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A Comparison of Shear Bond Strengths of Orthodontic Brackets Bonded using  
Self-Etching Primer with Varying Time Intervals between Activation and  
Application

Katherine D. Plunkett, D.M.D.

A thesis submitted to the faculty of the Medical University of South Carolina in partial  
fulfillment of the requirement for the degree of Master of Science in Dentistry in the  
College of Dental Medicine.

Department of Pediatric Dentistry and Orthodontics  
Division of Orthodontics

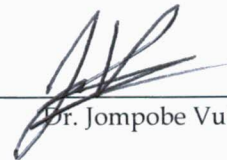
2016

Approved by:



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Dr. Jing Zhou, Committee Chair



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Dr. Jompobe Vuthiganon



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Dr. Roberta Gardner

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KATHERINE DEAN PLUNKETT. A Comparison of Shear Bond Strengths of Orthodontic Brackets Bonded using Self-Etching Primer with Varying Time Intervals between Activation and Application (Under the direction of JING ZHOU)

**Objective:** This study aims to determine the shear bond strength of orthodontic brackets using Transbond Plus Self Etching Primer with varying time intervals between activation and application. The scheduled bonding times include 0, 2, 8, and 24 hours after self-etching primer mixing and activation. Specifically, this research hopes to elucidate the following: compare the shear bond strength of self-etching primer to conventional etch, assess bond strengths of self-etching primer at varying times after activation, and determine whether the shear bond strength (SBS) of self-etching primer (SEP) is clinically acceptable when applied hours after activation. A secondary objective is to evaluate the interface at which bond failure occurs.

**Materials and Methods:** Extracted human teeth (50 premolars, 50 incisors) were divided into 5 groups of 10 specimens per tooth type, as follows: Group 1- conventional 37% phosphoric acid etch (CM), Group 2-SEP at time of activation, Group 3- SEP 2 hours after activation, Group 4- SEP 8 hours after activation, Group 5- SEP 24 hours after activation. The self-etching primer for groups requiring activation at a time prior to bonding application was mixed according to manufacturer's guidelines, sealed in Ziploc bags, then stored in a dark, dry drawer at room temperature until ready for bonding. Teeth were bonded in accordance with

manufacturer's instructions, differing only in the time at which the bonding procedure takes place after the self-etching primer is opened and activated. Shear bond strengths of each specimen were recorded using the UltraTester Machine. To assess the efficacy of the product with a more accurate representation of the procedure that would occur to correct an emergency loose bracket, the incisors were rebonded. The brackets were sand-blasted, and the excess adhesive was removed from enamel. The same bonding procedures were followed for each of the 5 groups above, and the bond strengths were once again recorded. Brackets were analyzed under a stereomicroscope with 15x magnification to determine ARI values.

**Results:** Shear bond strengths were not affected with increasing time between activation and application. No statistically significant differences existed between time intervals within a tooth type nor between tooth types within any single time group. Statistically significant differences were found within the data were ARI values between the following: group 1 and group 2 ( $p=0.0045$ ), group 1 and group 5 ( $p=0.0016$ ), group 3 and group 5 ( $p=0.0261$ ), and combined incisors and premolars ( $p=0.0302$ ).

**Conclusions:** It is clinically viable, cost-effective, and time-efficient for orthodontists to maintain a single package of Transbond Plus Self Etching Primer for any brackets requiring bonding or rebonding within a 24-hour period without compromising bond strengths.

## **Introduction**

Chair time is extremely valuable in an orthodontist's private practice. The more efficient the practice is run, the greater number of patients can be seen. When emergencies arise, the schedule is interrupted. It is imperative to deal with any emergencies in a timely, effective manner. One of the most frequent orthodontic emergencies is a debonded bracket.

A reliable bond that has the ability to last throughout orthodontic treatment is critical to running a successful practice. When brackets become loose during treatment, there are multiple consequences. The unscheduled appointments are exasperating to the orthodontist because they interfere with the flow of the practice, put the clinician behind with their regularly scheduled appointments, and negatively impact the financial state of the office<sup>3</sup>. From another perspective, the patients and/or parents are inconvenienced with another office visit. If this continues to happen, the patient and/or parent may lose confidence in the orthodontist. In some instances, debonded brackets at inopportune times have the potential to compromise treatment outcomes<sup>4,5</sup>. Therefore, it is critical for orthodontists to choose products and techniques that will help reduce the likelihood of untimely debonded brackets.

Bond failures can occur for a number of reasons, including bonding techniques, contamination, and patient noncompliance. Some have even purported

that dietary habits and gender may affect bracket failure rates clinically <sup>6</sup>. While a practitioner has control over many variables, the patient's behavior is a huge factor that can only be dealt with by patient education and emphasis on compliance in order to complete treatment without delays.

One must also keep in mind that when treatment is completed in 24 months, the brackets must be safely removed from the enamel surface. A bond strength must be strong enough to endure the forces of orthodontic movements as well as reasonable masticatory forces without passing a threshold of bond strength that may become dangerous upon bracket removal. Ideally the bond strength should not exceed the fracture point of enamel, which is 14 MPa <sup>7</sup>. Hence, despite an orthodontist's best effort in taking all the necessary steps for successful bonding, there will still be the occasional emergency appointment just from the nature of the required temporary bond. Over the duration of comprehensive orthodontic treatment, bracket failure rates have been estimated somewhere between 1.18% and 8.06%. <sup>8,9</sup>

When emergency appointments do arise, it behooves the orthodontist to effectively correct the problem in the most time-efficient manner. Self-etching primer is a unique product that can save the clinician chair time by eliminating a step in the bonding process. It has been suggested that the use of self-etching primer can save up to 65% of chair time during the bonding process compared to



conventional etching methods <sup>10</sup>. While self-etching primer is marketed for single use, one package contains enough to bond brackets of an entire arch or 14 teeth. It is wasteful and costly to open a new package of self-etching primer for each individual with an emergency loose bracket. Therefore, many private practice orthodontists have advocated for the idea of opening a single self-etching primer package to be used for any such emergencies throughout the day. Of course, in this scenario quick tip applicators are always replaced after each tooth application to prevent cross-contamination. This seems to be a timesaver as well as a financially sound decision, as the single-use packages are rather costly.

However, there is limited research to indicate if the bond strength of the self-etching primer is affected when the product is mixed and activated hours before use. This is critical information. For instance, if bond strength is reduced when using the product 6 hours after opening, then using the primer at that time increases the likelihood of another emergency in the future, costing more valuable chair time and materials. Therefore, understanding the shear bond strengths using self-etching primer that has been activated at various times prior to use (up to 24 hours) is clinically relevant.

There are several aims in this study. The primary aim is to compare the shear bond strengths of self-etching primer used at varying times after activation in order to determine whether self-etching primer is clinically acceptable when applied

hours after activation. The data collected also allows confirmation of previous comparisons between the shear bond strengths of self-etching primer and the conventional bonding technique using phosphoric-acid. Secondly, this study evaluates the bracket bases microscopically to better understand the location of bond failure.

The null hypothesis of this study is that there will be no significant difference in shear bond strengths with varying durations of storage time after activation and before application of self-etching primer. The knowledge to be gained by this research could potentially affect how orthodontists choose to deal with emergency patients on a daily basis.

## **Literature Review**

The field of dentistry was revolutionized when Buonocore applied chemical principles used to enhance the adhesion of paint and acrylic to metal surfaces and proposed using an etchant of 85% phosphoric acid to prepare the enamel surface for direct bonding with a resin <sup>11</sup>. This was the birth of composite restorations, but many began to research how this concept could be applied to other fields. In 1965, Newman suggested that this bonding technique be used in orthodontics, and by the late 1970s many studies had shown this could be successful <sup>12-16</sup>. Since then, orthodontists have routinely used these etching techniques, albeit with a lesser concentration, to correct malocclusions by directly bonding brackets in lieu of banding every tooth.

There are many benefits to bracket bonding, including, but not limited to: improved esthetics, increased comfort due to less bulky appliances, streamlined placement and removal procedures, simplified plaque-removal for patients leading to improved oral hygiene, reduced gingival irritation and hyperplasia, eliminated the need for full-mouth separators, decreased risk of unseen decalcification under loosened bands, and easier caries detection and restoration during treatment <sup>17-19</sup>. Additionally, bonding eliminates the excess space of approximately 3.5 mm that full-mouth banding would create during treatment that would require correction in the retention phase <sup>20</sup>. Presently, the gold standard and most common technique used

for bracket bonding in orthodontics is etching with 37% phosphoric acid followed by separate priming and adhesive steps <sup>21, 22</sup>. While the advantages of direct bonding are immense, there are still problems with current techniques that can plague orthodontists during and after treatment.

One of the main concerns is the risk of decalcification around appliances. Approximately 5% of patients undergoing orthodontic treatment with fixed appliances have decalcifications <sup>23</sup>. While brackets cover less of the tooth and allow for easier brushing and flossing, the appliances are still cumbersome with a shape that is conducive to harboring plaque, especially if excess cement is not removed. According to Sorake, “the creation of new retentive areas favors the local growth of *Streptococcus mutans*, which in turn increases the general infection level of this organism” <sup>24</sup>. The same author asserts that the decalcification process can begin as early as 4 weeks after the band or bracket placement. Thorough oral hygiene instruction is imperative to reduce the risk of decalcification. Research has shown that it is most effective to include visual examples of hygiene techniques as well as the consequences of poor brushing habits, namely white spot lesions and gingival inflammation <sup>25</sup>. Interestingly, some research postulates that phosphoric acid etching may actually cause the development of decalcification, and eliminating this procedural step may reduce the formation of decalcification or white spot lesions <sup>23, 26, 27</sup>.

Another dilemma is the risk of enamel fractures during the debonding process. Both patients and practitioners alike strive to maintain healthy, pristine enamel after orthodontic treatment. Despite orthodontists' best efforts, damage to the enamel continues to be a major clinical problem <sup>28</sup>. Research has shown that enamel fractures are seen when bond strengths exceed 14MPa, and an increase in bond strength above that threshold is proportional to an increased risk of fractures <sup>7,29-33</sup>. While there is currently no scientific evidence to prove a specific threshold of bond strength that must be reached in order to withstand orthodontic treatment, a range of 5.9-7.8 MPa is widely reported as producing clinically acceptable bond strengths <sup>34-38</sup>. Brackets bonded with conventional techniques using 37% phosphoric acid almost always exceed 14 MPa, with bond strengths typically ranging from 20 to 25 MPa <sup>39</sup>. Not only is the strength of the bond a risk factor for enamel fractures, but the location at which the bond failure occurs can affect the incidence of enamel damage during bracket removal. It is safer and more desirable to have a cohesive bond failure within the adhesive layer than an adhesive bond failure at the enamel-adhesive interface <sup>36,40</sup>. While a stronger bond may seem better in order to reduce the risk of bond failures and emergency appointments, the location at which the bond is likely to fail and the future risk of damage is something that needs to be deliberated.

As there are so many factors to consider when determining what is best for the patient and the practice, orthodontists are constantly re-evaluating their routine techniques in order to preserve healthy tooth structure, shorten appointment times, and reduce overall treatment duration. It has subsequently been a never-ending quest to improve the efficacy and efficiency of the bonding process without detriment to the long-term oral health of the patient.

Researchers have studied variations in all steps of the bonding process including enamel preparation, etching techniques, adhesive agents, and even isolation methods. As a result, new products are constantly invented to simplify and eliminate the deficiencies of the current direct bonding procedures. As past research suggests that phosphoric acid etching may increase risk of decalcification and fractures, a major focus was placed on finding a replacement for this step. Scientists experimented with other acids, such as 10% maleic acid, as a substitute and found bond strengths similar to that of phosphoric acid <sup>41</sup>. In the late 1990s, promising bonds were also achieved with acidic primers composed of phenyl-P (etchant) as well as hydroxyethylmethacrylate (HEMA) and dimethacrylate (primer components) <sup>42</sup>. The shear bond strength achieved with the acidic primer was lower than, but comparable to, the gold standard bonding protocol with 37% phosphoric acid when used in conjunction with a highly filled adhesive. Manufacturers continued to revise products and combine new chemical compounds for more

successful outcomes. The accomplishment of this combined solution ultimately led to the birth of the orthodontic self-etching primer.

Originally, self-etching adhesives were created for bonding restorations to dentin <sup>43</sup>. Shortly after, these dual-acting primers were also found to be effective in enamel bonding <sup>44</sup>. The switch to self-etching primer was appealing in the field of orthodontics because of its potential to reduce chair time required during bond up appointments. Assuming the bond strength was not compromised, self-etching primer saves time by eliminating the initial etching step in the bonding process <sup>45</sup>. When this idea was tested, an average of 25 seconds per bracket was saved with the use of self-etching primer compared to the traditional 2-step phosphoric acid etch and primer technique <sup>46</sup>. That equates to a potential savings of over 10 minutes of chair time for a full upper and lower arch bond up.

The traditional bonding method follows the construct of a 4<sup>th</sup> generation bonding agent, meaning there are 3 separate materials: etchant to demineralize the hydroxyapatite, primer to remove the water from rinsing and create resin tags within the demineralized areas, and unfilled adhesive to produce a hybridized tooth surface prior to a filled resin adhesive/composite. Because orthodontic brackets are bonded solely on enamel, the primer step is unnecessary. Still, within orthodontic nomenclature, the steps are traditionally called etchant, primer, and adhesive. Technically, the primer step is not a primer at all, but rather an unfilled resin

adhesive similar to the 'bond' step in the 3-step etch, prime, bond used when bonding restorative composites to dentin. However, as this product is ubiquitously referred to as primer within the orthodontic community and literature, we will continue with this nomenclature throughout the study. Self-etching primers fall under the umbrella of 6<sup>th</sup> generation bonding agents <sup>47</sup>. This newer generation of bonding agents offers many advantages. In addition to time-savings as a result of eliminating a separate step in the bonding process by combining the etchant and primer, one less step translates to one less opportunity for costly mistakes to be made during the bonding process. Manufacturers emphasize that the success or failure of a bond using 4<sup>th</sup> and even 5<sup>th</sup> generation systems is heavily influenced by small changes in technique, stressing the importance of consistent etching times and adequate rinsing and drying. However, many of these sources of error are eliminated with the use of 6<sup>th</sup> generation products <sup>48</sup>.

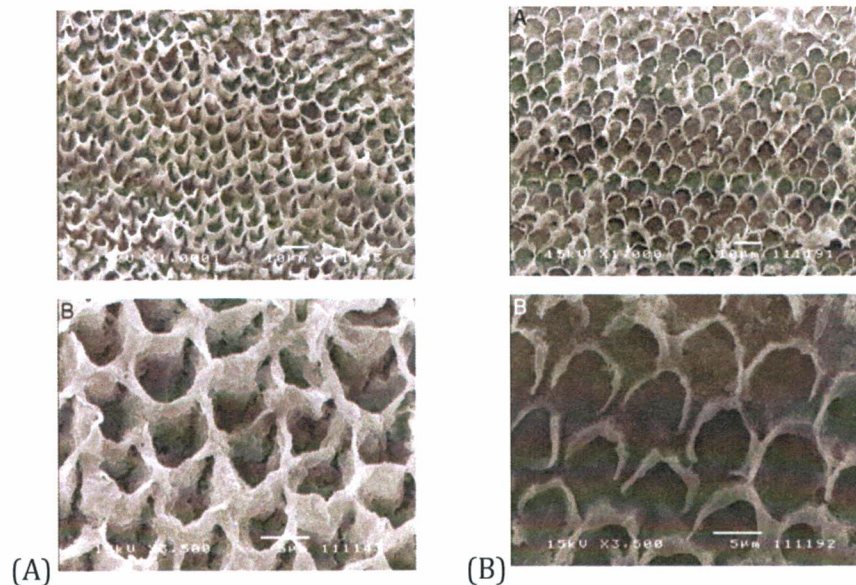
It is also important to note that self-etching primers have been shown to outperform conventional etching methods when bonding in blood- or saliva-contaminated environments <sup>49-52</sup>. As bond strengths still show a reduction in these conditions, it is nevertheless recommended to continue with proper isolation protocols to avoid contamination when possible <sup>53, 54</sup>. However, if blood or saliva is likely to enter the bonding field it has been advised to use a self-etching primer in these situations <sup>55, 56</sup>. Moreover, the risk of contamination during bonding is reduced



with the use of self-etching primers because the rinsing step is no longer indicated<sup>57</sup>. These facts should be seriously considered since bonding in the presence of saliva or blood is one the common causes of bond failures and emergency appointments. If orthodontists routinely utilized a product that performed successfully in these environments, many of the frustrations associated with unscheduled bond failures would be eliminated. In summary, self-etching primer procedures are simplified and the product is less technique-sensitive and more resistant to contamination<sup>35, 58</sup>.

Additionally, self-etching primers appear to minimize the amount of enamel lost during the etching process<sup>59, 60</sup>. Scanning electron microscopy studies have shown that a milder etch and shorter resin tags are obtained with these newer acidic primers<sup>61</sup>. Acid-etching creates a porous enamel surface layer that ranges in depth from 5 to 50  $\mu\text{m}$  (figure 1A), while the combined etchant and primer appears much shallower as shown in figure 1B below<sup>2, 62</sup>. As long as there is no compromise to bond strengths, this shallower enamel demineralization may be beneficial in reducing the risks of the previously discussed concerns of decalcifications during orthodontic treatment as well as iatrogenic enamel damage upon bracket removal<sup>28, 63</sup>. There are studies confirming there is no correlation between resin tag length and shear bond strength, rather bond strengths are primarily associated with the ability of the resin to penetrate the demineralized enamel rods<sup>64</sup>. Furthermore, the

combination of etchant and primer guarantees that the depth of hydroxyapatite dissolution and primer penetration is simultaneous and indistinguishable, which ensures that no enamel rods are left exposed and reduces the likelihood of micro-leakage or post-bonding sensitivity<sup>65,66</sup>.



**Figure 1 (A) and (B).** Enamel etching pattern after (A) phosphoric acid and (B) Transbond Plus SEP under 1000x and 3500x magnification. Note the deep, uniform penetration of resin tags with phosphoric acid, while SEP resin tags are shorter but still regularly formed .<sup>2</sup>

One potential downfall of the newer combined etchant/primers compared to conventional etching techniques is the lack of visible confirmation that the etching process has been successful<sup>37</sup>. In contrast to the uniformly frosty enamel created after phosphoric acid etching before the primer is applied to re-wet the tooth surface, the simultaneous priming that occurs with self-etching primers hides this

intermediate step. This makes it more difficult for the clinician to determine if adequate etching has taken place. So while 6<sup>th</sup> generation products are known as less technique sensitive, it is still imperative to follow manufacturer's application instructions to be reasonably assured that the tooth surface is prepared for a successful bond.

Released in 2000 by 3M Unitek (Monrovia, CA, USA), Transbond Plus Self-Etching Primer was the first self-etching primer released solely for orthodontic purposes. Transbond Plus SEP is packaged for single-use applications including a micro brush and a foil package with 3 compartments. It is difficult to maintain chemically stable concentrations of the various components (acid, primer, and adhesive) of 6<sup>th</sup> generation bonding systems. Typically their components are stored separately until mixed for clinical use in order to prevent changes in the initializers that may occur when exposed to acids over time <sup>67</sup>. When the bottom 2 compartments are squeezed into the top reservoir and stirred with the micro brush, the product is activated and ready to be applied for bonding. One reservoir is filled with methacrylated phosphoric acid ester (the primary active etchant/primer), bis-GMA, (resin filler) camphorquinone (photo-initiator), dimethylbenzocaine (slow-acting polymerization accelerator), and stabilizers. The other reservoir contains water (the solvent), 2-HEMA (monomer), fluoride complex, and other stabilizers <sup>68</sup>. <sup>69</sup>. The exact composition of these ingredients is not published as it is proprietary

information. The pH of Transbond Plus Self Etching Primer is reported as 1.0<sup>5,58</sup>. As described by Cinader, the following chemical reaction occurs with the application of this self-etching primer:

The phosphate group of the methacrylated phosphoric acid ester dissolves the calcium and removes it from the hydroxylapatite. Rather than being rinsed away, the calcium forms a complex with the phosphate group and is incorporated into the network when the primer polymerizes. Three processes serve to arrest the action of the acid in the material. First, the phosphate group forms a complex with the calcium of the hydroxylapatite (as with phosphoric acid). Second, the air burst drives the solvent from the primer, thus increasing the viscosity of the material and slowing the transport of acid groups to the enamel surface. Third, as the primer is light-cured and the monomers are polymerized, the transport of acid groups to the enamel surface is finalized.<sup>70</sup>

The benefits of adding fluoride to the bonding system are two-fold. Firstly, fluoride has a bactericidal effect, creating an environment that is difficult for *S. mutans* to survive. Secondly, fluoride has the ability to substitute a hydrogen ion to make fluorapatite crystals within the enamel which are less vulnerable to decalcification in the presence of acid created by oral bacteria feeding on plaque<sup>71</sup>. Because of the presence and subsequent release of fluoride from this self-etching

primer, the incidence of demineralization may be reduced with the use of this product <sup>72</sup>.

There have been a multitude of in vitro studies measuring shear bond strength of these self-etching primers. Shortly after the release of 3M's product, Bishara reported that the shear bond strength of Transbond Plus Self Etching Primer ( $7.1 \pm 4.4$  MPa) was lower than that found with phosphoric acid etch ( $10.4 \pm 2.8$  MPa), but the strength was still within clinically acceptable values <sup>73</sup>. Grubisa reiterated these findings, with shear bond strengths of  $9.8 \pm 4.2$  MPa and  $7.1 \pm 4.2$  MPa <sup>35</sup>. Arnold reported no significant differences in bond strengths between the groups, with averages of  $9.7 \pm 3.1$  MPa for conventional techniques and between  $8.0 \pm 1.3$  MPa and  $9.8 \pm 3.7$  MPa for self-etching primer <sup>5</sup>. Similarly, Mirzakouchaki found equivalent bond strengths between conventional and Transbond Plus Self Etching Primer when bonding metal brackets, with an average of  $8.5 \pm 1.1$  MPa versus  $9.2 \pm 1.4$  MPa for self-etching primers and conventional etch, respectively <sup>57</sup>.

While laboratory studies are one of the first steps in testing the efficacy of a new material, variations in tooth selection, protocol modifications, and standardization required for lab testing can cast doubt upon the clinical relevance of their findings <sup>74</sup>. Ultimately, in vivo research is crucial to determining the clinical viability of the product. Aljubouri found no statistically or clinically significant differences in bond failure rates between groups after 12 months, with bond failure

rates per patient at 1.54% for the 1-step combined etchant/primer and 2.78% for the 2-step etch then prime method <sup>46</sup>. Many studies corroborate the above findings of similar failure rates between the 2 groups <sup>75-77</sup>. Several other studies have also found low failure rates with Transbond Plus Self Etching primer irrespective of conventional techniques, with one 14-month study reporting an exceptionally low 0.94% <sup>6, 78</sup>. A 6-month trial actually found a reduction in failure rate with the use of self-etching primers, with the failure rate of conventional techniques being 0.3 times greater <sup>79</sup>. In contrast, one article found a higher rate of bond failure within the self-etching primer group (10.99% vs 4.65%), but with the sample size and other variables the authors deduced that there is only weak evidence to suggest the failure rates would be higher with the use of self-etching primers <sup>80</sup>. With a majority of evidence supporting that the use of self-etching primer is not likely to increase bond failures, the advantages of time-savings and a possible reduction in enamel damage during treatment have led to an increased number of orthodontists choosing this product.

When clinicians begin using 6<sup>th</sup> generation bonding agents, it is likely that a new package of self-etching primer will be opened as each emergency arises. As of February 2016, the product is purchased as a box of 100 for \$229.38, so each lollipop package costs approximately \$2.29, which amounts to \$0.16 per tooth bonded. That means the practitioner is throwing away \$2.13 at each unscheduled

appointment that requires rebonding only a single bracket. As there is always a desire to reduce overhead as well as limit the required inventory, the orthodontist must decide between 2 options, both with their own drawbacks: 1) accept the wasted cost of using self-etching primer for emergencies to maintain the ease of use and time-savings of the product, or 2) maintain a larger inventory that includes phosphoric acid to be used for such situations and accept that the time required for these untimely appointments will be longer than if using self-etching primer. Essentially, the orthodontist must decide between time and money. Ideally, the excess self-etching primer could be saved for future use to avoid any such waste. While the number of published articles on the shear bond strength of self-etching primers is vast, there is extremely limited research on the bond strength of this product when there is time elapsed between chemical activation of the product and application to a tooth for bonding. This study tries to offer some insight in the possibility of using a single package of self-etching primer for a number of loose brackets over a period of time to eliminate the waste of both time and money. Although this is an in vitro study, promising results could begin the process of revolutionizing the current protocol for how orthodontists deal with these unscheduled appointments.

## **Materials and Methods**

### *Tooth Collection*

This laboratory study was approved with an exempt status by the Medical University of South Carolina's Institutional Review Board. Extracted human teeth were collected by the Department of Graduate Oral Surgery within the Medical University of South Carolina College of Dental Medicine as well as multiple private practice oral surgery offices in Charleston. All teeth were extracted for purposes other than this study. Collected teeth were stored in phosphate-buffered saline to ensure a bio-inert environment that would avoid dehydration and not affect the properties of enamel. Only teeth with intact facial surfaces were included in the study. Those with large caries or visible enamel cracks were excluded. A total of 50 premolars and 50 incisors were utilized during this research.

### *Tooth Preparation*

The teeth fitting the above criteria were sectioned below the cemento-enamel junction with a diamond disk to remove the roots. Acrylic mounting is necessary in order to stabilize the specimen in the machine for testing. These teeth were embedded in cylinders of Dentsply Orthodontic Resin (York, PA) leaving only the facial surface available for bonding. During this process, a level was used to confirm the bonding surface of the crown was parallel to the ground which ensured that the shear bond strength measurements would be obtained with purely an



occlusogingival load. Any excess acrylic that may interfere with the testing machine engaging the bracket was removed with acrylic bur. Each tooth was maintained in the phosphate-buffered saline solution unless actively being prepared for testing, and the mounted specimens were placed in the same solution once the acrylic was set.

The specimens were divided into 5 premolar test groups and 5 incisor test groups, each containing 10 teeth. The groups are as follows:

Group 1- Conventional Etch Technique (CM)

Group 2- Self-etching Primer applied 0 hours after activation (0-HR SEP)

Group 3- Self-etching Primer applied 2 hours after activation (2-HR SEP)

Group 4- Self-etching Primer applied 8 hours after activation (8-HR SEP)

Group 5- Self-etching Primer applied 24 hours after activation (24-HR SEP)

The acrylic cylinders containing incisors required a label in order to compare the original shear bond strength with the bond strength of the same tooth during the rebond test. Each incisor-containing acrylic block was numbered 1-50 during the mounting process, and a random number generator was used to create groups of 10 numbers to determine the specimens for each of the 5 test groups. Since the premolars would only be run for a single test cycle, no labels were used and the premolar-containing acrylic blocks were placed into multiple jars containing phosphate-buffered saline solution in a non-systematic order and pulled for groups

randomly during testing to avoid any potential bias. The premolars were only labeled after bonding completion to allow shear bond strengths to be assessed by group.

#### *Self-Etching Primer Activation*

The self-etching primer allotted for groups involving a lapse in time prior to application were activated at their specified times prior to bonding. The self-etching primer was mixed according to manufacturer's recommendations as follows:

Begin by firmly squeezing the bottom black reservoir to allow the contents to flow into the middle white reservoir. Ensure the black reservoir is completely empty, and then fold the black reservoir on top of the white reservoir to ensure the liquid does not flow backward in the package. Firmly squeeze white reservoir contents into the top purple reservoir and swirl the applicator for 5 seconds to completely mix chemicals. If the applicator is removed and the liquid isn't yellow, re-squeeze contents to middle reservoir to remix liquids. <sup>81</sup>

Once the mixed self-etching primer appeared yellow, the activation phase was completed. The quik tip applicator was then removed from the lollipop system, and the top foil of the package was folded over to prevent the liquid from leaking. A sealed Ziploc bag was used for storage to prevent evaporation. These bags were

labeled for time of activation and group number, then stored in a dark, dry drawer at room temperature until ready for bonding.

### *Bonding Procedure*

Brackets were bonded to the teeth by group, allowing a 20-minute window per group for the bonding process to be completed. The materials and set-up used for this process can be seen in figure 2. All bonding surfaces were cleaned with unflavored, oil-free pumice and a slow speed handpiece with a rubber prophylaxis cup for 10 seconds to remove any debris. The teeth were then thoroughly rinsed with an air-water spray and dried. Teeth were prepared and bonded in accordance with product manufacturer's instructions, differing only in the etching methods for group 1 and the time at which the bonding procedure takes place after the self-etching primer is opened and activated for the remaining groups. Lower incisor brackets were used for the incisor groups and lower first premolar brackets were used for the premolar groups (American Orthodontics Low Profile series, Sheboygan, WI). The lingual pad of these brackets consists of 80-gauge mesh layered over an etched foil base. Transbond Plus color-changing adhesive (3M, Monrovia, CA) was used for all groups. Adhesive was applied to the bracket base, then the bracket was firmly pressed onto the enamel surface and excess adhesive was removed with a periodontal probe. The position of the bracket was visually inspected to maintain parallelism of the bracket to the ground to allow for a pure occlusogingival load

during shear bond strength testing (figure 3). The brackets were then light cured with a Valo Ortho Cordless light (South Jordan, UT) with a reported wavelength emission of 395-480 nm for 10 seconds from the occlusal and 10 seconds from the gingival.

For the conventional method group, the 37% phosphoric acid etch was in place for 30 seconds prior to rinsing and drying until the enamel surface appeared frosty. Assure (Reliance, Itasca, IL) was used as the primer in this group; the primer was placed with a quik tip applicator and air thinned prior to bracket placement.

The self-etching primer for the remaining groups was applied in accordance with 3M's bonding protocol, a continuation from above, as follows:

The saturated applicator is rubbed onto each tooth surface for at least 3-5 seconds per tooth, with reapplication of liquid on the micro brush between each tooth. Once all teeth are primed, gently air dry the primer into a thin film. Lastly, bond brackets with adhesive (Transbond Plus) and light cure.

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To clarify, this research applied the self-etching primer for 5 seconds, timing this step to ensure consistency.

It is common practice with bond strength laboratory research to allow a 24-hour delay between bonding and testing<sup>82</sup>. Therefore, this study followed a similar

protocol, storing specimens once again in phosphate-buffered saline solution for 24 hours before testing shear bond strength.



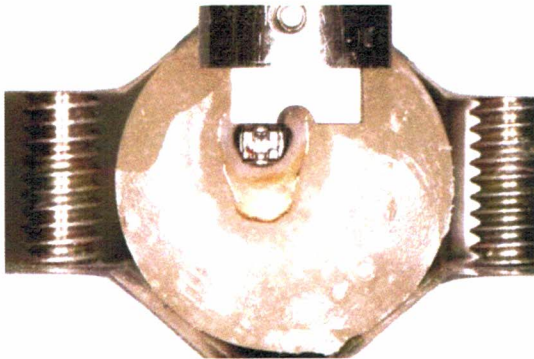
**Figure 2.** Basic set-up used during bonding procedures for each group.



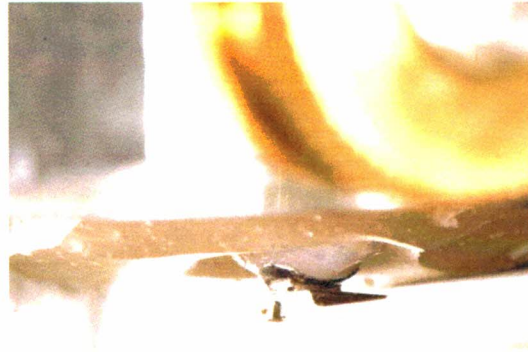
**Figure 3.** A close-up view of visual inspection of the bracket parallelism prior to light curing. This ensures the shear bond strength will be tested with purely an occlusogingival load.

### *Shear Bond Strength Testing*

Shear bond strength of each bonded bracket was recorded using the UltraTester Machine (Ultradent, South Jordan, UT). An example of a specimen positioned on the machine for testing is shown in figures 4 and 5. The bond strength on the machine was computed as a pound-force unit. This was converted to MPa ( $\text{N}/\text{mm}^2$ ) by first converting pounds to Newtons using a known equation ( $\text{lb} \times 4.44822162 = \text{N}$ ) then dividing the product by the bracket base surface area. The manufacturer (American Orthodontics) provided the surface area of the lingual pad of the bracket bases, which is  $8.2960737304 \text{ mm}^2$  for the lower first premolar and  $8.4215057376 \text{ mm}^2$  for the lower incisor. The conversion to MPa was to allow a more accurate comparison to other studies' data.



**Figure 4.** A premolar positioned properly in the UltraTester ready for testing. Note both occlusal wings will be pressed by the load simultaneously.



**Figure 5.** Same premolar specimen from an oblique view. This view was always checked to ensure only the wings were engaged and there were no other interferences.

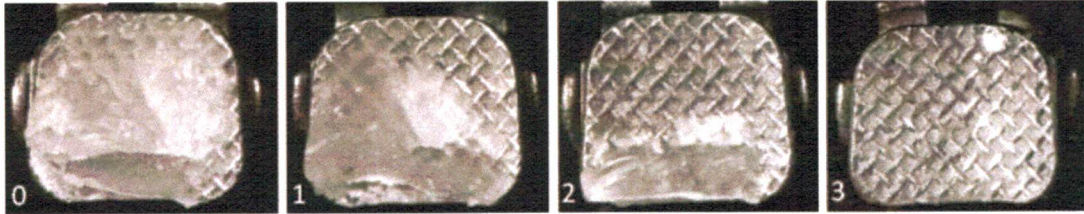
### *Rebonding Test Groups*

Because one of the main objectives of this research is to determine the efficacy of conserving a single self-etching primer package for emergency appointments with debonded brackets throughout the day, the researcher decided to test a 3<sup>rd</sup> set of groups using rebonded brackets on previously bonded teeth to better simulate the clinical presentation of these unscheduled patients. The mounted incisors were selected. The incisor brackets were sand blasted to remove residual adhesive and visually inspected for any irregularities. One bracket was thrown out for distortion of the base, therefore taking the 24-hour SEP group to 9 specimens. Excess adhesive on the enamel surface was removed with a tungsten carbide bur (Henry Schein FG 7803, Melville, NY). The activation and bonding protocols from the previous groups were repeated with the 5 incisor groups.

Another 24-hour storage period took place prior to shear bond strengths being recorded with the UltraTester Machine.

#### *Adhesive Remnant Index*

Each bracket was viewed under a stereomicroscope (Carl Zeiss Stemi 508) at 15x magnification in order to determine at which interface the bond failed. While bond failure at the enamel-adhesive interface makes cleanup at debond appointments faster and easier, the risk of enamel fracture during the debond procedure is increased. Therefore, the ideal bond failure is cohesive within the adhesive layer as opposed to an adhesive bond failure at either the enamel-adhesive interface (increased risk of enamel fracture) or the bracket-adhesive interface (longer time required for cleanup). Årtun and Bergland first introduced the adhesive remnant index (ARI) for the purpose of understanding the bond failure locations<sup>83</sup>. They created a 4-point scale to classify the amount of the residual adhesive on the bracket after debonding. Others have expounded on this idea to include 5- or 6-point scales. This study used the original scale as this is quite common in literature. The scale and corresponding examples can be found in figure 6 below.



**Figure 6.** Adhesive remnant index evaluation used a 4-point scale in which 0 = the entire adhesive left on the bracket base, 1= more than half of the adhesive left on the bracket base, 2 = less than half of the adhesive left on the bracket base, and 3= no adhesive left on the bracket base. <sup>1</sup>

Each bracket base was photographed under the microscope. From the photos, the ARI was recorded for each bracket twice by a single judge on 2 consecutive days to gauge the intra-rater reliability. For any bracket with a discrepancy in the 2 scores, the judge reviewed the bracket again to determine the final score.

#### *Statistical Analysis*

Previous studies were used as guides to help determine the power analysis and required sample size. First, a manuscript with large variability was used to estimate requirements in the case that this study's results include large bond strength differences among the groups. This yielded an effect size of 1.24 with a common standard deviation of 5.5 in order to detect a difference in shear bond strength between the five groups <sup>34</sup>. This creates a power of >99% at an alpha level of 0.05 with a sample size of 8 per group (n=40 total). A manuscript with smaller



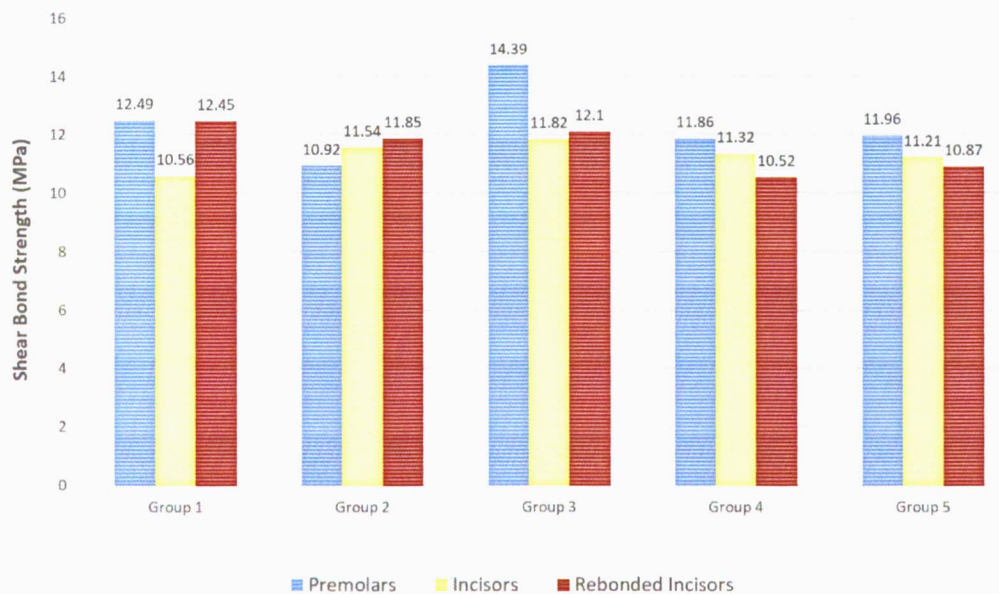
variability was also reviewed to estimate sample size in the event that this study's results produce only small differences between bond strengths <sup>72</sup>. This yielded an effect size of 0.75 with a common standard deviation of 4. This also gave us power of >99% at an alpha level of 0.05 to detect a difference between the groups, with a sample size of 8 per group (n=40 total). With both calculations producing the same sample size, it can confidently be said that this study requires 8 specimens per group for a total of 40 specimens. Due to unknown variability of extracted teeth and to avoid falling under the minimum number or required specimens on the chance that a specimen is invalid, it was decided to include 10 specimens in each test group.

Once data collection is completed, bond strengths of each group will be recorded and summarized as means, standard deviations, minimum and maximum values. To analyze the difference in the 5 groups within tooth type, a linear mixed model will be utilized. For the post-hoc pairwise comparisons, Scheffe's method will be applied. The averages will also be compared to what is currently known as standard clinical bond strength for orthodontic brackets (5.9-7.8 MPa) in order to determine whether a lapse in time between activation and application of self-etching primer will allow for clinically acceptable bonding in an orthodontic practice.

## **Results**

Out of the total 150 specimens prepared, there were 6 that were disregarded when calculating results: 1 specimen in the premolar 8-hour group for an enamel fracture that occurred during the bond strength testing with the UltraTester, 1 specimen in the premolar 2-hour group for inadequate acrylic retention, 2 specimens, 1 in the premolar 2-hour group and 1 in the incisor 0-hr group, for incorrect tooth selection (specimens were inadvertently switched prior to bonding), 1 specimen in the rebonded incisor 0-hr group because the previous incorrect tooth was unavailable for rebonding, and 1 specimen in the rebonded 24-hour group because the bracket was distorted from the initial debonding test and was discarded. Hence, there were 144 total specimens used when calculating results. The shear bond strengths of each group, including mean, standard deviation, and range, are reported in Tables 1-3. The diagrammatic representation (Figure 7) of the average bond strengths illustrates how similar bond strengths appeared between groups. Statistical analysis included multiple linear mixed models where the fixed effect was time, and Scheffe's method was applied for the post-hoc pairwise comparisons. The p-values obtained for differences in time intervals within each tooth type can be seen in Table 4. A similar linear mixed model was used to compare between the 3 tooth types within a specific time group, using tooth type as the fixed effect in this model, and the p-values calculated can be found in Table 5. With such

high p-values, no statistically significant differences existed between time intervals within a tooth type nor between tooth types within any single time group. It should also be noted that no trends in bond strength changes between time intervals were identified, with the average shear bond strengths increasing and decreasing randomly between the various groups.



**Figure 7.** Average shear bond strengths for each time interval group by tooth type. Group 1- Conventional Etch, Group 2-SEP 0 hrs after activation, Group 3-SEP 2 hrs after activation, Group 4-SEP 8 hrs after activation, Group 5-SEP 24 hrs after activation.

**Table 1.** Average Shear Bond Strengths of Premolar Groups

| Time                             | Variable | N  | Mean  | Std Dev | Minimum | Maximum |
|----------------------------------|----------|----|-------|---------|---------|---------|
| Conventional Method              | SBS lbs  | 10 | 23.29 | 6.23    | 16.1    | 38.3    |
|                                  | SBS MPa  | 10 | 12.49 | 3.34    | 8.63    | 20.54   |
| SEP at 0 hours after activation  | SBS lbs  | 10 | 20.37 | 6.95    | 7.5     | 29.4    |
|                                  | SBS MPa  | 10 | 10.92 | 3.73    | 4.02    | 15.76   |
| SEP at 2 hours after activation  | SBS lbs  | 10 | 26.83 | 4.8     | 20      | 36.8    |
|                                  | SBS MPa  | 10 | 14.39 | 2.58    | 10.72   | 19.73   |
| SEP at 8 hours after activation  | SBS lbs  | 9  | 22.12 | 10.19   | 9.2     | 34.2    |
|                                  | SBS MPa  | 9  | 11.86 | 5.46    | 4.93    | 18.34   |
| SEP at 24 hours after activation | SBS lbs  | 8  | 22.31 | 6.22    | 11.9    | 27.4    |
|                                  | SBS MPa  | 8  | 11.96 | 3.34    | 6.38    | 14.69   |

**Table 2.** Average Shear Bond Strengths of Incisor Groups

| Time                             | Variable | N  | Mean  | Std Dev | Minimum | Maximum |
|----------------------------------|----------|----|-------|---------|---------|---------|
| Conventional Method              | SBS lbs  | 10 | 20    | 4.79    | 12.4    | 24.5    |
|                                  | SBS MPa  | 10 | 10.56 | 2.53    | 6.55    | 12.94   |
| SEP at 0 hours after activation  | SBS lbs  | 9  | 21.84 | 5.45    | 7.9     | 25.6    |
|                                  | SBS MPa  | 9  | 11.54 | 2.88    | 4.17    | 13.52   |
| SEP at 2 hours after activation  | SBS lbs  | 10 | 22.37 | 4.56    | 11.3    | 26.8    |
|                                  | SBS MPa  | 10 | 11.82 | 2.41    | 5.97    | 14.16   |
| SEP at 8 hours after activation  | SBS lbs  | 10 | 21.43 | 4.45    | 14.5    | 28.2    |
|                                  | SBS MPa  | 10 | 11.32 | 2.35    | 7.66    | 14.9    |
| SEP at 24 hours after activation | SBS lbs  | 10 | 21.23 | 5.39    | 10.2    | 27.3    |
|                                  | SBS MPa  | 10 | 11.21 | 2.85    | 5.39    | 14.42   |

**Table 3.** Average Shear Bond Strengths of Rebonded Incisor Groups

| Time                             | Variable | N  | Mean  | Std Dev | Minimum | Maximum |
|----------------------------------|----------|----|-------|---------|---------|---------|
| Conventional Method              | SBS lbs  | 10 | 23.57 | 1.86    | 20.6    | 26.8    |
|                                  | SBS MPa  | 10 | 12.45 | 0.98    | 10.88   | 14.16   |
| SEP at 0 hours after activation  | SBS lbs  | 9  | 22.43 | 2.26    | 20      | 26.1    |
|                                  | SBS MPa  | 9  | 11.85 | 1.2     | 10.56   | 13.79   |
| SEP at 2 hours after activation  | SBS lbs  | 10 | 22.9  | 4.62    | 14.1    | 27.5    |
|                                  | SBS MPa  | 10 | 12.1  | 2.44    | 7.45    | 14.53   |
| SEP at 8 hours after activation  | SBS lbs  | 10 | 19.91 | 4.67    | 12.5    | 25.6    |
|                                  | SBS MPa  | 10 | 10.52 | 2.46    | 6.6     | 13.52   |
| SEP at 24 hours after activation | SBS lbs  | 9  | 20.58 | 4.68    | 14.1    | 28.1    |
|                                  | SBS MPa  | 9  | 10.87 | 2.47    | 7.45    | 14.84   |

**Table 4.** P-values evaluating shear bond strength differences in varying time points within a single tooth type

|           |        |
|-----------|--------|
| Premolars | 0.3369 |
| Rebonded  | 0.1916 |
| Incisors  | 0.8300 |

**Table 5.** P-values evaluating shear bond strength differences in varying tooth types within a single time point

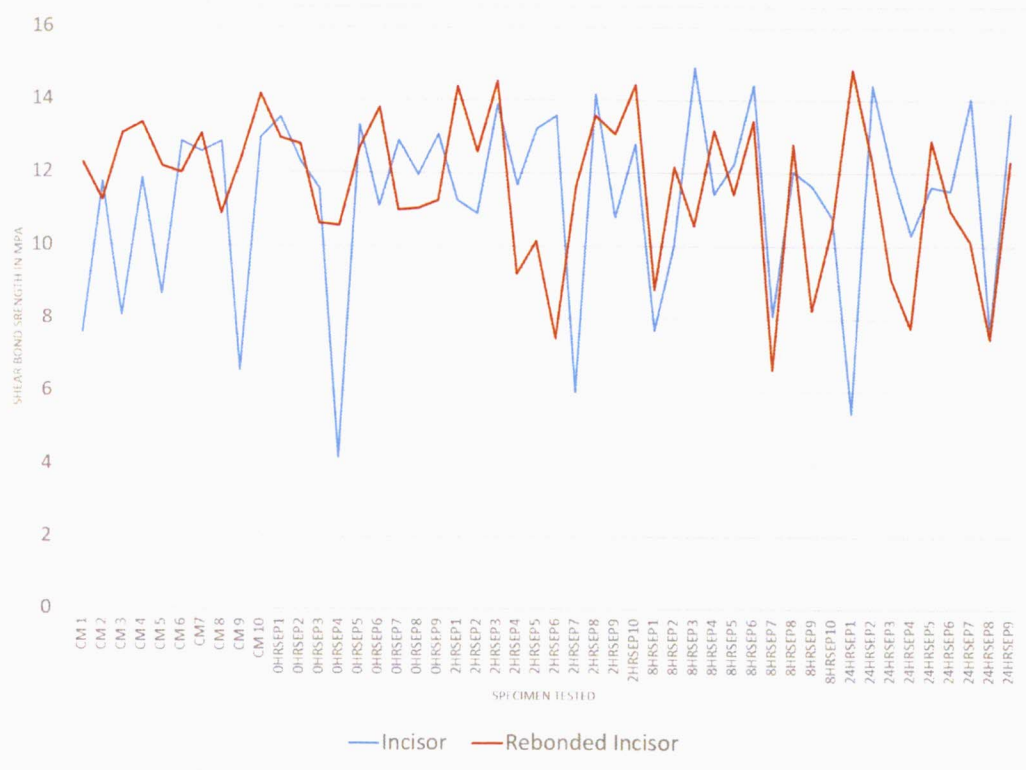
|                                  |        |
|----------------------------------|--------|
| Conventional Method              | 0.1606 |
| SEP at 0 hours after activation  | 0.7710 |
| SEP at 2 hours after activation  | 0.0547 |
| SEP at 8 hours after activation  | 0.7208 |
| SEP at 24 hours after activation | 0.7323 |

As the same incisors were bonded twice, there was an attempt to compare the original bond strength to that obtained after rebonding (simulating an

emergency appointment). Figure 8 depicts the values of each incisor for both groups. Out of the 48 specimens bonded twice, 22 had an increased shear bond strength with rebonding while 26 had a reduced bond strength. The average increase in bond strengths for those specimens that did so was only 3.13 MPa, and the average decrease was 2.13MPa. Moreover, the teeth bonded using conventional methods had the most specimens show an increase in bond strength after rebonding (7 out of 10), and the teeth bonded using self-etching primer with a 2-hour interval between activation and application were not far behind (6 out of 10). The remaining 3 groups had more specimens with a reduction in bond strength (6 out of 9 for SEP 0-Hr, 6 out of 10 for SEP 8-Hr, and 7 out of 9 for SEP 24-Hr).

A secondary aim of this study was to evaluate the bond failure locations of each specimen using the Adhesive Remnant Index (ARI). All brackets were viewed under a stereomicroscope at 15x magnification. The photos were taken and the ARI values were determined from the obtained photos. To ensure consistency in scoring, the same clinician evaluated all brackets twice, with a 24-hour interval between evaluations. The intra-rater reliability between the 2 evaluations was 95.8% (91 out 95). The 4 specimens where an inconsistency was recorded were reviewed for a 3<sup>rd</sup> time to determine the ultimate ARI value. Interestingly, all specimens requiring review were incisors, 2 in the 0-hour group and 2 in the 8-hour group. Half of these reviewed brackets were determined to be the higher of the 2 values while the other

half were determined to be the lower of the 2 values, so no general pattern of error could be determined. Summary statistics, frequency distribution, and significant p-values for the tooth and time groups are presented in terms of mean and standard deviation in Tables 6 and 7. A depiction of the average ARI values for each of the 5 groups within each of the tooth types is illustrated on figure 9.



**Figure 8.** Comparison of shear bond strengths of original vs rebonded incisors. Note that 22 of the 48 teeth had increased shear bond strength at rebond.

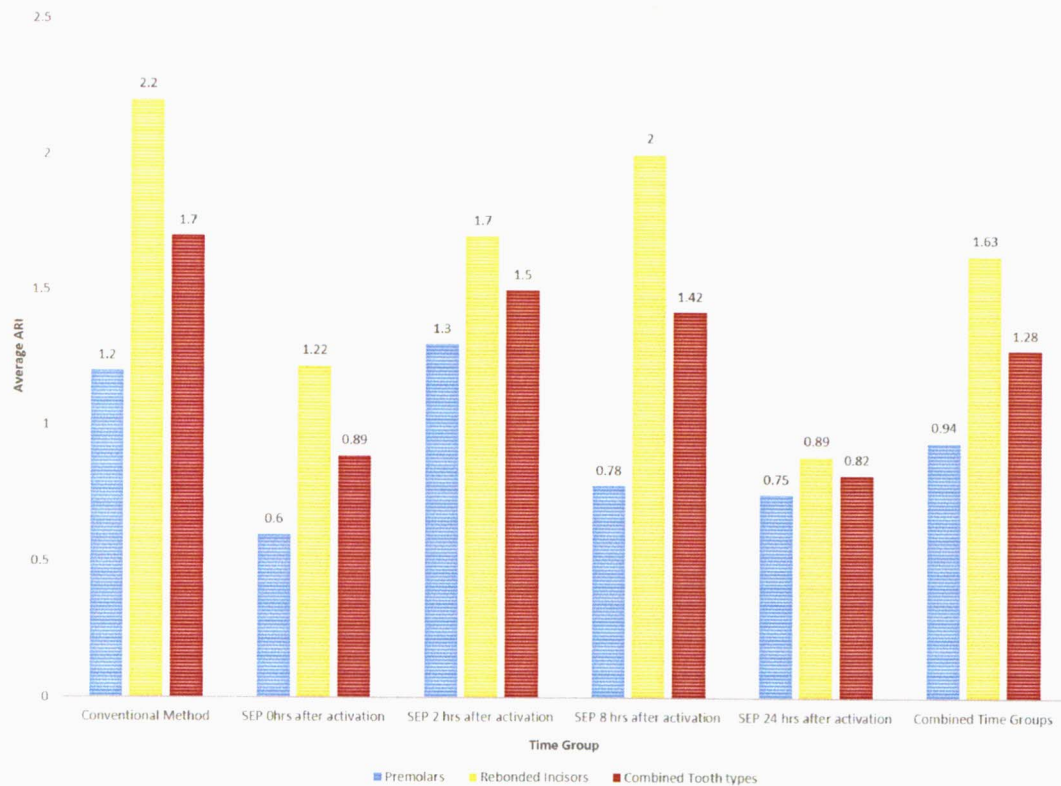
**Table 6.** Frequency Distribution and Statistical Summary of ARI values by tooth type combining all time groups (p value=.0302)

|           | <b>N</b> | <b>0</b> | <b>1</b> | <b>2</b> | <b>3</b> | <b>Mean</b> | <b>Std Dev</b> | <b>Min</b> | <b>Max</b> |
|-----------|----------|----------|----------|----------|----------|-------------|----------------|------------|------------|
| Incisors  | 48       | 4        | 14       | 26       | 4        | 1.63        | 0.76           | 0.00       | 3.00       |
| Premolars | 47       | 14       | 20       | 12       | 0        | 0.94        | 0.76           | 0/00       | 2.00       |

**Table 7.** Frequency Distribution and Statistical Summary of ARI values by time group combining all tooth types

| <b>Time</b>   | <b>N</b> | <b>0</b> | <b>1</b> | <b>2</b> | <b>3</b> | <b>Mean</b>        | <b>Std Dev</b> | <b>Min</b> | <b>Max</b> |
|---|----------|----------|----------|----------|----------|--------------------|----------------|------------|------------|
| Group 1- CM   | 20       | 2        | 4        | 12       | 2        | 1.70 <sup>ab</sup> | 0.80           | 0.00       | 3.00       |
| Group 2- 0 hr SEP   | 19       | 5        | 11       | 3        | 0        | 0.89 <sup>a</sup>  | 0.66           | 0.00       | 2.00       |
| Group 3- 2 hr SEP   | 20       | 0        | 10       | 10       | 0        | 1.50 <sup>c</sup>  | 0.51           | 1.00       | 3.00       |
| Group 4- 8 hr SEP   | 19       | 5        | 3        | 9        | 2        | 1.42               | 1.02           | 0.00       | 2.00       |
| Group 5- 24 hr SEP  | 17       | 7        | 6        | 4        | 0        | 0.82 <sup>bc</sup> | 0.81           | 0.00       | 2.00       |
| a. Groups 1 and 2 are significantly different, p=0.0045<br>b. Groups 1 and 5 are significantly different, p=0.0261<br>c. Groups 3 and 5 are significantly different, p=0.0016 |          |          |          |          |          |                    |                |            |            |





**Figure 9.** Average ARI Values by tooth and time. ARI Scale represents % of adhesive on bracket, as follows: 0= 100%, 1= >50%, 2= <50%, 3= 0%.

A linear mixed model was utilized to analyze the accuracy for statistically comparing the incisors with premolars where the outcome is ARI and the fixed effects were tooth, time, and their interaction, using a random intercept to account for the replicates. A one-way ANOVA with a random intercept was used. For the post-hoc pairwise comparisons, Scheffe's method was applied. The interaction effect in the linear mixed effects model analysis for accuracy was not found to be significant ( $p$ -value = 0.9071), meaning the ARI values for each tooth type at any

given time group were similar. Therefore, the analysis was repeated without the interaction to compare the differences in: 1) tooth groups (consolidating time groups) and 2) time groups (consolidating incisors and premolars). From this newly arranged data, statistics revealed that the ARI values between varying tooth groups as well as 3 different pairs of time groups were significantly different.

## **Discussion**

Statistical analysis of the data collected in this study suggests no significant difference between shear bond strengths of conventional etch and self-etching primer groups. This confirms that self-etching primer performs equally to conventional methods. Researchers have been very interested in shear bond strengths using various bonding materials and techniques, therefore the information associated with this subject is vast. Many studies indicate that while phosphoric acid etching has a significantly larger bond strength, self-etching primer still provides adequate strength to withstand orthodontic forces<sup>5, 34, 72</sup>. Grubisa's research included multiple clinicians and observed that self-etching primer not only produced clinically acceptable, albeit significantly lower (9.8 MPa vs 7.5 MPa), bond strengths, but the bond strengths also differed less between operators than those using phosphoric acid etching; this reinforces the idea that self-etching primer performs more consistently and is less technique-sensitive<sup>35</sup>. In contrast, other studies have found no differences in shear bond strengths between conventional etch and Transbond Plus Self Etching Primer<sup>30, 39, 66, 84</sup>. When using metal brackets, Uysal found no difference in bond strengths (25.5 ±5.1 MPa for CM vs 22.9± 7.3 MPa for SEP)<sup>85</sup>. However, ceramic brackets bonded with phosphoric acid had a significantly greater bond strength (36.7 MPa vs 26.6 MPa), a finding which may further support the use of self-etching primers over conventional acid-etching

considering enamel fracture risks<sup>85</sup>. Surprisingly, there are even studies illustrating greater bond strengths for Transbond Plus Self Etching Primer over phosphoric acid, reporting bond strengths of 16.0 MPa and 13.1 MPa respectively<sup>65</sup>. As there are so many available studies with conflicting statistics, it is difficult for clinicians to draw definitive conclusions. However, nearly all studies showed adequate bond strengths with combined etchant/primers, suggesting that this technique is an acceptable alternative to the current gold standard protocol using phosphoric acid etching.

There have also been a multitude of studies to test shear bond strength specifically of Transbond Plus Self Etching Primer. Table 8 compiles several studies' findings for comparison. A number of studies have found higher average shear bond strengths than this study<sup>49, 65, 72, 86, 87</sup>. Bishara has found an average shear bond strength of 5.9 MPa with a standard deviation of 2.7-3.2 MPa for Transbond Self Etching Primer, which is considerably lower than the bond strengths recorded in this study<sup>88-90</sup>. Several studies' findings concurred with Bishara<sup>5, 26, 35</sup>. Similar bond strengths were found with research testing the variations in shear bond strength with immediate force loading versus a 24-hour delay. This study proved that it is acceptable to load brackets with 120-g of force without delay after bonding, with Transbond Plus Self Etching Primer showing bond strengths of  $7.8 \pm 2.6$  MPa with no

load compared to  $7.5 \pm 1.3$  MPa with force applied <sup>91</sup>. This is assuring, as orthodontists typically place a light wire at the time of bracket bonding.

**Table 8.** Comparison of Average Shear Bond Strength of Transbond Plus Self Etching Primer According to Multiple Studies

| Author  | Sample Size | Tooth Type              | Bracket                      | Average SBS (MPa) |
|---|-------------|-------------------------|------------------------------|-------------------|
| Abdelnaby et al 2010 <sup>91</sup>            | 10          | Human Premolars         | Unspecified metal            | $7.8 \pm 2.6$     |
| Arhun et al 2006 <sup>26</sup>                | 12          | Human Premolars         | Unspecified Metal            | $6.39 \pm 2.87$   |
| Arnold et al 2002 <sup>5</sup>                | 12          | Human Unspecified       | Unspecified Metal            | $9.7 \pm 3.1$     |
| Bishara et al 2004 and 2006 <sup>88, 89</sup> | 20          | Human Molars            | Mx Central Incisor Metal     | $5.9 \pm 2.7$     |
| Bishara et al 2008 <sup>90</sup>              | 20          | Human Molars            | Mx Central incisor Metal APC | $5.9 \pm 3.2$     |
| Buyukyilmaz et al 2004 <sup>65</sup>          | 20          | Human Premolars         | Premolar Metal               | $16.0 \pm 4.5$    |
| Cacciafesta et al 2003 <sup>49</sup>          | 15          | Bovine Incisors         | Mx Central Incisor Metal     | $12.29 \pm 1.37$  |
| Grubisa et al 2004 <sup>35</sup>              | 66          | Human Premolars         | Mx Premolar Metal            | $7.5 \pm 4.2$     |
| Iijima et al 2008 <sup>84</sup>               | 12          | Human Premolars         | Mx Premolar Metal            | $9.74 \pm 1.54$   |
| Pithon et al 2009 <sup>86</sup>               | 30          | Bovine Incisors         | Mx Central Incisor Metal     | $15.42 \pm 2.06$  |
| Rajagopal et al 2004 <sup>56</sup>            | 20          | Human Premolars         | Mx Premolar metal            | $11.10 \pm 2.56$  |
| Scougall-Vilchis et al 2009 <sup>72</sup>     | 35          | Human Premolars         | Premolar Metal               | $16.6 \pm 7.3$    |
| Sorake et al 2015 <sup>24</sup>               | 25          | Human Premolars         | Md Premolar Metal            | $9.67 \pm 1.34$   |
| Vicente et al 2006 <sup>87</sup>              | 25          | Human Premolars         | Mx premolar Metal            | $12.20 \pm 4.27$  |
| Plunkett et al 2016                           | 47          | Human Premolars         | Md Premolar Metal            | $10.92 \pm 3.73$  |
| Plunkett et al 2016                           | 49          | Human Incisors          | Md Incisor Metal             | $11.54 \pm 2.88$  |
| Plunkett et al 2016                           | 48          | Human Rebonded Incisors | Md incisor Metal             | $11.85 \pm 1.20$  |

Through statistical analysis of this study's outcomes, there was no significant difference found between shear bond strengths of any of the groups, including conventional methods or techniques using self-etching primer at any time interval. This was true for both premolars, incisors, and rebonded incisors. Combining all tooth groups, the average shear bond strengths of each time group were: 11.83 MPa for CM, 11.44 MPa for 0-hr SEP, 12.77 MPa for 2-hr SEP, 11.23 MPa for 8-hr SEP, and 11.35 MPa for 24-hr SEP. Not only were there no differences between groups, there was also no pattern in changing bond strengths according to time intervals. The reasoning for the 2-hour interval group having the highest shear bond strength could not be determined.

It is important to note that the outcomes of this study illustrate that the bond strengths in each group tested exceed the widely accepted minimum requirements for an effective orthodontic bond. Of the 144 specimens, only 6 (4.2%) fell below the clinically acceptable range of 5.9-7.8 MPa<sup>38</sup>. This small group with low bond strengths could not be attributed to any specific event, but variations in enamel of the extracted teeth, including undetectable micro-fractures or caries or desiccation, may be to blame. The remaining were well within or above the accepted values.

There is only one other known published study that has tested the impact of storage intervals of Transbond Plus Self Etching Primer after activation on bond strengths. Pithon's study used storage times between 1 and 30 days and observed

no significant differences in bond strengths up to 15 days between activation and application, after which a reduction in bond strengths were seen <sup>86</sup>. The bond strengths recorded in this study were as follows: 15.4 ± 2.1 MPa for 0 days, 14.7 ± 3.3 MPa for 1 day, 14.4 ± 2.4 MPa for 3 days, 14.4 ± 2.3 MPa for 7 days, 12.7 ± 2.5 MPa for 15 days, 11.0 ± 1.8 MPa for 21 days, and 8.4 ± 1.4 MPa for 30 days. This study emphasized the need for re-mixing the primer just prior to application to allow proper mixture of camphorquinone and other components, and this advice was certainly followed in the current study. A need for airtight storage was also stressed, and our study utilized Ziploc bags to ensure no air exposure and avoid evaporation of the water solvent. However, various selections in the design of the previous study necessitated further research. Firstly, bovine teeth were used. While bovine incisors have been shown to have similar properties to human teeth <sup>92</sup>, there have been studies to suggest the difference in tooth specimen may affect bond strength outcomes by 21-44% <sup>93</sup>. Therefore, the results obtained in studies using bovine teeth cannot always be used to draw conclusions about bonding to human enamel <sup>94</sup>. Another variation in study method was the storage of the activated self-etching primer at room temperature as opposed to storage in an 8°C refrigerator. This modification was a conscious decision, as the researcher felt that clinicians may be deterred from applying these findings to their practice if refrigerator storage became a requirement. If this extra step could be eliminated without compromising

outcome, this data would be extremely relevant clinically. The storage times were reduced to what the researcher deemed more compatible with what may be utilized in the average orthodontic practice. Even with these changes, both studies substantiate the idea that self-etching primer applied at various times after activation can still produce viable orthodontic bonds.

The findings of this research suggest that bond strengths would not be compromised if a clinician chose to open a single pack of self-etching primer and use it for any emergencies that may arise within a 24-hour period. Although there is likely a threshold at which the self-etching primer would no longer produce clinically acceptable bond strengths, this study did not extend to that time point.

Bond failure can occur in 3 primary locations during debonding: bracket-resin interface, enamel-resin interface, or within the resin layer. One study examined specimens with scanning electron microscopy and found that nearly all brackets bonded with mechanical retention (similar to our protocol) failed at the bracket-resin interface or left >50% of the adhesive on the enamel surface, equivalent to an Adhesive Remnant Index (ARI) of 2 or 3<sup>95</sup>. Another team researching bond failures validated the above, stating that bonds involving metal brackets with mechanical retention failed primarily at the bracket-resin interface<sup>96</sup>. These results were promising, as bond failures at this interface, or even within the adhesive, are safer when considering risk of enamel damage during the debonding



process<sup>36</sup>. This study recorded ARI values by viewing brackets under a stereomicroscope with 15x magnification. The results obtained show that 75.8% (72 out of 95) resulted in some adhesive remaining on both enamel and bracket (values 1 or 2), suggesting a cohesive failure within the resin layer. While this does not entirely coincide with the previous studies discussed, this outcome is still one of the safer bond failure locations. There were only 3 time-group pairings where significant differences in ARI values were noted: CM's 1.7 vs 0hr SEP's 0.89, CM's 1.7 vs 24hr SEP's 0.82, and 2hr SEP's 1.5 vs 24hr SEP's 0.82. In general, these differences in ARI coincide with increases or decreases in bond strengths (despite no significance noted), with the large ARI values seen in groups with greater bond strengths. This proportional relationship of ARI and bond strengths has been reported elsewhere as well<sup>57</sup>.

When grouped by tooth types, the incisors had a significantly higher ARI value, interpreted as more adhesive remaining on the enamel surface at debond. This may be due to a host of factors, including: the slightly larger bonding surface area of the incisor brackets leading to more resistance at this interface, the relatively flat bonding surface compared to the rounded premolar buccal surfaces allowing more uniform thickness of adhesive, or the fact that the incisors were rebonded before ARI values were recorded and, therefore, the process of removing adhesive from enamel with a bur may affect the future bond and subsequent bond failure location.

Upon further investigation, it was noted that the brackets bonded with self-etching primer had a greater frequency of brackets with 100% adhesive coverage when compared to the conventional group (22.6% vs 10%, respectively). Previous studies have corroborated this finding that self-etching primers tend to leave less adhesive on the enamel compared to conventional etching techniques, postulating that this would facilitate quicker and easier debonding appointments<sup>34, 49</sup>. While the lower average ARI value suggests that self-etching primer is more likely to result in bond failure at the enamel-adhesive interface than phosphoric acid etching, the sample size disparity of CM (N=20) versus combined SEP groups (N=75) could have skewed the results. Also, the ARI scale does not take into account thickness of the adhesive layer remaining on the bracket. While 100% of the bracket base may have a thin layer of adhesive coverage, it is still possible that a cohesive failure occurred and adhesive remained on the enamel surface as well. Therefore, researchers cannot conclusively state bond failure locations based exclusively on this ARI scale. Further research is needed to modify ARI or create a new test that incorporates adhesive thickness as a factor. Until then, it may be prudent for researchers to examine not only the bracket base after debonding, but also examine the enamel surface prior to recording ARI values in order to achieve a more accurate depiction of the interface at which bond failure occurred.

Interestingly, there have been studies testing variations in the instruction protocol for Transbond Plus Self Etching Primer. Research has shown that increasing the application time of the acidic primer from 3-5 seconds up to 15 seconds increases the shear bond strength <sup>79</sup>. However, a report utilizing this longer application time showed that the incidence of enamel fractures became equivalent to that of techniques using conventional phosphoric acid <sup>72</sup>. In contrast, another study shows that increasing the self-etching primer application time up to 15 seconds did not affect shear bond strengths <sup>97</sup>. While this present study employed the recommended self-etching primer application time of 3-5 seconds, there were still several teeth that exceeded the 14 MPa that is considered the threshold at which enamel fracture risk increases <sup>7</sup>. In fact, more teeth bonded with self-etching primer had a shear bond strength greater than 14 MPa than teeth bonded with conventional methods (21.9% vs 6.7%). However, a majority of these were within 1 MPa of the threshold (17 of the 25 bonded with SEP and 2 of 2 bonded with CM). When taking into consideration variability in testing machines and standard deviations, it is likely that only the remaining 8 specimens, or 7.1%, would have the potential increased risk for enamel fracture. This is higher than one study's observed 3% fracture rate with Transbond Plus but still lower than the reported 10% fracture rate for phosphoric acid etch <sup>35</sup>. One additional specimen was thrown out of the study data because an enamel fracture actually occurred (in the 8-HR SEP

Premolar group). In this scenario, however, one must take into account that the process of testing shear bond strengths in vitro lends to a higher chance of fractures, and this fracture may have been avoided in an in vivo test <sup>29</sup>. Future in vivo testing involving bond failure rates and enamel fractures when using self-etching primer applied hours after activation is certainly needed to solidify the clinical viability of this economical protocol.

The differences in the bond strength values of the various presented studies and this current study may be attributed to a host of factors, including variations in the following: tooth specimen selection (human vs bovine, incisor vs molar, etc.), specimen storage medium and duration, bracket base size and design (retention mechanisms), adhesive used in bonding, enamel preparation, debonding techniques utilized (machine and crosshead speed), and study design <sup>57</sup>. A limitation of this current study may be the absence of thermocycling from the study design.

Thermocycling is one of these study variations that may affect final outcomes.

Thermocycling is the process of exposing the bonded specimen to extreme thermal changes in an attempt to simulate the oral environment. This procedure typically rotates the specimens between 5°C and 55°C water baths in 15-30 second increments for 500-5000 cycles. This temperature cycling is recommended in in vitro testing to reflect the effect of the moist conditions of the oral environment on bond strengths <sup>98,99</sup>. Some studies suggest a reduction in bond strengths when the

specimens are subjected to these temperature changes <sup>100, 101</sup>, however other studies depict no change with cycles of thermal conditioning <sup>102, 103</sup>. In fact, one study utilized a thermal cycling method that adds an intermediate temperature of 37°C between the 5°C and 55°C, which likely better simulates temperature changes in the oral environment and reduces thermal shock to the adhesive bond. This study found no effect of this type of 3-step thermocycling on bond strengths <sup>104</sup>. Therefore, with the lack of access and several studies suggesting no impact on bond strengths, thermocycling was not employed in this study. However, further research should be completed adding thermal cycling (at least the 3-temperature process) to this current study design before any definitive conclusions are drawn.

## **Summary and Conclusion**

The primary aim of this study was to determine the effects of increasing time intervals between activation and application of Transbond Plus Self Etching Primer on shear bond strengths. There were no significant differences found among shear bond strengths using the self-etching primer at 0-, 2-, 8-, or 24-hours between activation and application. This is true for premolars, incisors, and rebonded incisors. Bond strengths were also compared to conventional 37% phosphoric acid etching techniques, and no differences were noted. All average bond strengths found were well within the clinically acceptable range of 5.9-7.8 MPa <sup>38</sup>.

Secondarily, bracket bases were examined after debonding to assign Adhesive Remnant Index (ARI) values on a 0-3 scale. Significant differences were noted between tooth types, incisors having a larger ARI than premolars. This suggests more adhesive remains on the enamel after incisors are debonded. There were also 3 pairings of time-groups in which statistics showed significant differences: CM vs 0-hr SEP, CM vs 24-hr SEP, and 2-hr SEP vs 24-hr SEP. These discrepancies follow a general trend of increasing ARI values with increasing shear bond strengths. However, these variations in ARI values are so small that the clinical significance of the differences is likely minimal.

The final outcomes of this study suggest that orthodontists could maintain an opened package of Transbond Plus Self Etching Primer to use for any brackets

requiring bonding or rebonding within a 24-hour period without compromising bond strengths, assuming that cross-contamination is prevented by using a new quik tip applicator with each package insertion. This has the potential to reduce appointment durations as well as eliminate product and financial waste. Future in vivo research is needed to test this idea in a more clinical setting.

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