental patient (Krifka et al, 2008).

2.4.2 Adper Prompt L-Pop (3M ESPE) (One Step)

The Adper Prompt L-Pop (3M ESPE) is a self etching adhesive used in restorative dentistry. No current studies are available on the shear bond strength of the product to deciduous enamel structure. As a result, the following table represents a summary of findings for shear bond strength of Adper Prompt L-Pop adhesive on permanent tooth enamel.

Table 2.4.2 – Comparison of Shear Bond Strengths using Adper L Pop on Permanent Human Tooth Enamel *In Vitro*

Author	SBS (MPa)	N	Enamel	Storage Medium	Crosshead Speed	Application Time	Test Condition
Nagayassu et al.	22.17+/- 6.05	12	flat ground	Distilled water, 24 hours	0.5mm/min	Manf. Directions	Resin Composite
Kimmes et al.	30.07+/- 4.3	40	flat ground	Distilled water, 24 hours	1mm/min	Manf. Directions	Resin Composite
Pivetta et al.	21.5 +/- 3.3	6	Unground	Water, 24 Hours	0.5mm/min	Manf. Directions	Resin Composite

2.4.3 Optibond Solo Plus (Kerr) (Two Step)

Optibond Solo Plus is a dental adhesive which utilizes an acid etch conditioning step prior to placement. No current studies are available on the shear bond strength of the product to deciduous enamel structure. As a result, the following table represents a summary of findings for shear bond strength of Optibond adhesive on permanent tooth enamel.

Table 2.4.3 – Comparison of Shear Bond Strengths using Optibond Solo Plus on Permanent Human Tooth Enamel *In Vitro*

Author	SBS (MPa)	N	Enamel	Storage Medium	Crosshead Speed	Application Time	Test Condition
Kimmes et al.	35.2+/-6.2	40	flat ground	Distilled water, 24 hours	1mm/min	Manf. Directions	Resin Composite
Pivetta et al.	23.3+/-2.0	6	unground	Water, 24 Hours	0.5mm/min	Manf. Directions	Resin Composite
Lopes et al.	16.4+/-3.9	10	flat ground	Distilled water, 24 hours, thermocycled	5mm/min	Manf. Directions	Resin Composite

2.4.4 Differences in Shear Bond Strength of the Two Adhesives in this Study

It must be noted that the studies by Kimmes et al., and Pivetta et al., both compared the shear bond strength of Adper Prompt L-Pop and Optibond Solo Plus to permanent tooth enamel using similar protocols with conflicting results. Pivetta et al. found the mean bond strengths of Adper Prompt L-Pop and Optibond Solo Plus to be statistically similar ($p>0.641$) (Pivetta et al, 2008). Alternatively, Kimmes et al. found that the mean shear bond strengths for the etch-and-rinse systems produced higher bond strengths than the self etch systems ($p<0.05$) (Kimmes et al., 2010).

3 PURPOSE

The purpose of this study was to determine the shear bond strength to primary enamel of two different adhesive systems, namely a self etch and a total etch (etch-rinse-adhesive) system, and consider whether the systems differ at clinically relevant markers. The chairside time saved using the self etch could aid in the behavior management of pediatric dental patients.

4 NULL HYPOTHESES

1. There is no difference in shear bond strength to enamel between any of the treatment groups.
2. There is no difference in the scores of the Adhesive Remnant Index between the treatment groups.

5 MATERIALS AND METHODS

5.1 Materials Used In The Study

5.1.1 Adper™ Prompt™ L-Pop™ Self Etch Adhesive (3M ESPE)

The Adper™ Prompt™ L-Pop™ is a seventh (7th) generation self etching adhesive system. The system has two separate materials compartments (A & B) in a unidose applicator which are mixed together immediately prior to use. Part A contains 2-PROPENOIC ACID, 2-METHYL-,PHOSPHINICOBIS (OXY-2,1-ETHANDIYL)ESTER [20-25%wt], Mono HEMA phosphate [15-30%wt], METHACRYLATED PYROPHOSPHATES [15-30%wt], BISPHENOL A DIGLYCIDYL ETHER DIMETHACRYLATE (BisGMA) [10-15%wt], TRIS[2-(METHACRYLOYLOXY)ETHYL]PHOSPHATE [1-10%wt] and less than 2%wt of: ETHYLENE DIMETHACRYLATE, PHOSPHORIC ACID, BIS (2,6-DICHLOROBENZOYL) BUTYLPHENYL PHOSPHINE OXIDE, 2-HYDROXYETHYL METHACRYLATE, 4-METHOXYPHENOL, HYDROQUINONE, and ETHYL 4-DIMETHYL AMINOBENZOATE. Part B contains water (78-80%wt), 2-HYDROXYETHYL METHACRYLATE (20-30%wt), and less than 2% of 2-PROPENOIC ACID, POLYMER WITH METHYLENEBUTANEDIOIC ACID. (MSDS: 3M, 2011)

Figure: 5.1 Adper Prompt L-Pop

5.1.2 Optibond Solo Plus Adhesive (Kerr)

Optibond Solo Plus is a dental adhesive system composed of ethyl alcohol (20-25%], alkyl dimethacrylate resins [55-60%], barium aluminoborosilicate glass [5-10%], fumed silica (silicon dioxide) [5-10%], and sodium hexafluorosilicate [0.5-1%]. (MSDS: Kerr Corporation, 2009)

Figure 5.2 Optibond Solo Plus

5.1.3 TPH3 Micro Matrix Restorative (Dentsply)

TPH 3 Micro Matrix Restorative Material is a universal composite material that contains barium boron alumino silicate glass (< 50%), barium boron fluoro alumino silicate glass (<30%), urethane modified Bis-GMA dimethacrylate (<10%), polymerizable dimethacrylate resin (<10%), and less than 3% silica (amphorous), hydrophobic amphorous fumed silica, and titanium dioxide. (MSDS: Dentsply Caulk, 2010).

Figure 5.3 TPH 3 Micro Matrix Restorative

TABLE 5.1: Materials Used In This Study

Material	Manufacturer	Lot
Sample Preparation		
Copper Rings		
Petroleum Jelly (Vaseline)	Chesebrough-Pond's, USA	
Bosworth Fastray (Custom Tray and acrylic base plate material)	Bosworth, USA	D6810921389OF
-Monomer Liquid		1207-323
-Monomer Powder		1207-323
558 Carbide Burs	Beavers Dental, Morrisburg, ON	2530457
Sample Surface Preparation		
34% Phosphoric Acid Etch	Pulpdent Corporation, Watertown, MA	130830

Pumice		
Used in Bonding		
Adper Prompt L-Pop	3M ESPE, St. Paul, MN	521911
Optibond Solo Plus	Kerr Corporation, Orange, CA	4950941
TPH Composite	Dentsply Caulk, Milford, DE	1306283
Debonding Equipment		
Universal Testing Machine	Zwick GmBH, Ulm, Germany	
Bencor Multi-T Testing apparatus	Danville Engineering, San Ramon CA	
Other		
Crown and Bridge Caliper		
Baseplate Wax		
LED Light	Caulk, Dentsply, Milford, DE	Serial: 58457
Incubator 37C	Thelco/Canlab Model 2, Precision Scientific, Chigaco, IL	
Composite Plugger #1A	American Dental Manufacturing Company	
Plastic Containers	Ziploc, Gladware	

5.2 Experimental Method

5.2.1 Ethics Approval

Ethics Approval was obtained from the University of Manitoba Human Research Ethics Board (See Appendix).

5.2.2 Tooth Collection

A total of 80 extracted human primary molar teeth were collected from the University of Manitoba Graduate Pediatric Dentistry Clinic and other Pediatric Dental specialist offices in the City of Winnipeg. The teeth collected were extracted for purposes other than this study (such as normal exfoliation, pathologic root resorption, over retention and serial extraction). Teeth that did not have one smooth surface of clinically intact enamel were excluded.

Extracted teeth were stored in saline at room temperature until investigation.

5.2.3 Sample Preparation and Storage

5.2.3.1 *General Preparation*

The 80 enamel tooth samples were cleaned with distilled water and any remaining roots sectioned using a #558 carbide bur below the cemento-enamel junction prior to being mounted in the acrylic jig. The acrylic jigs were made using copper rings that fit the Bencor Multi T testing apparatus; each copper ring had Vaseline applied prior to being filled with Bosworth Fastray acrylic custom tray material (chemical cure). Teeth were immediately placed in the pre-set acrylic with the most flat surface parallel to the level surface of the copper jigs. This surface was confirmed as the sample was rotated 360 degrees. The samples were removed from the copper rings once the acrylic hardened, and the samples were stored in distilled water at 37C for 24 hours in an incubator until the designated adhesive system was applied.

After 24 hours, the eighty (80) samples were randomly divided into four groups of twenty (20), which would be treated with the following adhesive system:

1) Group 1 – Optibond Solo Plus with 15s 37% phosphoric acid etch

2) Group 2 – Optibond Solo Plus with 30s 37% phosphoric acid etch

3) Group 3 – Adper Prompt L-Pop alone

4) Group 4 – Adper Prompt L-Pop combined with 30s etch

Prior to bonding of the adhesive and composite, all 80 sample groups had the enamel surface cleaned with a pumice paste and water.

5.2.3.2 *Adhesive System Placement by Treatment Group*

<u>Treatment Group 1 (20 Samples)</u>

The full area of the enamel was prepared for 30s with 37% phosphoric acid, rinsed with distilled water and lightly air dried. A thin layer of Optibond Solo Plus adhesive was applied to all enamel according to manufacture's directions. As per manufacture's direction, once application was complete each sample was light cured for 20s using an LED curing light which had been tested for accuracy prior to all samples being investigated.

<u>Treatment Group 2 (20 Samples)</u>

The full area of the enamel was prepared for 15s with 37% phosphoric acid, rinsed with distilled water and lightly air dried. A thin layer of Optibond Solo Plus adhesive was applied to all enamel according to manufacture's directions. As per manufacture's direction, once application was complete each sample was light cured for 20s using an LED curing light which had been tested for accuracy prior to all samples being investigated.

Treatment Group 3 (20 Samples)

A thin layer of Adper Prompt L-Pop adhesive was applied to all enamel according to manufacture's directions. As per manufacture's directions, once application was complete each sample was light cured for 10s using an LED curing light which had been tested for accuracy prior to all samples being investigated.

Treatment Group 4 (20 Samples)

The full area of the enamel was prepared for 15s with 37% phosphoric acid, rinsed with distilled water and lightly air dried. A thin layer of Adper Prompt L-Pop adhesive was applied to all enamel according to manufacture's directions. As per manufacture's direction, once application was complete each sample was light cured for 10s using an LED curing light which had been tested for accuracy prior to all samples being investigated.

5.2.3.3 *Composite Block Placement*

Once the adhesive was cured, a cylindrical 2.71mm in diameter piece of composite measuring approximately 2mm in length was bonded to the enamel surface using a wax jig. The wax jig was created using a 2.71mm in diameter composite plugger to punch a hole in baseplate wax of thickness approximately 2mm. The composite sample was lightly packed, and cured for 20s according to manufacturer's directions.

5.2.4 Storage Conditions

All samples were stored in distilled water for 24 hours in an incubator at 100% relative humidity at 37C until shear peel bond testing could be performed.

5.2.5 Shear Bond Strength Testing

24 hours after the bonding procedure was completed, each sample was loaded into the Bencor Multi-T testing apparatus. The parameters on the Zwick universal testing machine were a 10kN load cell with a crosshead speed of 0.5mm per minute. The attachment apparatus diameter of 2.71mm was used. The machine values for each sample were recorded on a spreadsheet, and the type of debond was noted. An adhesive Remnant Index (ARI) score for each sample was recorded using visual inspection by 2 investigators working blind.

5.3 Statistical Analysis

Statistical analysis was calculated in collaboration with the University of Manitoba, Biostatistical Consulting Unit. Descriptive statistics and a table of the summarizing the distribution of the scores was completed. As the distribution was considered non-parametric, a Kruskal-Wallis test was used to analyze the results between the 4 groups.

The Adhesive Remnant Index (ARI) scores were evaluated using two statistics. The first was the Fisher's exact test, which looks for any general difference between groups with respect to the distribution of the sample in each ARI category. The second is a generalized Hantel-Haenszel test, which takes into account the ordinal nature of the column (ARI). P-values were considered significant at $P<0.05$ for all tests completed.

6 RESULTS

Prior to the testing of the 80 specimens, a pilot trial of the methodology was performed to ensure the experimental design was appropriate and could produce consistent results.

6.1 Shear Bond Strength (SBS) Descriptive Statistics

It was noted (Figure 6.1) that the distribution of the shear bond strength between the four experimental groups showed similar minimum and maximum values, with the median values between 15.45 MPa (low, Optibond Solo Plus with 30s etch) and 18.045 MPa (high, Adper Prompt L-Pop self etch). Notably, the standard deviations were also similar between the tested groups indicating similar variations between the shear bond strengths of the tested adhesives (see Figure 6.1 and Table 6.1).

Figure 6.1 – Graph of Shear Bond Strengths of the different adhesives

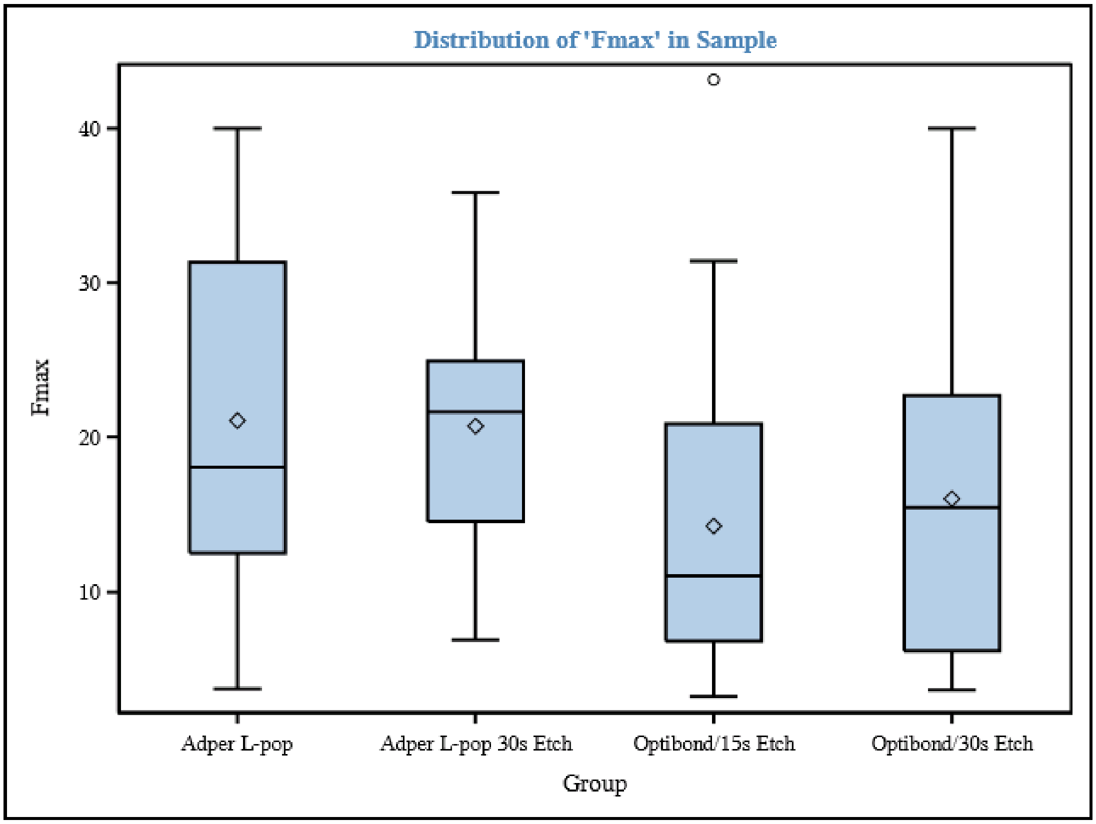

Table 6.1 – Tabulated Data of Shear Bond Strengths of the different adhesives

Group	N	Mean (MPa)	Median (MPa)	Minimum (MPa)	Maximum (MPa)	Std Dev (MPa)	C.V. (%)
Adper Prompt L-Pop, Self Etch	20	21.111	18.045	3.750	39.990	11.127	52.70
Adper Prompt L-Pop, 30s Etch	20	20.725	21.630	6.930	35.830	8.243	39.77
Optibond, 15s Etch	20	14.296	11.035	3.250	43.130	10.334	72.28
Optibond, 30s Etch	20	16.048	15.475	3.69	39.94	9.961	62.07

Table 6.1 shows that Adper Prompt L-Pop exhibited the highest shear bond strength between the four groups (21.11 MPa), followed by Adper Prompt L-Pop with 30s etch (20.72 MPa). However, the median value was higher with a 30s etch, compared to no etching (21.63 MPa vs. 18.05 MPa). In addition, when etching for 30s, Adper Prompt L-Pop produced the highest minimum values (6.93 MPa vs. 3.75 MPa). Optibond with either a 15s or 30s etch produced lower mean, median and lowest minimum values. Interestingly, the highest shear bond strength obtained was with Optibond and a 15s etch (43.13MPa). The standard deviations were all in the same order of magnitude (9.96MPa to 11.13MPa). The coefficients of variation in this study were all very high and significantly above the 15-30% sought in shear bond strength studies. Of note, is that Adper Prompt L-Pop with the addition of 30s etch, achieved not only the highest mean and median shear bond strength values, the highest maximum shear bond strength value, the highest minimum shear bond strength, but Adper Prompt L-Pop with 30s etch also had the lowest coefficient of variation (39.77%) suggesting it is the most reliable bonding method within all the evaluation parameters.

6.2 Kruskal-Wallis Test of SBS

Given the non-parametric pattern of the data, a Kruskal-Wallis test was used to compare the shear bond strengths between the sample groups.

Table 6.2.1: Kruskal-Wallis Test Analysis of all 4 treatment groups

Kruskal-Wallis Test	
Chi-Square	8.2233
DF	3
Pr > Chi-Square	0.0416

Table 6.2.2: Kruskal Wallis Test Analysis between specific treatment groups

Treatment Groups Compared	Statistical Significance
Adper Prompt L-Pop (self etch) vs. Adper Prompt L-Pop with 30s Etch	p=1.00 (p>0.05)
Optibond Solo Plus 15s Etch vs. Optibond Solo Plus 30s Etch	p=0.402 (p>0.05)
Adper Prompt L-Pop Combined vs. Optibond Solo Plus Combined	p=0.0059 (p<0.05)

The initial Kruskal-Wallis test (see Table 6.2.1) showed a statistically significant difference (p<0.05) between individual treatment groups. Further analysis was performed to evaluate the significance between specific groups (see Table 6.2.2). It was found that there was no significant difference comparing the etching time between either product (Adper Prompt L-Pop p=1.00, Optibond Solo Plus p=0.402). There was a significant difference in shear bond strength values when comparing all combined Adper Prompt L-Pop test specimens against all combined Optibond Solo Plus test specimens (p=0.0059).

6.3 Adhesive Remnant Index (ARI)

All specimens were evaluated for their Adhesive Remnant Index (see Table 6.3.1 and Table 6.3.2). The adhesive remnant index was compared between treatment groups using two statistics. The treatment groups were analyzed using a Fisher's exact test, which compares the general difference in the distribution of the ARI scores for each sample group, and with Hantel-Haenszel test which takes into account the ordinal nature of the ARI. Both tests found no statistical significance between the treatment groups for ARI (see Table 6.3.3 and Table 6.3.4). The majority of the samples ARI values were 1 or 2 equating to 75-95% of the total samples. Adper Prompt L-Pop was the only sample were 10% of the samples were an ARI of 4.

Table 6.3.1 – ARI Index Key

ARI	Description
1	Clear – No composite left on tooth
2	Up to ½ composite left on tooth
3	More than ½ composite left on tooth
4	100% of composite left on tooth

Table 6.3.2 – Distribution of ARI scores as utilized for statistical analysis in Table 6.3.3 and Table 6.3.4

Frequency Row Pct

Table of Group by ARI					
	ARI				
Group	1	2	3	4	Total
Adper L-pop	6 30.00	9 45.00	3 15.00	2 10.00	20
Adper L-pop 30s Etch	5 25.00	10 50.00	4 20.00	1 5.00	20
Optibond/15s Etch	10 50.00	8 40.00	1 5.00	1 5.00	20
Optibond/30s Etch	7 35.00	11 55.00	1 5.00	1 5.00	20
Total	28	38	9	5	80

Table 6.3.3 – Fisher's Exact Test Results

Fisher's Exact Test	
Table Probability (P)	3.524E-06
Pr <= P	0.7629

Table 6.3.4 – Hantel-Haenszel Test Results

Cochran-Mantel-Haenszel Statistics (Based on Table Scores)				
Statistic	Alternative Hypothesis	DF	Value	Prob
1	Nonzero Correlation	1	1.8661	0.1719
2	Row Mean Scores Differ	3	3.2983	0.3479
3	General Association	9	6.0497	0.7349

7 DISCUSSION

The introduction of new restorative materials in adhesive dentistry is of great importance to the Pediatric Dentist. Any material that may provide a simplified technique, reduced number of applications, or reduced chair-time, may directly aid in the behavioral management of the pediatric dentistry patient. The new 'one-step' adhesive systems, such as the Adper Prompt L-Pop (3M ESPE), may aid in this process. This study investigated the shear bond strength of two restorative adhesives; namely, a two-step adhesive, Optibond Solo Plus (Kerr), and a one-step adhesive, Adper Prompt L-Pop (3M ESPE) on smooth surface enamel of deciduous teeth with minimal surface preparation. The main finding of this study is that the one-step adhesive system performed equally to, and even out performed, the two step adhesive system at clinically relevant markers.

7.1 Shear Bond Strength

The findings in this study were difficult to compare to the existing literature as there are limited studies with respect to the shear bond strengths to enamel of deciduous teeth. A recent study evaluating the SBS of a one-step adhesive system (Adper Easy Bond, 3M ESPE) on permanent and deciduous enamel, with or without acid etching, found that the shear bond strength to deciduous enamel was statistically significantly less than to permanent enamel ($p<.03$), and the use of acid etching prior to single step adhesive placement improved the shear bond strength ($p<0.001$) (Lenzi, Guglielmi, Umakoshi, & Raggio, 2014). This positive trend suggesting the use of pre-conditioning with acid etch prior to single-step adhesive placement were found in several other studies (Miguez, Castro, Nunes, Walter, & Pereira, 2003; Van Landuyt, Kanumilli, De Munck, Peumans, Lambrechts, & Van Meerbeek, 2006; Watanabe, Tsubota, Takamizawa, Kurokawa, Rikuta, Ando & Miyazaki, 2008). This finding of improved shear bond strength through the use of acid etching prior to single step adhesive placement is coincident with the results of the current study.

This finding may be due to the process of enamel bonding; namely the property that enamel bonding is largely impacted by the degree of mechanical interlocking of the adhesive resin into the enamel, which is impacted by the acidity of the conditioning or acid etching system (Pediagao, Lopes, Lambrechts, Leiteo, Van Meerbeek, & Vanherlee, 1997).

In terms of clinical application, there were no studies found in the literature that directly describe the minimum required shear bond strength of adhesive systems to deciduous human enamel. A minimum shear bond strength of 6-8 MPa was suggested by Reynolds to be clinically relevant when bonding orthodontic attachments to enamel (Reynolds, 1975). Alternatively, an *in vitro* “ideal bond strength” of 3-4 MPa has been suggested as a clinical minimum based on bonding orthodontic brackets with glass ionomer to enamel (Wiltshire & Nobel, 2010). Using this range (3-8MPa) as a baseline, it could be suggested that all 4 bonding systems used in this study preformed within the clinically acceptable *in vitro* range. The minimum shear bond strength values in all 4 bonding systems were above the minimum suggested shear bond strength value of 3 MPa reported by Wiltshire and Nobel (2000). Using this deduction, it could be considered that the clinician may choose any of the 4 bonding system protocols described in this study to achieve clinically acceptable results. One may then propose that the Pediatric Dentist should choose the system with the least amount of chairside time to aid in the patient’s behavior management. In this study, the shortest working time protocol of adhesive system was the Adper Prompt L-Pop alone.

It must also be noted that there was no significant difference in shear bond strength values of either the Adper Prompt L-Pop or Optibond Solo Plus products when changes to etching protocol were completed. This suggests that additional etching may not be required to provide superior results with respect to either product alone. There was, however, a significance difference in shear bond strength between the combined Adper Prompt-L Pop and combined Optibond Solo Plus treatment groups. This could imply that the Adper Prompt L-Pop was superior to the Optibond Solo Plus product using the protocol of this study; nonetheless, further investigation will be needed as both products meet the minimum required shear bond strength as described by Wiltshire and Nobel (2000).

Due to a limitation in the number of samples that could be gathered for this study, 'in-tact' deciduous enamel was considered an inclusion criteria for sample collection. Samples were gathered from teeth which were extracted for reasons such as caries, minimal restorability or periapical abcess. As a result, the enamel tested in this study may have been minimally compromised with respect to enamel integrity leading to a concern with respect to bonding quality. In a recent publication describing the bonding considerations in orthodontics to normal, hypoplastic and fluorosed enamel by Wiltshire and Nobel, *in vitro* studies were identified which found no statistical significance between the shear bond strength of fluorosed or normal teeth ($p>0.05$). The article also cautioned against simply interpreting the mean shear bond strength values of study groups; rather, a much broader sense of interpretation must be utilized (Wiltshire & Nobel, 2010).

Taking this into account, the statistical analysis results using the Kruskal-Wallis test suggest that there is a statistically significant difference between treatment groups. This would suggest that the two Adper Prompt L-Pop groups (median 18.045, 21.63) preformed better than the Optibond Solo Plus groups (median 11.03, 15.475). However, the clinical relevance must be taken into account. If one accepts the minimum clinical shear bond strength to be 3-4 MPa as described by Wiltshire and Noble, then all 4 treatment groups preformed adequately based on their respective minimum shear bond strength of any one sample. As a result, the statistical significance of the Kruskal-Wallis test may not be of clinical relevance.

The range between minimum and maximum shear bond strength values for all four treatment groups is large. It is noted that there is a relative difference between the Optibond Solo Plus and Adper Prompt L-Pop treatment groups. This may be due to the aforementioned differences in enamel integrity, the particular properties of primary tooth enamel, or it may be due to the randomization of teeth in each treatment group. Hobson, McCabe & Hogg found that tooth type had a significant effect on bond strength ($p<0.001$). Differences in bond strength were also found when teeth were analyzed by adult tooth type (Hobson, McCabe & Hogg, 2001). As deciduous teeth have a greater crest of contour than permanent teeth, it would be fair to extrapolate that the bond quality may have greater variability using deciduous teeth; notwithstanding this data, the current study utilized randomization of teeth to treatment groups. In order to verify that there was no statistical difference in the type of bond breakage between all four treatment groups, an Adhesive Remnants Index (ARI) was evaluated for all samples. The four treatments groups were evaluated against each other, and no statistical difference was found using either the Fisher's exact test ($p>0.05$) or Hantel-Haenszel test ($p>0.05$). This rules out any potential error in type of composite breakage (cohesive vs adhesive) in the samples between the 4 groups, and verifies appropriate randomization.

7.2 Potential Clinical Application and Performance

The purpose of this study was to evaluate the shear bond strength of a one-step and a two-step adhesive bonding system on the deciduous dentition in order to evaluate the possibility of shifting clinical practice to a one-step adhesive system in order to save chairside time and improve patient management of the pediatric patient. Given the results discussed above, it would be fair to assume that both the Optibond Solo Plus and Adper Prompt L-Pop systems performed within the clinically acceptable *in vitro* minimum as described by Wiltshire and Nobel. As a result, the clinician may choose to opt for the one-step adhesive system to limit chairside time for appropriate clinical cases of the deciduous dentition. The clinician may also recognize that the use of pre-adhesive conditioning of the enamel for 30s with 37% phosphoric acid prior to placement of the one-step system will increase the shear bond strength. Not factoring the potential price difference of the product systems, a clinician may opt to carry the one-step adhesive system in the office and utilize it with a pre-enamel etching for the majority of cases; selectively eliminating the pre-conditioning step as patient management dictates.

7.3 Limitations of the current study

1. The study was an *in vitro* study. Future tests *in vivo* are highly recommended.
2. This study was performed on smooth surface enamel only. The effect of shear bond strength of one and two step adhesive systems to dentin were not considered.
3. The study did not use artificial saliva as a storage medium. However, it has been suggested that water storage of specimens before testing may increase the predictability of the bonding performance of the tested adhesives (Heintze & Zimmerli, 2011)
4. The present study did not include thermo cycling as this may be too rigorous compared to the clinical reality; samples were stored in water at 37% at 100% relative humidity.

7.4 Null Hypotheses Revisited

After completion of this study, it is concluded that:

1. We reject the null hypothesis that there is no difference in shear bond strength to enamel between any of the treatment groups as there was a statistically significant difference ($p<0.05$) in shear bond strengths between the Adper Prompt L-Pop and Optibond Solo Plus shear bond strengths samples using Kruskal-Wallis statistical analysis.
2. We accept the null hypothesis that there is no difference in the scores of the Adhesive Remnant Index (ARI) between the treatment groups as there were not a statistically significant finding ($p>0.05$) using both a Fisher's Exact and Hantel-Haenszel analysis of ARI distributions.

8 CONCLUSION

Based on this *in vitro* study, the following conclusions can be made:

1. The two products, Adper Prompt L-Pop and Optibond Solo Plus, using various etching times, all produced adequate shear bond strengths to extracted primary tooth enamel using the in-vitro conditions of this study.
2. Most adhesive remained on the composite block after breakage, proposing that enamel breakage would not be expected with either of the two adhesive systems.
3. From a bond strength and reliability perspective, Adper Prompt L-Pop with 30s etch had the highest median, and minimum values.
4. It could be suggested that both the Adper Prompt L-Pop and Optibond Solo Plus are appropriate for clinical use given the findings of this in-vitro study, and that Adper Prompt L-Pop be considered a useful adjunct to save restorative chairtime and aid in patient management of the pediatric dental patient.

RAW DATA					
Optibond/15s Etch			Optibond/30s Etch		
	Fmax.			Fmax.	
Sample	MPa	ARI	Sample	MPa	ARI
1	5.35	1	1	15.29	1
2	9.67	2	2	3.69	1
3	13.08	1	3	25.45	2
4	43.13	2	4	17.63	4
5	15.64	1	5	6.4	1
6	21.54	2	6	30.52	2
7	24.64	2	7	10.83	2
8	8.67	1	8	17.04	1
9	7.54	1	9	4.89	1
10	3.62	1	10	39.94	2
11	22.26	2	11	22.27	2
12	20.25	2	12	12.5	2
13	10.75	3	13	4.23	2
14	3.25	2	14	23.19	3
15	9.11	2	15	17.82	2
16	31.4	4	16	27.77	2
17	6.11	1	17	15.66	2
18	3.67	1	18	6.08	1
19	14.92	1	19	4.55	1
20	11.32	1	20	15.21	2
Adper Prompt L-Pop			Adper Prompt L-Pop, 30s Etch		
	Fmax.			Fmax.	
Sample	MPa	ARI	Sample	MPa	ARI
1	31.04	2	1	26.17	3
2	32.34	2	2	9.17	1
3	6.06	2	3	19.49	2
4	15.88	2	4	16.04	1
5	19.5	1	5	23.94	2
6	8.07	2	6	22.44	2
7	30.76	1	7	35.83	3
8	38.52	4	8	20.93	2
9	39.99	1	9	6.93	1
10	16.63	1	10	23.81	2
11	34.28	3	11	14.38	2
12	14.41	2	12	13.34	1
13	17.42	3	13	33.74	3
14	16.26	1	14	22.33	4
15	10.17	2	15	16.18	2
16	26.21	2	16	11.04	2
17	3.75	1	17	25.99	3
18	10.65	2	18	22.65	2
19	31.62	4	19	35.37	1
20	18.67	3	20	14.72	2

10 APPENDIX

Health Research Ethics Board Approval

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UNIVERSITY OF MANITOBA | BANNATYNE CAMPUS
Research Ethics Boards

HEALTH RESEARCH ETHICS BOARD (HREB)

CERTIFICATE OF FINAL APPROVAL FOR NEW STUDIES

Delegated Review

PRINCIPAL INVESTIGATOR: Dr. B. Klus	**INSTITUTION/DEPARTMENT:** UofM / Preventative Dental Science	**ETHICS #:** HS15909 (H2012:375)
APPROVAL DATE: December 3, 2012	**EXPIRY DATE:** December 3, 2013	
STUDENT PRINICIPAL INVESTIGATOR SUPERVISOR (if applicable): Dr. W. Wiltshire		

PROTOCOL NUMBER: NA	**PROJECT OR PROTOCOL TITLE:** Composite bond strength of one and two - step adhesive systems for restorations on primary teeth using varying acid etch application times
SPONSORING AGENCIES AND/OR COORDINATING GROUPS: UofM Internal Funds	

Submission Date of Investigator Documents: November 9 and 30, 2012	**HREB Receipt Date of Documents:** November 9 and 30, 2012

THE FOLLOWING ARE APPROVED FOR USE:

Document Name	Version(if applicable)	Date
Protocol:		
Protocol	1	October 19, 2012
Consent and Assent Form(s):		
Consent Form	1	October 19, 2012
Other:		
Recruitment Letter to Pediatric Dentists	2.0	November 30, 2012
Script	1.0	November 30, 2012

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 -

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