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Bond Strength of One and Two - Step Adhesive Systems on Primary Teeth with Varying Acid-Etch Application Times

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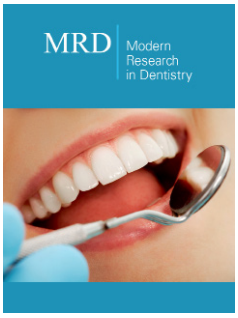

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Abstract

Purpose: To determine whether the composite acquired bond strength using a single step self-etching bonding system is similar compared to a two-step etch-rinse-bond system.

Methods: Eighty extracted human deciduous teeth were randomly assigned to four treatment protocols (n=20): one-step self-etch (Adper Prompt L-Pop, 3M ESPE), two-step etch(15s)-rinse-bond (Optibond Solo Plus, Kerr), one-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 and specimens were tested with a universal testing machine.

Results: Median shear bond strengths for all specimens ranged from 3.25MPa to 43.13MPa. 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 and no statistically significant difference was found ($p<0.05$) between the samples.

Conclusion: 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. 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 of clinical value in pediatric dentistry.

Keywords: Shear bond strength; Dental bonding; Primary teeth; Optibond; Adper prompt

Introduction

In the modern day of adhesive dentistry, the availability of new materials with properties such as simplified techniques, improved adhesivity 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 in practice [1]. Specifically, these advances may save restorative procedure time and ultimately aid in pediatric dental patient management [2]. As the American Academy of Pediatric Dentistry has recommended the use of additional sedation methods such as nitrous oxide for lengthy procedures [3], the use of dental materials which reduce chair-time may help negate the risk of uncooperative behavior during chairside treatment [4] 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 enamel or dentin [4]. 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 [5]. These anatomical differences may affect the clinical qualities of the adhesive.

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. When 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 6-8MPa was suggested by Reynolds to be clinically relevant [6]. Alternatively, an *in vitro* "ideal bond strength" of 3-4MPa has been suggested as a clinical minimum when bonding orthodontic brackets [7-9]. A recent study by Ozoe-Ishida et al. [10] found shear bond strength of orthodontic brackets bonded on primary teeth using 3 different primers to be in the range of 5.38-13.15MPa collectively for teeth that were tested without moisture contamination [10].

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 management4 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 [11]. It has been suggested that one step systems would be advantageous to the pediatric dentist in order to reduce chairside time [1] 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 twostep 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.

Materials & Methods

Ethics approval

Ethics Approval was obtained from the Human Research Ethics Board.

Tooth collection

A total of 80 extracted human primary molar teeth were collected from the University Graduate Pediatric Dentistry Clinic and other Pediatric Dental specialist offices in the city. 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 at least one smooth surface of clinically intact enamel were excluded. Extracted teeth were stored in saline at room temperature until investigated.

Sample preparation and storage

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 flattest 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 37 °C for 24 hours in an incubator at 100% RH until the designated adhesive system was applied. After 24 hours, the eighty (80) samples were randomly divided into four groups of twenty (20), which were treated with the following adhesive systems:

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

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

C. Group 3 – Adper Prompt L-Pop alone

D. Group 4 – Adper Prompt L-Pop combined with 30s phosphoric acid etch

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

Adhesive system placement

The full area of enamel was prepared using the adhesive system specific to the treatment group. Manufacturers' directions for application of the adhesive products were followed. If the protocol required a phosphoric acid etch, the acid etch application was rinsed with distilled water and the tooth was lightly air dried prior to application of the adhesive system. Each sample was light cured for 20s using a LED curing light which had been tested for accuracy prior to all samples being investigated.

Composite block placement

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

Storage conditions

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

Shear bond strength testing

Twenty-four 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

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10kN load cell with a crosshead speed of 0.5mm per minute. The attachment apparatus with a diameter of 2.7mm 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 (Table 1) for each sample was recorded using visual inspection by 2 blinded investigators.

Table 1: ARI 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

Statistical analysis

Statistical analysis was calculated in collaboration with the University, 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: a Fisher's exact test and a Hantel-Haenszel test. P-values were considered significant at $P<0.05$ for all tests completed.

Result

Shear Bond Strength (SBS) Descriptive Statistics

Figure 1 shows the distribution of the Shear Bond Strength (SBS) between the four experimental groups with similar minimum and maximum values, as well as the median values between 15.45MPa (low, Optibond Solo Plus with 30s etch) and 18.045MPa (high, Adper Prompt L-Pop self-etch). Notably, the standard deviations were similar between the tested groups indicating similar variations between the shear bond strengths of the tested adhesives (Figure 1 & Table 2).

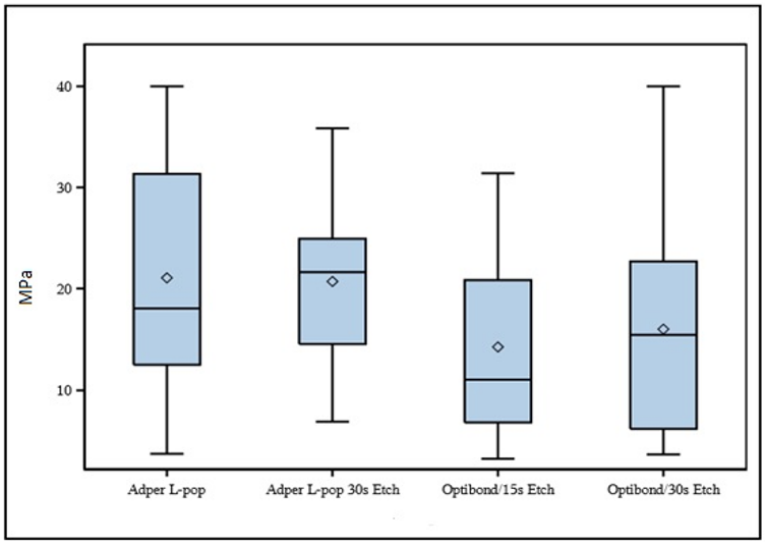

Figure 1: Graph of Shear Bond Strengths of the different adhesives.

Table 2: 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	3.75	39.99	11.127	52.7
Adper Prompt L-Pop, 30s Etch	20	20.725	21.63	6.93	35.83	8.243	39.77
Optibond, 15s Etch	20	14.296	11.035	3.25	43.13	10.334	72.28
Optibond, 30s Etch	20	16.048	15.475	3.69	39.94	9.961	62.07

Table 2 shows that Adper Prompt L-Pop exhibited the highest wear shear bond strength between the four groups (21.11MPa), followed by Adper Prompt L-Pop with 30s etch (20.72MPa). However, the median value was higher with a 30s etch compared to no etching (21.63MPa vs. 18.05MPa). In addition, when etching for 30s, Adper Prompt L-Pop produced the highest minimum values (6.93MPa vs. 3.75MPa). 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

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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, the 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.

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. The initial Kruskal-Wallis test showed a statistically significant difference ($p<0.05$) between individual treatment groups. Further analysis was performed to evaluate the significance between specific groups. 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).

Adhesive Remnant Index (ARI)

All specimens were evaluated for their Adhesive Remnant Index (ARI). 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 the 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. 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 where 10% of the samples were an ARI of 4.

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 '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.

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 ($p<0.001$) [12]. This positive trend suggesting the use of pre-conditioning with acid etch prior to single-step adhesive placement were found in several other studies [13-15]. This finding of improved shear bond strength through the use of acid etching prior to single step adhesive placement is in agreement 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 [16].

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 6-8MPa was suggested by Reynolds to be clinically relevant when bonding orthodontic attachments to enamel [6]. Alternatively, an *in vitro* "ideal bond strength" of 3-4MPa has been suggested as a clinical minimum based on bonding orthodontic brackets with glass ionomer to enamel9. Using this range (3-8MPa) as a baseline, it could be conceivable 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 3MPa 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 significant 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).

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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 [9].

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 [17]. 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, 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.

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 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.

Limitations of the current study:

The study was an *in vitro* study. Future tests *in vivo* are highly recommended.

1. This study was performed on smooth surface enamel only.
2. 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.

Conclusion

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

A. 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.

B. Most adhesive material remained on the composite block after breakage, indicating that enamel breakage would not be expected with either of the two adhesive systems.

C. From a bond strength and reliability perspective, Adper Prompt L-Pop with 30s etch had the highest median, and minimum values.

D. Judging from the *in vitro* findings, both the Adper Prompt L-Pop and Optibond Solo Plus are appropriate for clinical use and Adper Prompt L-Pop is considered a useful adjunct to save restorative chair time and aid in patient management of the pediatric dental patient.

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