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Enamel Bond Strength Comparison of Self-Limiting and Traditional Etchant Systems Using a Three-Stage Bonding Technique

Barry Peterson

APPROVED:

[redacted]

Brent Callegari, D.D.S, M.S.D., Supervising Professor and Program Director

[redacted]

Colin Mihalik, D.D.S., M.S.

[redacted]

Curtis Marsh, D.D.S., M.S.

[redacted]

Date

APPROVED: [redacted]

Drew Fallis, D.D.S., M.S., Col, USAF, DC

Dean, Air Force Postgraduate Dental School

Enamel Bond Strength Comparison of Self-Limiting and Traditional Etchant Systems Using a Three-Stage Bonding Technique

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Barry E. Peterson, D.D.S.

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The views expressed in this study are those of the authors and do not reflect the official policy of the United States Air Force, the Department of Defense, or the United States Government. The authors do not have any financial interest in the companies whose materials are discussed in this article.

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DEDICATION

I would first and foremost like to acknowledge God's hand in my life. He has been with me every step of the way. His guidance and reassurance in my life has been ever-present, and for that, I am eternally grateful.

This thesis is dedicated to my family. I would like to thank my parents for all they have taught. Your unconditional love, hard work, and righteous living, have shown me a pattern of living to emulate throughout my life. Thank you for giving me the world.

To my beloved wife Wendi: I know the time and effort put into this education has come at the expense of time spent with you. Because of your love and support, I was able to keep going. Thank you for preparing all of my wonderful lunches and fixing the most amazing dinners, which gave me the energy to carry on. Thank you for being such a wonderful mother and caring for the kids in all of their needs. There were many times over these years when you have been a single parent raising three children and taking care of the household. You exemplify what it means to serve, because you give all your time and effort in order to help others. Thank you for being my best friend! Thank you for being my wife! The journey of life is so much better with you at my side. I love you!!!

To Grant, Kalina, and Celine: I love being your father. You bring a smile to my face when I come home. Please never forget the importance of life and what you can accomplish when you work toward your goals and dreams. With great sacrifices come great rewards. Thank you for your patience, and I hope you know I will be there for you as you fulfill your dreams.

ACKNOWLEDGEMENTS

I would like to thank the supporting staff at the Tri-Service Orthodontic Residency Program. In particular, I would like to thank Dr. Brent Callegari who has been dedicated to the task of helping me write a thesis that is coherent and informative and who provided substantial guidance and encouragement along the way. Additionally Dr. Callegari has been an amazing clinician and educator during my two years as resident. Thank you for being so dedicated to your work and for striving to make each day memorable and fun! Thank you to Dr. Kraig Vandewalle for all of your lab expertise and excellent statistical support. Thank you to both Dr. Colin Mihalik and Dr. Brent Callegari, who have served as research directors and have provided the continued encouragement and guidance to meet all research deadlines.

ABSTRACT

Introduction: Vigilance in the application of phosphoric acid to the tooth surface is necessary to ensure that the tooth is not over-etched. For this reason, the availability of a self-limiting phosphoric acid would be beneficial in cases of indirect bonding. A self-limiting etchant could provide the same clinical results as traditional etchant, yet its self-limiting feature would avoid over-etching the tooth, and therefore, decrease the number of bond failures. **Method**: The facial surface of 150 bovine maxillary incisors were mounted and etched with one of two different etchants (Dentsply 34% Phosphoric Acid or Ultradent's Opal Etch 35%, a Self-Limiting Phosphoric Acid) at varying timed intervals of 15, 30, 60, 90, and 120 seconds. The teeth were then primed, cured, and bonded with resin rods. Shear bond strength and fracture mode were recorded. **Results**: The results of the two-way ANOVA found that Opal Etch had significantly greater bond strengths to enamel compared to Dentsply ($P < 0.001$) with no significant interaction ($p=0.32$). Ninety seconds of etching time resulted in the greatest bond strength, but was not significantly different than 30 seconds. 120 seconds had the lowest bond strength to enamel, but was not significantly different than 15 seconds. A one-way ANOVA was done per etchant. A Bonferronii correction was applied because multiple comparisons were completed ($\alpha=0.025$). For the Opal etchant, a significant difference was found based on time ($p=0.007$). **Conclusions**: The shear bond strength of Opal Etchant consistently tested higher than the Dentsply etchant, but did not prove to be self-limiting.

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I. BACKGROUND

A. Introduction

The desire to bond material to teeth has long been a desire of dentistry throughout the ages. A breakthrough came in 1955 when Michael B. Buonocore brought the idea of etching using acids from the industrial sector into the dental realm in order to increase tooth bonding strength. He noted that industries would etch surfaces with acids in order to create a surface more suitable for bonding paint to metal surfaces (Buonocore 1955). His study revolutionized the field of dentistry and created the method for bonding materials to teeth. The technique took years to refine in order to achieve ideal bond strength and is still being extensively studied today.

In his initial study, Buonocore tested two types of etchant materials: (1) 85% Phosphoric Acid and (2) 50% commercial phosphomolbdate reagent sodium tungstate, containing 10% oxalic acid solution. The teeth etched with 85% phosphoric acid retained the resin acrylic much longer than the commercial phosphomolbdate. However, the science of etching was ahead of composite development and issues quickly arose with micro-shrinkage, micro-leakage, and poor bond strengths of the composite material.

Breakthroughs in resin bonding came again in the 1970's when Retief and Sadowsky reintroduced the idea of etching and bonding teeth with improved composites that significantly reduced micro-shrinkage and micro-leakage. Additionally, this new method of bonding provided strength suitable for bonding brackets to teeth. In fact, it was suggested that all cases could be bonded without banding teeth (Retief et al. 1973, Retief et al. 1975).

Other etchant types have been reported in the literature (see Table 1-1). These include hydrofluoric acid, EDTA, citric acid, and nitric acid. Alternative enamel conditioners were also suggested. These include maleic acid and the newly introduced acidic primers that contain phenyl P. These may be beneficial if they can maintain a clinically useful orthodontic bracket bond strength while decreasing the depth of enamel dissolution (Bishara et al. 1998).

Table 1-1. Types of Etchants (Perdiagão 1995)

Etchants ranked by demineralization potency				
Etchant	**Composition**	**Etch time (sec)**	**pH**	**DID (μm)**
				<2.0
Clearfil CA Agent (Kuraray)	10% citric acid, 20% calcium chloride	15	-0.10	0.5
Gluma 2000 Solution 1 (Bayer)	1.6% oxalic acid, 2.6% aluminum nitrate, 2.7% glycine	15	1.38	0.7
Mirage ABC Condition (Den-Mat)	2.5% nitric acid	15	0.42	0.7
Clearfil CA Agent (Kuraray)	10% citric acid, 20% calcium chloride	40	-0.10	0.9
Amalgambond Universal Dentin Activator (Parkell)	10% citric acid, 3, 3%ferric chloride	10	0.59	1.3
Ultra-Etch (Ultradent)	10% phosphoric acid	15	1.31	1.7
Ultra-Etch	35% phosphoric acid	15	0.02	1.9
				<2.0-3.0
Scotchbond Multi-Purpose Etchant	10% maleic acid	15	0.87	2.1
Mirage ABC Conditioner	2.5% nitric acid	60	0.42	2.2
Mirage ABC Conditioner	10% phosphoric acid	15	*	2.2
Ultra-etch	10% phosphoric acid	30	1.31	2.2
All-etch (Bisco)	10% phosphoric acid	15	0.48	3.0
All-etch without surfactantia (Bisco)	10% phosphoric acid	15	0.78	3.0
All-etch	10% phosphoric acid, with surfactants	15	*	3.0
Scotchbond Etching Gel	35% phosphoric acid	15	-0.28	3.0
				>3.0
Aqueous phosphoric acid solution	10% phosphoric acid	15	0.48	3.2
ESPE Etching Gell	32% phosphoric acid	15	*	3.9
Uni-Etch (Bisco)	32% phosphoric acid with surfactants	15	*	4.0
De Trey Etch	36% phosphoric acid	15	-0.26	4.3
Mirage ABC Conditioner	10% phosphoric acid	30	*	4.5
Etch-Rite (Pulpdent)	38% phosphoric acid	15	-0.29	4.6
Uni-Etch	32% phosphoric acid	15	-0.17	4.8
Aqueous phosphoric acid solution	37% phosphoric acid	15	-0.43	5.0
Kerr Gel Etchant	37.5% phosphoric acid	15	*	5.6

Despite the discoveries of different etchant materials, phosphoric acid continues to remain the gold standard. Regardless of the etchant material used, the effectiveness of etching is dependent upon the following parameters (Gwinnett 1992, Tagami 1998):

- Type of acid used
- Acid concentration
- Etching time
- Form of etchant (aqueous, gel, semi-gel etc.)
- Rinse time
- Activation of the etch (rubbing, agitation, and/or repeated application of fresh etch)
- Enamel instrument preparation beforehand (smear or no smear layer)
- Chemical composition
- Primary tooth enamel versus secondary tooth enamel
- Prism structured enamel or prisimless enamel
- Condition of the tooth (fluoridation, demineralized, decalcification, staining, etc.)

B. Phosphoric Acid Concentrations

Buonocore's 85% phosphoric acid was not the ideal percentage of etch to be used on the tooth. Retief (1973) later reported that teeth etched with 50% phosphoric acid etchant had composite bond strengths that exceeded 600 psi and that this bonding strength would be sufficient for orthodontic brackets, sealants, and restorative dentistry. Further research found that phosphoric acid in concentrations of 30-40% produced very retentive enamel surfaces and increased bond strengths (Silverstone 1975, Lopes 2007). However, there are studies that show concentrations lower than 30% can have similar adhesion values. (Gottleib et al.1982, Gwinnett et al. 1998, Soetopo et al. 1978). Currently, ideal phosphoric acid concentrations are between 30-40%. At these concentrations, it is estimated that the enamel surface loss during etching prior to the bonding of brackets is between 10 and 30 μm (Lopes et al. 2007, Vicente et al. 2006).

C. Ideal Phosphoric Acid Etchant Time

Another question that faced early researchers was the amount of time required to ideally etch the tooth. Initial etchant times were recommended for to be 60 seconds (Sturdevant 2002). Since that time, subsequent research showed etchant times of 15-20 seconds were equally effective (Asmussen et al. 1989, Gwinnett et al. 1992, Lopes et al. 2007, Summitt et al. 1992, Summitt et al. 1993). It must be noted that etchant time must be varied according to the tooth and the situation. For example, if a tooth is suspected of having a high fluoride content, it is advisable to etch the tooth for a total of 25-30 seconds (Anusavice 1996).

Therefore, ideal etchant time can vary from 15-60 seconds (Barkmeir et al. 1986, Nordenvall et al. 1980, Swift et al. 1994). Over-etching is going beyond this point, such that bond strength, specifically shear bond strength, can be compromised. Wang et al. showed there were no statistically significant differences in bond strength among the 15, 30, 60, or 90 second etching times. However, the 120 second group showed significantly less bond strength. Over-etching occurs beyond 60 seconds, in which both tooth structure and bond strength become compromised. Wang showed that between 60-90 seconds, resin-enamel fractures occur and that at 120 seconds, bond strengths significantly decrease. Ideal etchant depth is gauged to be 5-50 μm, as determined by a scanning electron microscope (Lopes et al. 2007, Sturdevant 2002).

Recently, Ultradent (Sandy, Utah) promoted Opal Etch (35% phosphoric acid) as a self-limiting etch. The product claims it will not over-etch the tooth no matter how long it is left on the enamel surface. Ultradent promotes their product as follows on their website (http://www.opalorthodontics.com/adhesives/opalEtch.php):

- Unique self-limiting properties prevent over-etching
- Optimal viscosity with a proprietary surfactant penetrates the smallest fissures without migrating
- Proprietary etch solution rinses off enamel quickly and easily
- Brilliant blue color assures safe, accurate placement and complete removal

Currently there is no public data available or peer reviewed research that has been published to support this claim of being self-limiting.

D. Phosphoric Acid Composition

The initial delivery method for phosphoric acid used a brushing technique to apply the liquid phosphoric acid. While this method was effective for etching the tooth, complaints arose regarding etchant control intra-orally. Gel forms were developed and marketed in order to provide a better and more controlled application of the etchant. The gel forms were made by adding colloidal silica (the same fine particles used in micro-filled composites) or polymer beads to the acid (Anusavice 1996).

When applying gel etchants, one should avoid air bubbles at the interface between the etchant and the tooth. If air bubbles are present, the etchant will not contact the tooth and the bond strength to the tooth will significantly decrease (Anusavice 1996).

E. Bond Strength

While a patient is in treatment, it becomes time-consuming and inefficient to have brackets debond. For this reason, creating a self-limiting etchant will maintain bond strengths at their ideal levels and thereby lead to improved treatment efficiency for patients in orthodontic practices. Bonding orthodontic brackets to tooth enamel has been the standard now for three decades (Dorminey et al. 2003). This is accomplished by micro-mechanical retention, in which resin tags penetrate the etched enamel surface anywhere from 5-50 µm (Sturdevant et al. 2002). Current composite bond strength recommendations range from 3-10 MPa (Maijer and Smith 1981, Reynolds 1979, Ewoldsen et al. 1995).

Research on enamel etching and bonding was at a peak in the late 1970's and early 1980's. Due to these demands, it was suggested that the use of bovine teeth would provide an adequate alternative to human teeth. The adhesive strength to enamel showed no statistically significant difference between bovine and human teeth, although the mean values were always slightly lower with bovine teeth (Nakamichi et al. 1983).

F. Bonding Methods

There are two basic bracket bonding techniques today: (1) multi-step bonding (traditional bonding) and (2) self-etch bonding. A discussion of each follows.

Multi-step bonding involves etching the enamel surface to cause dissolution of interprismatic material, removing the smear layer (if present), and producing an irregular enamel surface (Larmour et al. 2003). After rinsing with water, a primer is applied. The primer is rubbed into the etched enamel surface prior to bonding with an adhesive. The bracket with the adhesive must then be placed and light cured to the enamel. This multi-step approach is the traditional technique and is still used in many orthodontic practices today; however, this technique is time-consuming and technique-sensitive (Williams 2008).

Self-etching primers have gained wide appeal in the orthodontic community because they decrease the chair-time needed to etch and bond. These self-etching materials eliminate the etch step, and manufacturers claim a significant reduction in bonding time, clinical error, and contamination (Cal-Neto et al. 2006, Aljubouri et al. 2003). There is still debate over which system is better; however, both bonding methods are clinically acceptable and have come a long way from their 1955 beginning.

II. OBJECTIVES

To compare shear bond strengths of Opal® Etch™ to a gold standard multi-step system at 15, 30, 60, 90, and 120 second time intervals by measuring the shear bond strength of a resin material bonded to the facial surface of bovine enamel.

III. HYPOTHESIS

Hypotheses: There is a difference in shear bond strength between self-limiting phosphoric acid etchant and regular etchant as etching time increases.

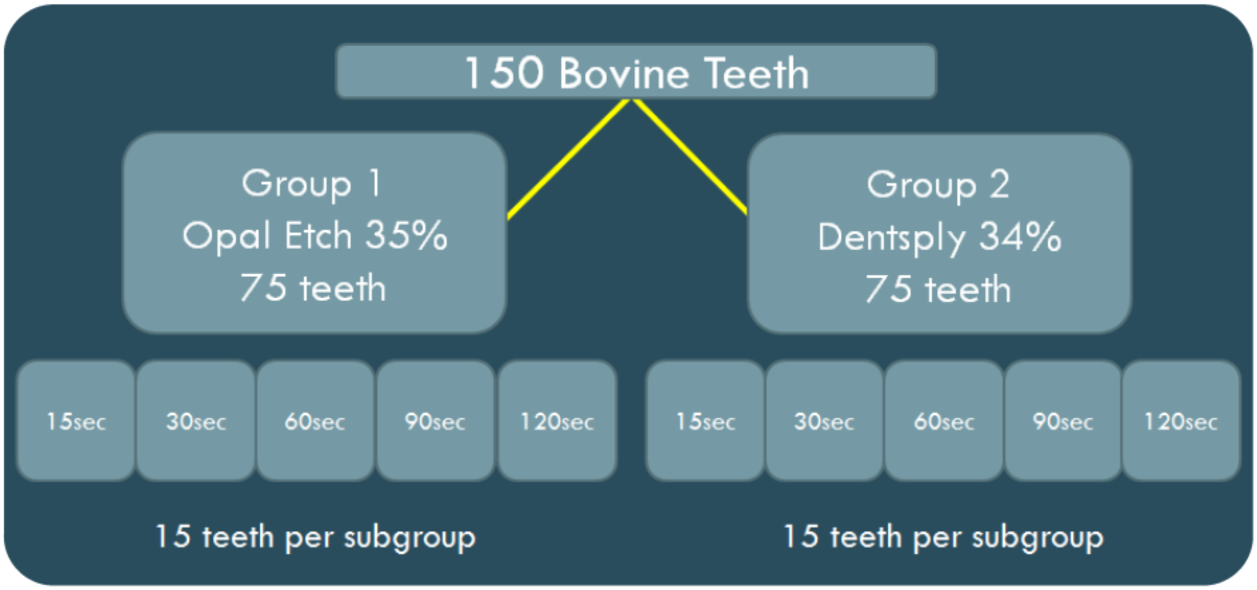
Null Hypotheses: There is no difference in shear bond strength between self-limiting phosphoric acid etchant and regular etchant as etching time increases.

III. MATERIALS AND METHODS

Two hundred bovine maxillary incisors were purchased for this study (Animal Technologies, Inc., a USDA licensed slaughter facility, Tyler, Texas) with the intent of using only 150 teeth. Once extracted, the frozen teeth were shipped in dry ice via FedEx. Upon arrival, the teeth were immediately stored and frozen for one week until they could be prepared for testing. Specimens with internal tooth defects, craze lines, cracks, or chips were removed from the test sample. At the end of the week, the specimens were transferred to a 0.5% chloramine-T solution (using distilled water) and stored at 37° Celsius in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL) to thaw the specimens. The selected 150 teeth were to be tested for shear bond strength.

In order to prepare the bovine teeth, they were removed from the chloramine solution and lingual retention cuts were made in a mesial-distal direction (approximately 1 mm wide, 3-4 mm long, and 2 mm deep) by using a high speed handpiece (Midwest, Quiet-Air, In-Sight) and a tapered diamond bur (Brassler Super Course RCBK #5877K.31.016) (Figure 3-2). Retention cuts allowed each tooth to be mounted in the PVC piping, preventing tooth dislodgement during shear bond strength testing. Once lingual retention cuts were placed in the crown, the root was sectioned at the cementoenamel junction (CEJ). For large bovine incisors, it was necessary to cut the root coronal to the CEJ to allow the specimen to fit within the PVC piping. After retention cut placement and root removal, the teeth were stored in the laboratory oven in a 0.5% chloramine-T Solution (using distilled water) at 37° Celsius. An organizational overview of the research project can be viewed in Figure 3-1.

Figure 3-1. Overview of Research Design



A. Mounting the Teeth

Before mounting, each tooth was removed from the solution and placed on soft boxing plate wax (Regular Size: 1/16” thick x 1-1/2” wide x 12” long [1.59 x 38.1 x 304.8 mm] KINCO® Red Boxing Wax Strips, Avon, Ohio). After placing the tooth in wax, the facial surface of the crown was pressed into the wax so that the tooth was raised above the height of the PVC piping. The PVC piping was then placed around the tooth and Yellow Dental Stone (Golden Microstone, Whipmix, Louisville, KY) was poured into the PVC piping and allowed to set (Figure 3-3). Once set, the red boxing wax was removed and the mounted specimens were placed back into the 0.5% chloramine-T solution and stored in the oven at 37 degrees Celsius (Figure 3-4 and Figure 3.5).

B. Preparing the Teeth for Bonding

The bovine teeth were tested within a six month period following their extraction. The two etchant groups, each containing 75 teeth, were subdivided into five subgroups of 15 specimens. Each subgroup was etched with phosphoric acid using either 35% Opal Etchant or 34% Tooth Condition Gel by Dentsply for the subgroup's designated time (15, 30, 60, 90, and 120 seconds). Two corresponding subgroups were prepared within one hour of each other (i.e. the subgroup for the 15 second Opal Etch and the subgroup for the 15 second Dentsply etch were prepared within one hour of each other). The corresponding subgroup pairs were then tested 24 hours after preparation. Alterations to bonding order between the two corresponding subgroups was performed throughout the testing process (i.e. the 15 second Opal Etch subgroup was prepared prior to the 15 second Dentsply subgroup, but in the next preparing/bonding cycle, the 30 second Dentsply subgroup was prepared prior to the 30 second Opal Etch subgroup, etc.).

Each tooth followed the procedure described below from beginning to end with the only variations being etchant time and etchant type as established by this study. The preparation of the teeth required removing them from the chloramine solution. The facial surface of the crown was flattened using a drill press (Proxxon TBM 115 Type 38128, Luxemburg,) and a diamond course wheel (Brassler diamond course wheel #6909DC.31.040) (Figure 3-6 and Figure 3-7). Each tooth was then sanded flat using 600 grit sand paper (3M Monrovia, CA) by rubbing the enamel surface against the sand paper ten times in a mesial-distal direction and then ten times in an incisal-cervical direction. During the sanding, the PVC was held perpendicular to the sandpaper on a flat surface (Figure 3-8). Each tooth was then rinsed using a heavy stream of tap water for ten seconds (all times were kept using a timer from the Samsung Galaxy Exhibit II, Ridgefield Park, NJ) (Figure 3-9). Each specimen was then dabbed dry using a clean paper towel (Figure 3-10). The designated etchant was then placed over the entire tooth surface and allowed to etch for its designated time (Figures 3-11 and Figure 3-12). All times were kept using a timer (Samsung Galaxy Exhibit II, Ridgefield Park, NJ). After the completed etchant time, each tooth was then rinsed using a heavy stream of tap water for ten seconds (Figure 3-13).

C. Priming and Bonding

After etching, and prior to bonding, the curing light output was verified (L.E.D. Radiometer by Demetron, Kerr Corporation, Orange, California) to verify a consistent energy output per cure (a minimum of 1200 mw/cm^2) (Figure 3-14). A thin layer of Transbond XT Primer Light Cure Adhesive Primer (3M Unitek, Monrovia, California) was applied to the tooth and brushed vigorously on the facial surface of the tooth for three seconds (Figure 3-15). A gentle stream of oil and moisture-free air was used to produce a thin, uniformed layer of primer on the tooth surface by squeezing a plastic bottle three times per tooth (empty 24 oz. plastic bottle with a narrow tip, Next Day Gourmet, Plymouth, Minnesota) (Figure 3-16). A dental curing light (Valo LED Curing Lite, Ultradent, South Jordan, Utah) was used to cure the primer at maximum intensity for three seconds (Figure 3-17). The mounted tooth was then placed in a jig (Bonding Clamp, Ultradent, South Jordan, Utah) and secured beneath a white plastic bonding mold insert (Ultradent, South Jordan, Utah). The bonded area was limited to the 2.4 mm circle diameter determined by the mold. Composite resin (Z250 Filtek, 3M ESPE, St. Paul, Minnesota) was applied incrementally using a modified microbrush to adapt the composite within the plastic mold according to the manufacturer's instructions, to a height of 3 to 4 mm (Figure 3-18 — Figure 3-20). Each incremental layer was polymerized, as recommended by the manufacturer, using the light-curing unit (Figure 3-21). A permanent marker was used to label each specimen to identify its subgroup and bonding order (Figure 3-22). After preparing all the teeth for the shear bond strength (SBS) testing, the specimens were again stored in the laboratory oven in a 0.5% chloramine-T Solution (using distilled water) at 37° Celsius. All samples were stored in the aforementioned solution and oven for one day until all testing was completed 24 hours later.

D. Shear Bond Strength Testing

After 24 hours, the samples were removed from the laboratory oven and placed in a jig (Test Base Clamp, Ultradent, South Jordan, Utah) that held the flat surface of the sample vertical. Tooth specimens were loaded into the test base clamp so as to have the incisal edge of the enamel substrate facing upward (Figure 3-23). The jig was placed into a universal testing machine (Instron 5943R9153, Norwood, Maine) (Figure 3-24). A straight blade was mounted onto the 1 KiloNewton load cell. Prior to the initiation of the test, a crosshead speed of 1mm/min was designated to the Instron. The blade was placed against the flat surface of the test sample. Once the Instron machine setup was complete and the specimen was in position, the test was initiated and the blade descended vertically and was perpendicular to the resin rod until bond failure occurred. The Instron machine records in Newtons the maximum force required to create shear bond failure. Based on the maximum force at the time of the failure, shear bond strength was determined for each sample in megapascals (MPa). Shear bond strength in MPa was calculated from the peak load of failure in Newtons and divided by the specimen surface area (4.41 mm^2). The mean and standard deviation was determined per group and subgroup.

E. Instrumentation

The specifications of the Instron 5943, Norwood, MA are:

- 1 kN (225 lb) capacity
- 1123 mm (44.2 in) vertical test space
- Load measurement accuracy: +/- 0.5% of reading down to 1/1000 of load cell capacity option (2580 series load cells)
- Up to 2.5 kHz data acquisition rate option simultaneous on load, extension, and strain channels
- Speed range of 0.05 - 2500 mm/min (0.002 - 100 in/min)

E. Mode of Fracture Observation

Following shear bond strength (SBS) testing, the specimens were examined under a 20x stereomicroscope (Nikon Microscope Model #553730, Japan) to determine the failure mode as either: 1) adhesive fracture at the adhesive interface, 2) cohesive fracture in the enamel or composite, or 3) mixed (combination of adhesive and cohesive) in enamel and composite.

After shear bond strength testing and fracture mode observation results were recorded, all bovine teeth were disposed of as bio-hazardous waste in accordance with occupational Safety and Health Administration (OSHA) regulations and standard military protocol. Standard safety precautions were followed while handling all teeth.

F. Statistical Analysis

Prior to beginning the research project, a power analysis was calculated and used to anticipate the likelihood that the research project will yield a significant effect, thus providing guidelines to accept or reject the null hypothesis. A power analysis was performed to determine if 15 samples per test group provided sufficient strength in this study. The larger the effect size (ES) used in the power analysis, the larger the sample size. Additionally, the more liberal the criteria needed for an alpha level, the higher the expectation is that the study will yield a statistically significant result.

A two-way ANOVA and Tukey's Post Hoc Test were selected to evaluate the two independent variables of etchant type (2-levels) and time (5-levels) ($\alpha = 0.05$). The only dependent variable is Shear Bond Strength (SBS) measured in Mega Pascal (MPa). The test evaluates the standard deviation (SD) among sample means, and then makes inferences about the differences between population means.

A one-way ANOVA and Tukey's Post Hoc Test was selected to determine if there was a significant difference based on time of etching between the two etchants. A Bonferroni correction was applied because of multiple comparisons between time groups and etchant material ($\alpha=0.025$).

A sample size of 15 teeth per group will provide 80% power to detect an Effect Size (ES) of 0.23 (approximately 0.46 standard deviation (stdev) difference) among means for the main factor of etch material, and a small effect size of 0.29 (or approximately a 0.58 stdev difference) among means for the main factor of time and for the interaction term, when testing with a two-factor ANOVA at the alpha level of 0.05 (NCSS PASS 2002).

G. Figures of Materials and Methods Procedures.

The images of the research procedures are listed and documented in order of their occurrence.

Figure 3-2. Retention cuts placed in the bovine tooth using a high speed handpiece and a tapered diamond bur.

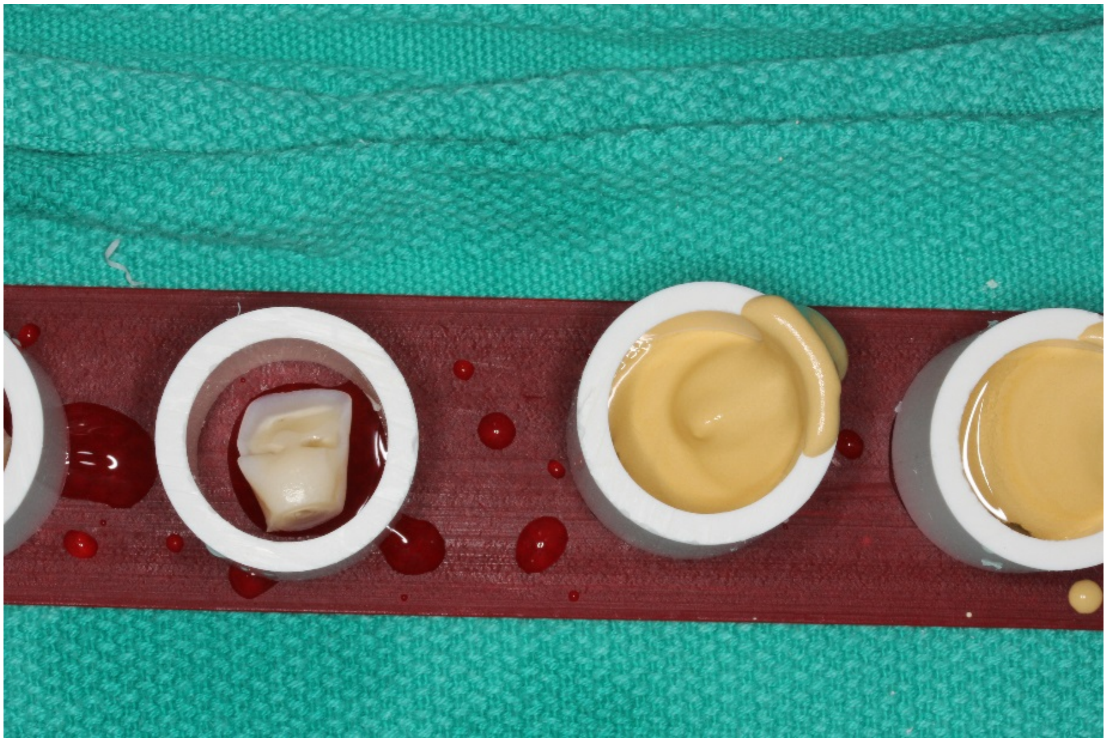


Figure 3-3. Tooth mounting process using box plate wax, PVC piping, and yellow gypsum mounting stone.

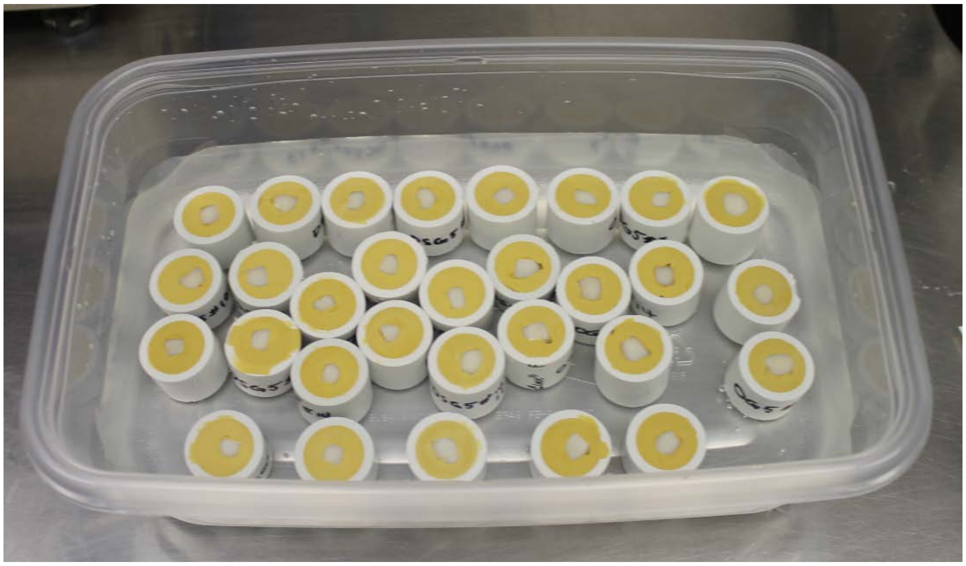


Figure 3-4. Storage of mounted specimens in the 0.5% Chloramine-T Solution.

Figure 3-5. Specimens stored at 37 degrees Celsius in a laboratory oven (Model 20GC, Quincy Lab, Chicago, IL).

Figure 3-6. The facial surface of the crown was flattened using a drill press (Proxxon TBM 115 Type 38128, Luxemburg) and a diamond course bur.

Figure 3-7. The facial surface of the crown was flattened using a drill press and a diamond course bur.

Figure 3-8. Each tooth was sanded flat using 600 grit sandpaper. The specimens were sanded perpendicular to the sandpaper on a flat surface for ten strokes. They were then rotated 90 degrees and sanded in like manner for another ten passes.

Figure 3-9. The teeth were then rinsed using a heavy stream of tap water for ten seconds.

Figure 3-10. The specimens were then dabbed dry using a clean paper towel.

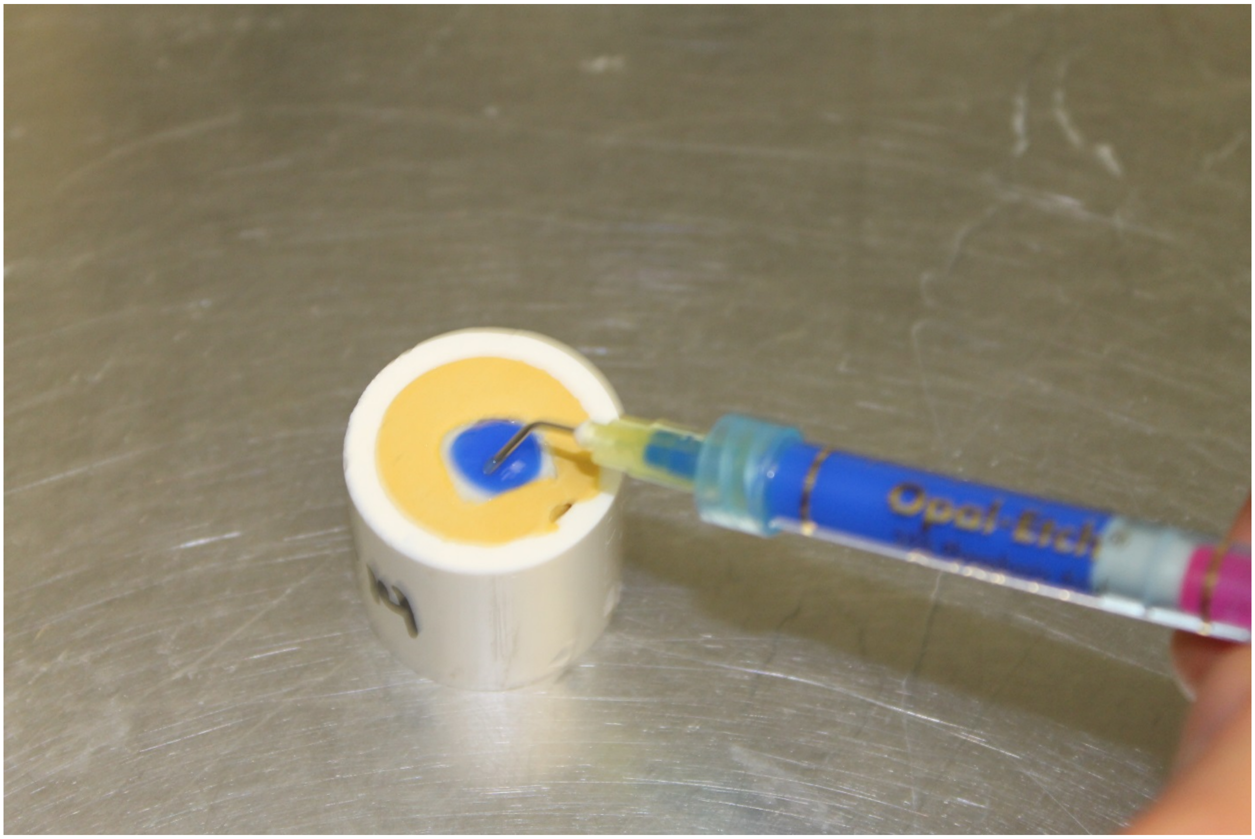


Figure 3-11. Seventy-five teeth had the facial surface of the crown etched using Opal Etch (35% Phosphoric Acid) for its designated time.

Figure 3-12. Seventy-five teeth had the facial surface of the crown etched using Dentsply (34% Phosphoric Acid) for its designated time.

Figure 3-13. After etching, the teeth were then rinsed using a heavy stream of tap water for ten seconds.

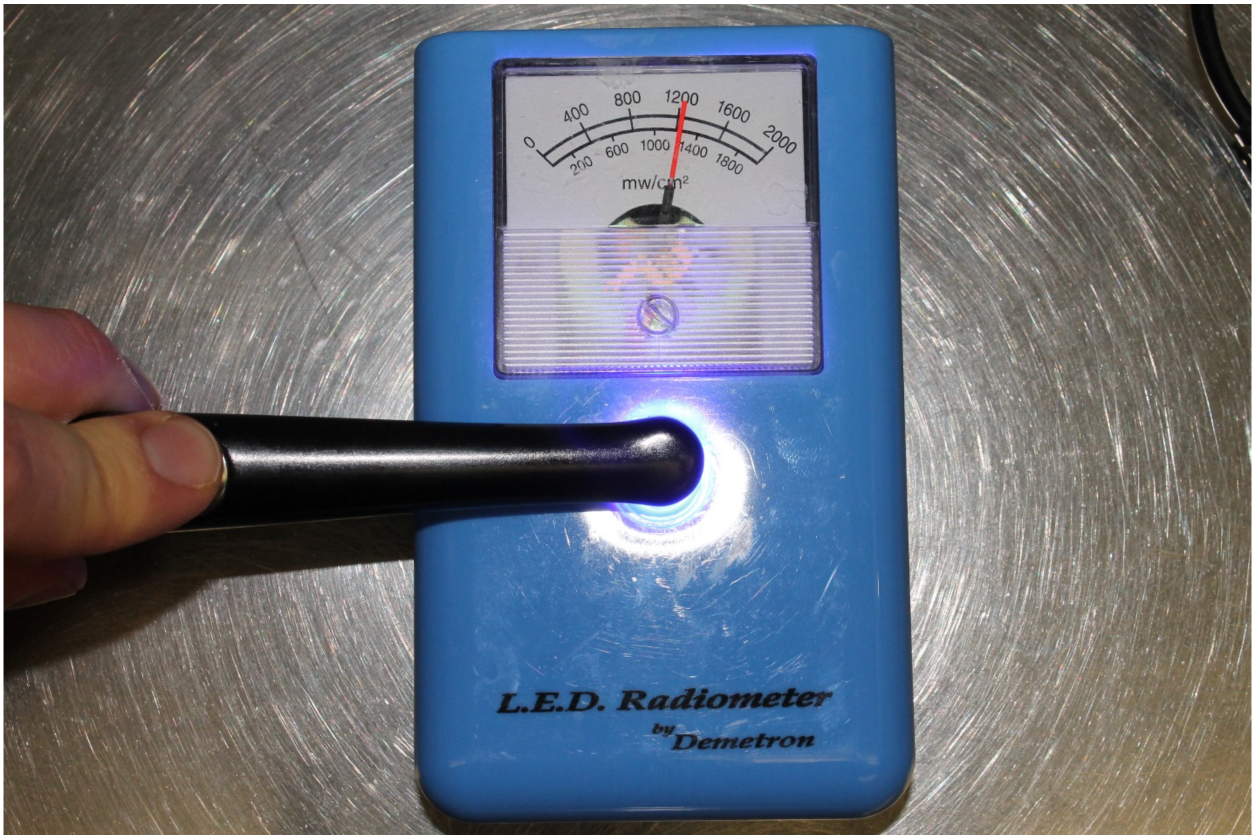


Figure 3-14. Prior to bonding, the curing light was tested using an L.E.D. Radiometer.

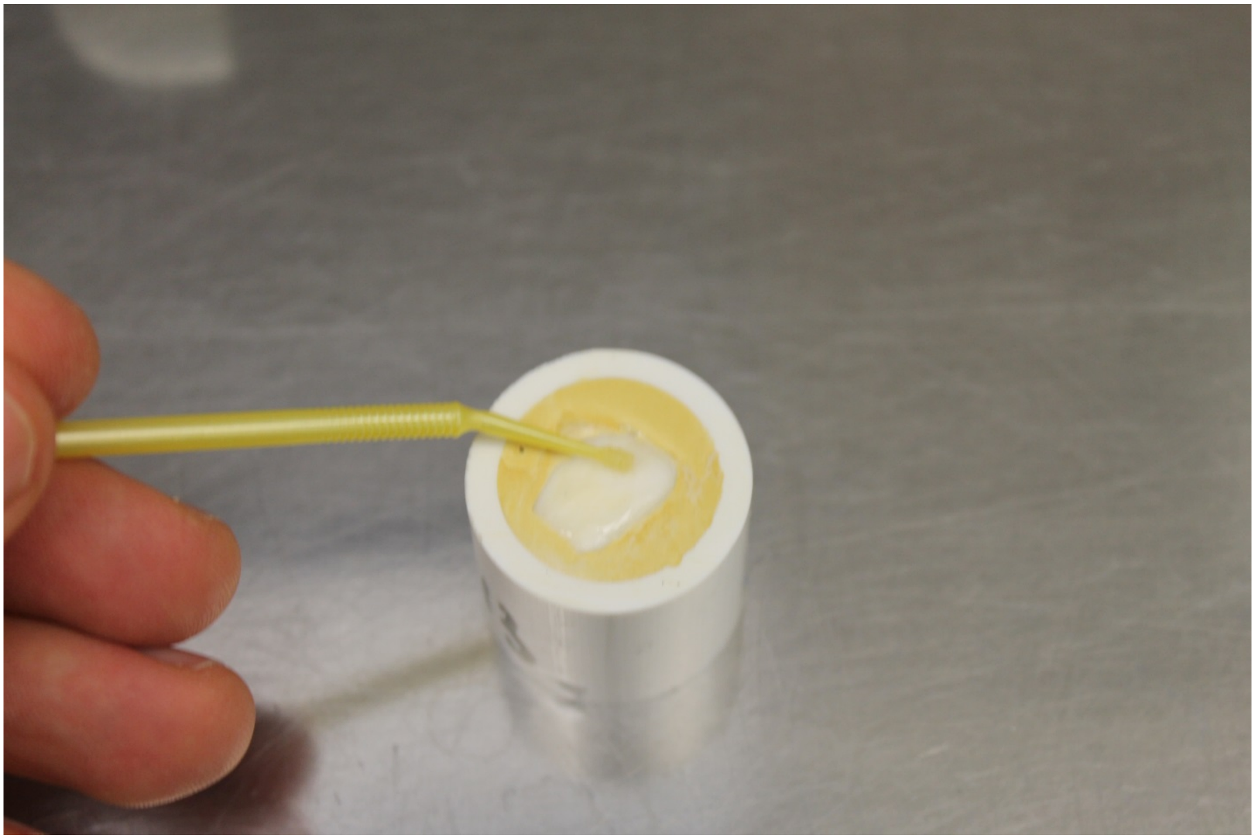


Figure 3-15. A thin layer of Transbond XT Light Cure Adhesive Primer (3m Unitek, Monrovia, CA) was applied to the tooth and brushed vigorously on the facial surface of the tooth for three seconds.

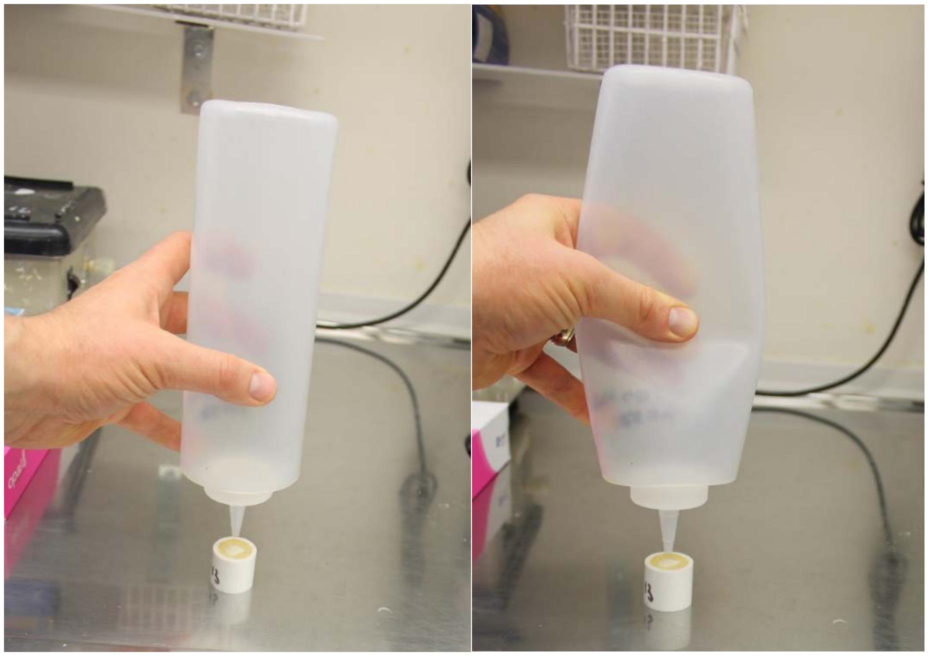


Figure 3-16. A gentle stream of oil and moisture-free air was used to produce a thin, uniformed layer of the primer on the tooth surface. The stream of air was generated by squeezing the bottle three times per tooth.

Figure 3-17. A dental curing light was used to cure the primer at maximum intensity for three seconds.

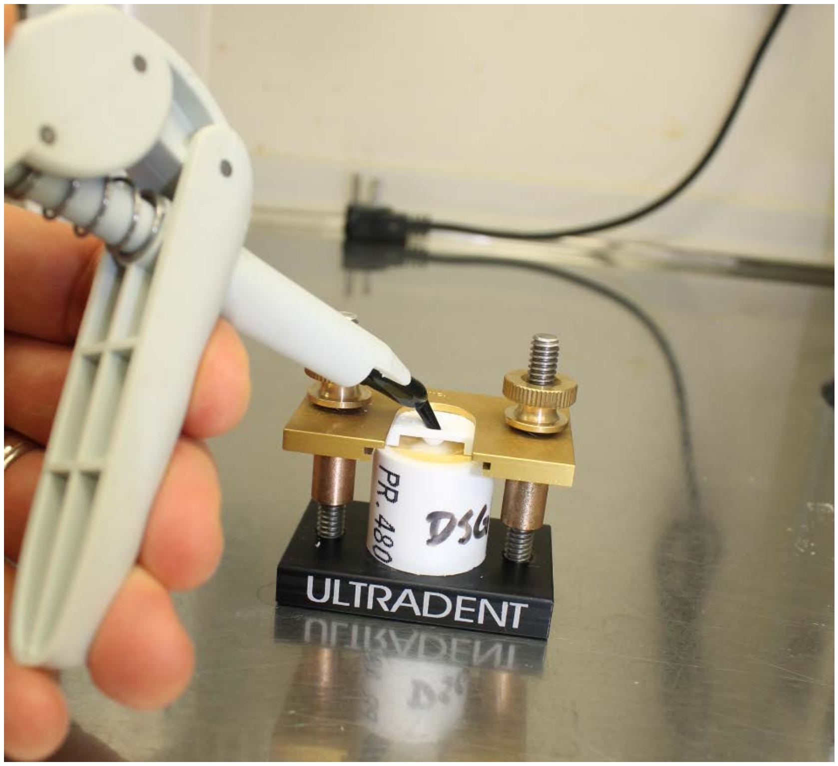


Figure 3-18. The specimen was mounted in the bonding clamp (Ultradent Products, South Jordan, UT) with the accompanying plastic mold. Z250 composite resin (3M ESPE, St. Paul, MN) was applied incrementally into the mold, according to 3M's manufacturer's instructions, to a height of 3 to 4 mm. Each layer was polymerized as recommended by the manufacturer using the light-curing unit.

Figure 3-19. Modified microbrush used to adapt the composite within the plastic mounting mold.

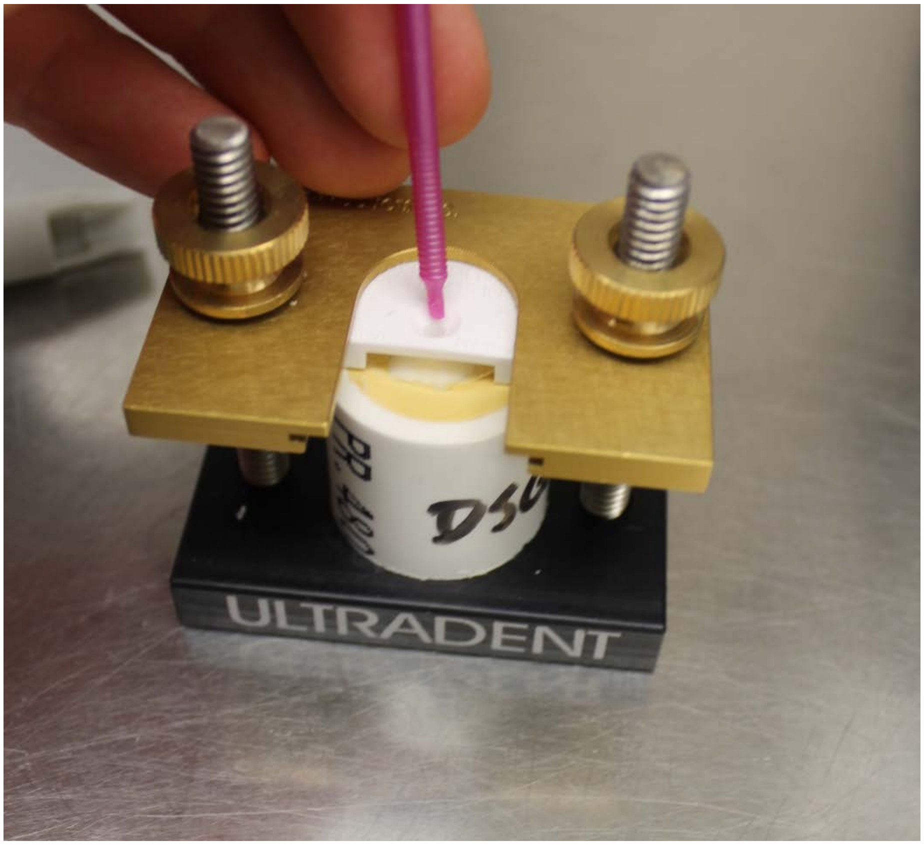


Figure 3-20. Incremental composite placement using a modified microbrush to adapt the composite to the tooth within the plastic mold.

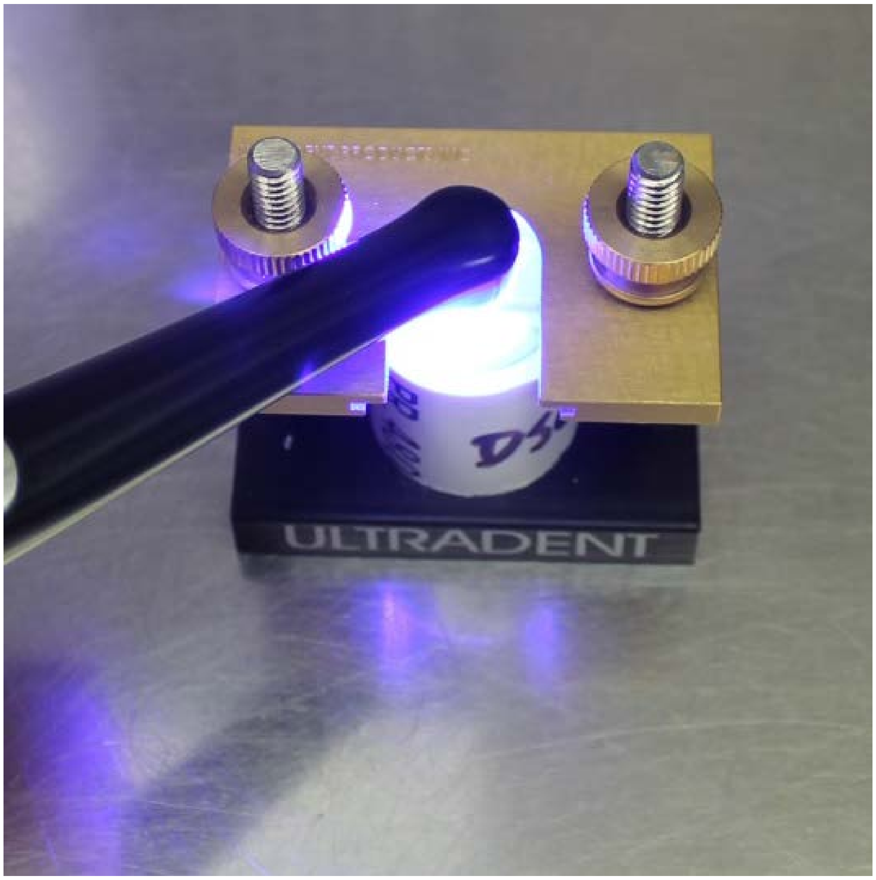


Figure 3-21. Curing light used to cure composite resin incrementally.

Figure 3-22. Composite resin rod after being removed from the mounting jig. Labeling with a permanent marker was done for each specimen to identify the specimen's subgroup and bonding order.

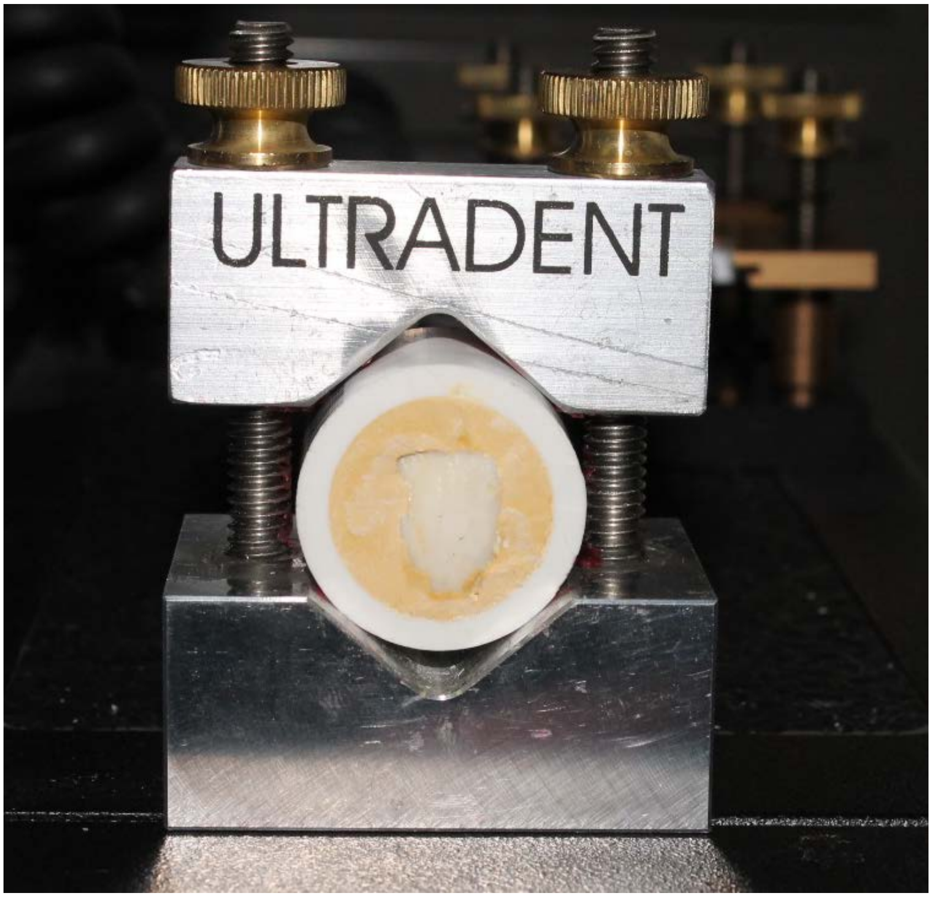


Figure 3-23. Mounted tooth placed in the test base clamp.

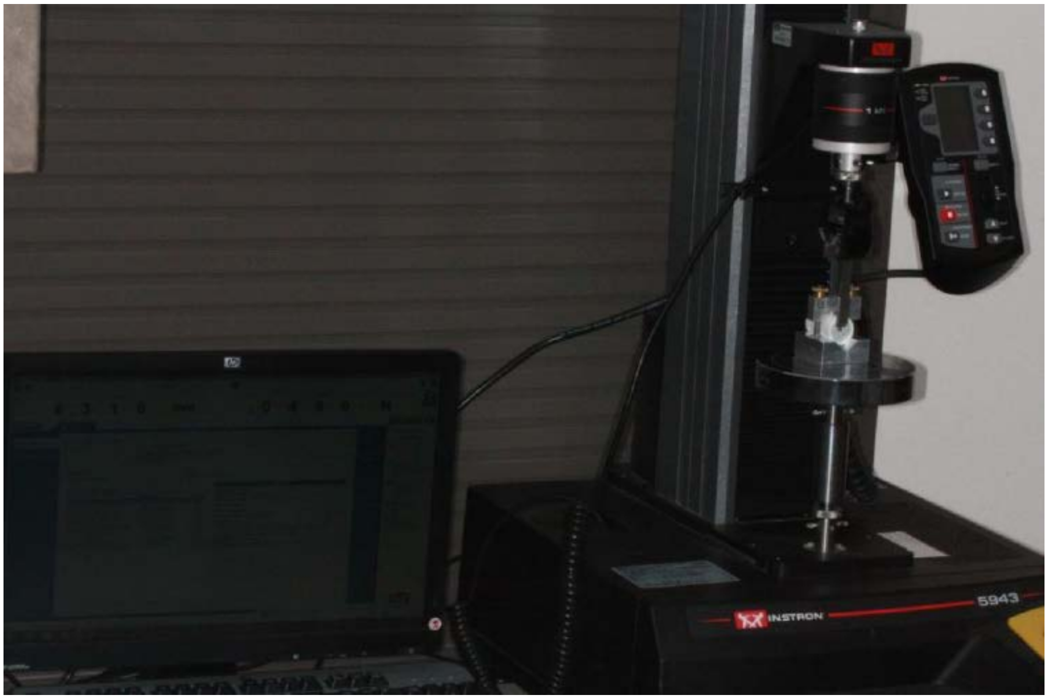


Figure 3-24. Instron machine setup and computer screen monitor

Figure 3-25. Microscope used to evaluate the fracture mode of the resin rods after shear bond strength testing.

IV. RESULTS

Results were collected after subjecting the bonded resin rods to shearing force in the Instron universal testing machine. As the blade attached to the load cell descended at a cross-head speed of 1 mm per minute, it compressed against the resin rod attached to the bovine tooth. The force on the load cell was recorded as the blade continued its downward movement. Once there was a significant drop in the force needed for the blade to continue downward, the blade stopped and the load cell recorded (in Newtons) the maximum force applied. The bovine teeth were then manually examined under a microscope to determine the mode of fracture. Once the fracture of the resin rod was recorded, the force in Newtons was then divided by the surface area of the resin rod (4.41 mm^2) to give the shear bond strength for that sample in Mega Pascals (MPa). The shear bond strength for all samples is recorded in the following tables along with the average shear bond strength and the standard deviation for each group and subgroup (Table 4-1— Table 4-11).

The results of the two-way ANOVA found that the Opal etchant had significantly greater bond strengths to enamel compared to the Dentsply etchant ($p < 0.001$) with no significant interaction ($p=0.32$). Ninety seconds of etching time resulted in the greatest bond strength, but was not significantly different than 30 seconds. 120 seconds had the lowest bond strength to enamel, but was not significantly different than 15 seconds. A one-way ANOVA was done per etchant. A Bonferroni correction was applied because multiple comparisons were completed ($\alpha=0.025$). For the both the Opal and Dentsply etchants a significant difference was found based on time. Both etchants showed a decrease in SBS at 120 seconds which was significantly less than 90 seconds ($p<0.008$) (Table 4-12 and Figure 4-1— Figure 4-3).

Additional non-parametric data was collected based on the fracture mode of the resin rods. A comparison was done to evaluate the mode of fracture and all groups tested similar in the mode of fracture with no significant difference (Table 4-13).

Table 4-1. Opal Etchant Group 1

Opal Etchant Group 1
Time: 15 Sec
Date tested: 16-Jul-13

	N	MPa	Fx Mode
1	84.040	19.06	A
2	81.440	18.47	A
3	112.890	25.60	A
4	95.210	21.59	A
5	77.330	17.54	A
6	84.020	19.05	A
7	85.150	19.31	A
8	102.240	23.18	A
9	75.000	17.01	A
10	54.570	12.37	A
11	69.040	15.66	A
12	76.210	17.28	A
13	105.430	23.91	MC
14	54.960	12.46	A
15	110.140	24.98	A
avg	84.5113333	19.16357	
st dev	18.0454101	4.09193	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-2. Opal Etchant Group 2

	Opal Etchant Group 2 Time: 30 Sec Date tested: 16-Aug-13		
	N	MPa	Fx Mode
1	84.900	19.252	A
2	91.990	20.859	MC
3	95.170	21.580	A
4	74.040	16.789	A
5	108.230	24.542	A
6	70.960	16.091	A
7	90.310	20.478	A
8	128.730	29.190	A
9	90.590	20.542	MC
10	66.760	15.138	A
11	76.180	17.274	A
12	125.880	28.544	E
13	68.010	15.422	A
14	82.740	18.762	A
15	93.380	21.175	A
avg	89.858	20.375964	
st dev	19.03490448	4.3163049	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-3. Opal Etchant Group 3

Opal Etchant Group 3
Time: 60 Sec
Date tested: 20-Jul-13

	N	MPa	Fx Mode
1	144.630	32.796	A
2	79.590	18.048	A
3	139.950	31.735	A
4	65.860	14.934	A
5	89.410	20.274	A
6	115.580	26.209	A
7	77.880	17.660	A
8	114.630	25.993	A
9	101.380	22.989	A
10	62.960	14.277	A
11	125.020	28.349	A
12	59.150	13.413	A
13	77.580	17.592	A
14	103.800	23.537	MC
15	76.190	17.277	A
avg	95.574	21.67211	
st dev	27.62882	6.265038	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-4. Opal Etchant Group 4

Opal Etchant Group 4
Time: 90 Sec
Date tested: 30-Jul-13

	N	MPa	Fx Mode
1	145.110	32.905	MC
2	94.870	21.512	MC
3	108.970	24.710	MC
4	106.230	24.088	A
5	133.580	30.290	MC
6	84.210	19.095	MC
7	130.750	29.649	MC
8	87.130	19.757	MC
9	127.320	28.871	A
10	138.470	31.399	A
11	117.280	26.594	A
12	136.810	31.023	A
13	96.110	21.794	A
14	58.830	13.340	MC
15	93.010	21.091	A
avg	110.5787	25.07453	
st dev	24.82637	5.629562	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-5. Opal Etchant Group 5

Opal Etchant Group 5
Time: 120 Sec
Date tested: 16-Aug-13

	N	MPa	Fx Mode
1	135.970	30.832	A
2	86.290	19.567	A
3	94.080	21.333	A
4	65.350	14.819	A
5	95.840	21.732	A
6	33.580	7.615	A
7	53.380	12.104	A
8	114.250	25.907	A
9	27.690	6.279	A
10	107.300	24.331	A
11	59.760	13.551	A
12	102.140	23.161	A
13	55.950	12.687	A
14	62.900	14.263	A
15	73.580	16.685	A
avg	77.87066667	17.657748	
st dev	30.58707021	6.9358436	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-6. Dentsply Etchant Group 1

	N	MPa	Fx Mode
	Dentsply Etchant Group 1 Time: 15 Sec Date tested: 16-Jul-13		
1	67.210	15.24	A
2	47.500	10.77	A
3	70.080	15.89	A
4	45.910	10.41	A
5	65.280	14.80	A
6	82.920	18.80	A
7	68.440	15.52	A
8	94.070	21.33	A
9	54.920	12.45	A
10	98.100	22.24	A
11	49.870	11.31	A
12	84.700	19.21	A
13	104.390	23.67	MC
14	75.940	17.22	A
15	105.990	24.03	A
avg	74.3546667	16.86047	
st dev	20.235196	4.58848	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-7. Dentsply – Etchant Group 2

Dentsply – Etchant Group 2
Time:30 Sec
Date tested: 24-Jul-13

	N	MPa	Fx Mode
1	73.200	16.599	A
2	80.640	18.286	MC
3	70.600	16.009	A
4	159.580	36.186	MC
5	111.820	25.356	A
6	114.820	26.036	MC
7	75.470	17.113	A
8	64.270	14.574	A
9	47.810	10.841	A
10	87.450	19.830	A
11	74.750	16.950	A
12	67.200	15.238	A
13	100.340	22.753	MC
14	133.600	30.295	A
15	77.800	17.642	MC
avg	89.29	20.247166	
st dev	29.56945794	6.7050925	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-8. Dentsply Etchant Group 3

Dentsply Etchant Group 3
Time: 60 Sec
Date tested: 20-Jul-13

	N	MPa	Fx Mode
1	47.360	10.74	A
2	70.080	15.89	A
3	88.080	19.97	A
4	66.890	15.17	A
5	80.580	18.27	MC
6	74.930	16.99	A
7	60.700	13.76	A
8	40.300	9.14	A
9	61.480	13.94	A
10	124.390	28.21	EM
11	72.390	16.41	A
12	92.660	21.01	A
13	103.710	23.52	A
14	100.530	22.80	A
15	55.790	12.65	MC
avg	75.99133	17.23159	
st dev	22.73439	5.15519	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-9. Dentsply Etchant Group 4

	Dentsply Etchant Group 4 Time: 90 Sec Date tested: 30-Jul-13		
	N	MPa	Fx Mode
1	112.510	25.51	A
2	105.530	23.93	A
3	102.650	23.28	MC
4	87.550	19.85	MC
5	109.930	24.93	A
6	79.800	18.10	A
7	64.190	14.56	MC
8	68.170	15.46	MC
9	131.560	29.83	MC
10	103.560	23.48	A
11	80.470	18.25	A
12	126.510	28.69	A
13	96.570	21.90	A
14	42.740	9.69	A
15	144.370	32.74	MC
avg	97.074	22.01224	
st dev	27.22251	6.172905	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-10. Dentsply Etchant Group 5

Dentsply Etchant Group 5
Time: 120 Sec
Date tested: 16-Aug-13

	N	MPa	Fx Mode
1	49.250	11.17	A
2	37.820	8.58	A
3	31.230	7.08	A
4	34.180	7.75	A
5	96.210	21.82	A
6	60.410	13.70	A
7	27.050	6.13	A
8	21.100	4.78	A
9	76.490	17.34	A
10	26.310	5.97	A
11	26.840	6.09	A
12	89.860	20.38	A
13	79.900	18.12	A
14	28.970	6.57	A
15	76.410	17.33	A
avg	50.802	11.519728	
st dev	26.41617097	5.9900614	

Fx Mode= fracture mode

A= adhesive fracture only

MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Table 4-11. Mean and Standard Deviations

Descriptive Statistics Dependent Variable: MPA				
ETCHANT	**TIME**	**Mean**	**Std. Deviation**	**N**
Dentsply	**120**	11.5197	5.9901	15
	15	16.8593	4.5878	15
	30	20.2472	6.7051	15
	60	17.2316	5.1552	15
	90	22.0122	6.1729	15
	Total	17.5740	6.6717	75
Opal	**120**	17.6567	6.9361	15
	15	19.1647	4.0928	15
	30	20.3747	4.3158	15
	60	21.6720	6.2652	15
	90	25.0740	5.6287	15
	Total	20.7884	5.9710	75
Total	**120**	14.5882	7.0913	30
	15	18.0120	4.4297	30
	30	20.3109	5.5408	30
	60	19.4518	6.0728	30
	90	23.5431	6.0095	30
	Total	19.1812	6.5126	150

Table 4-12. Shear Bond Strength MPa (Stdev) Results

Etchant	Shear Bond Strength MPa (Stdev)* Time (sec)					Total
	Sub-Group 1	Sub-Group 2	Sub-Group 3	Sub-Group 4	Sub-Group 5	
	15 sec	30 sec	60 sec	90 sec	120 sec	
Opal	19.2 (4.1)^{ba}	20.4 (4.3)^{ba}	21.7 (6.3)^{ba}	25.1 (5.6)^{a}	17.7 (6.9)^{b}	20.8 (6.0)^{A}
Dentsply	16.9(4.6)^{ab}	20.2(6.7)^{a}	17.2 (5.2)^{ab}	22.0(6.2)^{a}	11.5 (6.0)^{b}	17.6 (6.7)^{B}
Total	18.0(4.4)^{bc}	20.3(5.5)^{ab}	19.5 (6.1)^{b}	23.5 (6.0)^{a}	14.6 (7.1)^{c}	

*Groups with the same lower case letter by row are not significantly different. Groups with the same upper case letter per column are not significantly different.

Table 4-13. Fracture Mode Results

Fracture Mode	Fracture Mode Percentage (Number out of 15) Time (sec)				
	Sub-Group 1	Sub-Group 2	Sub-Group 3	Sub-Group 4	Sub-Group 5
	15 sec	30 sec	60 sec	90 sec	120 sec
Opal					
A	93% (14)	80% (12)	93% (14)	53% (8)	100% (15)
MC	7% (1)	13 %(2)	7% (1)	47% (7)	
CC					
EM					
E		7% (1)			
Dentsply					
A	93% (14)	73% (11)	80% (12)	60% (9)	100% (15)
MC	7% (1)	27% (4)	13 %(2)	40% (6)	
CC					
EM			7% (1)		
E					

Fx Mode= fracture mode

A= adhesive fracture only

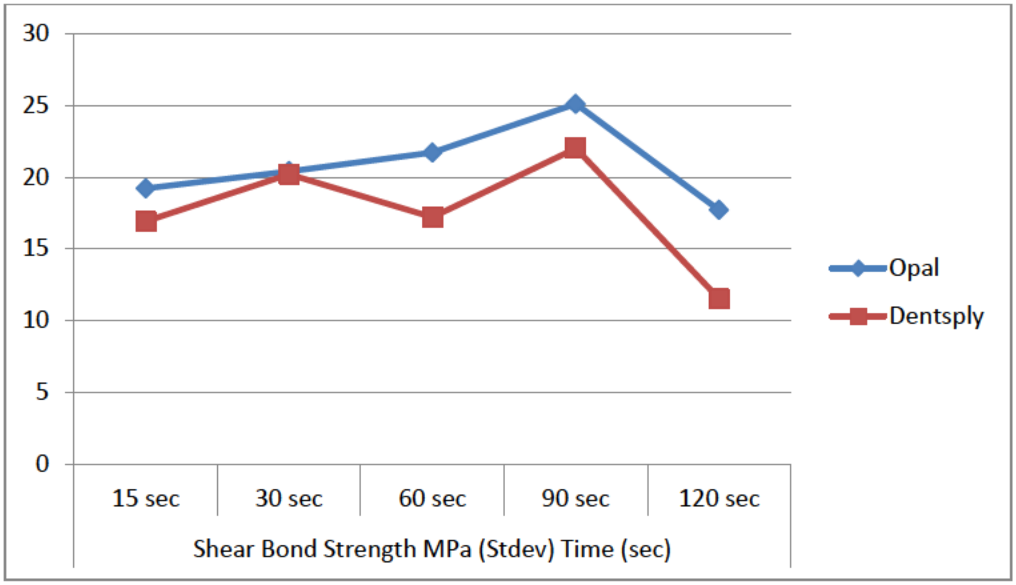
MC= mixed cohesive composite and adhesive fracture

CC= cohesive composite fracture only

EM=mixed adhesive and enamel fracture

E=fracture occurred within enamel only

Figure 4-1. Line Graph of Shear Bond Strength (SBS) Results



Opal etchant had significantly greater bond strengths to enamel compared to Dentsply ($P < 0.001$) with no significant interaction ($p=0.32$). Ninety seconds of etching time resulted in the greatest bond strength for both etchants, but was not significantly different than 30 seconds. 120 seconds had the lowest bond strength to enamel, but was not significantly different than 15 seconds.

Figure 4-2. Opal Shear Bond Strength Results

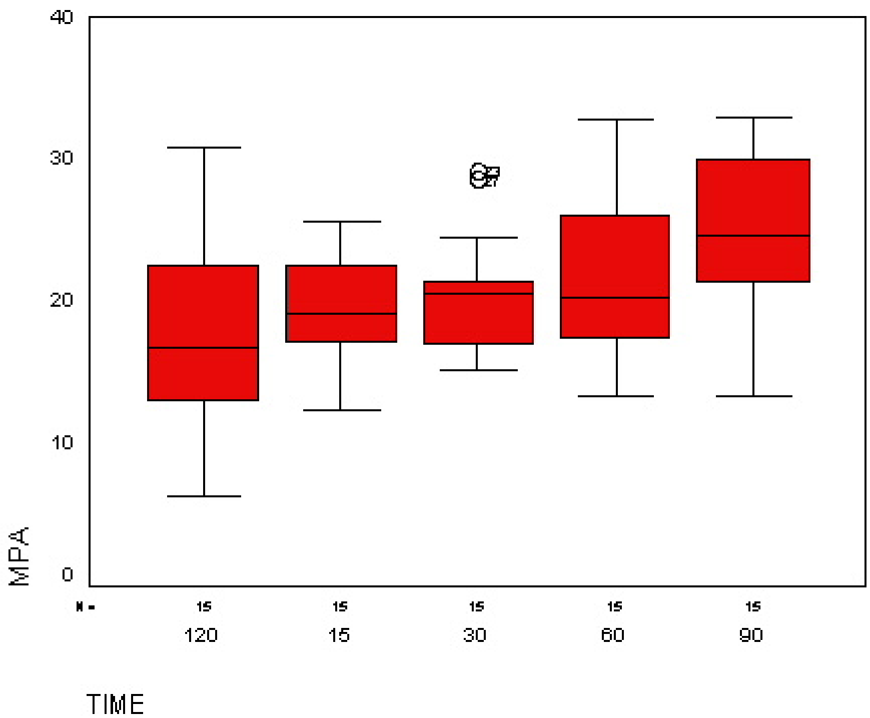
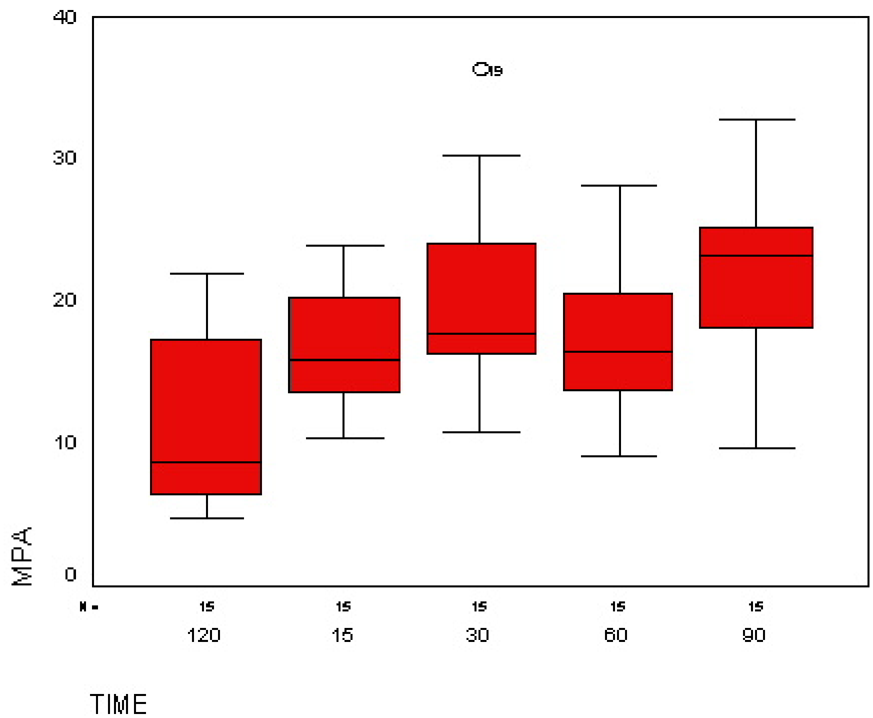


Figure 4-3. Dentsply Shear Bond Strength Results



V. DISCUSSION

Vigilance in the application of phosphoric acid to the tooth surface is necessary to ensure that the tooth is not over-etched. For this reason, the availability of a self-limiting phosphoric acid would be beneficial in cases of indirect bonding. A self-limiting etchant could provide the same clinical results as traditional etchant, but the self-limiting feature would avoid over-etching the tooth and therefore, decrease the number of bond failures.

It has been demonstrated that over-etching the tooth (etching for more than 60 seconds) significantly decreases bond strength and enamel is removed in the process (Barkmeir et al. 1986, Nordenvall et al. 1980, Swift et al. 1994, Wang et al. 1991). If self-limiting etchant works properly and does not over-etch the tooth, then one could potentially etch all of the teeth in the mouth without the worry of over-etching, or excessive removal of enamel, which would decrease bond strength. This could be especially useful for orthodontists who use indirect bonding methods and would like to etch and bond one single arch at a time. It would be convenient and reassuring to not be preoccupied with the amount of time the etchant remains on the tooth.

As far as the authors are aware, only one company (Ultradent) claims to have a self-limiting phosphoric acid etchant on the market. Ultradent produces two 35% self-limiting phosphoric acid etchants, Opal Etch® and Ultra-Etch®. According to company representatives, the etchants are exactly the same product, but are marketed differently. Opal Etch® is marketed to orthodontic customers and Ultra-Etch® is marketed to restorative customers.

This study sought specifically to compare etchants of similar strength. Dentsply 34% phosphoric acid was selected to serve as a control because it was similar in strength to

Opal Etch 35% phosphoric acid. Efforts were made to ensure all other parameters of the study were constant so as to evaluate any interaction between the two independent variables of etchant type (2-levels) and time (5-levels).

In 1991, Wang et al. tested 37% phosphoric acid etchant (no self-limiting etchants were tested) and showed there were no statistically significant differences in bond strength among the 15, 30, 60, or 90 second etching times. However, the 120 second group showed significantly less bond strength. This study was modeled after the Wang et al. study with a few key differences:

1. Bovine teeth were used instead of human teeth. Wang et al. used human teeth from children ages 9-16. Due to the number of teeth needed for this project, it was determined that bovine teeth were an adequate substitute. Nakamichi et al., in 1983, demonstrated the adhesive strength to enamel showed no statistically significant difference between bovine and human teeth with any of the materials used, although the mean values were always slightly lower with bovine teeth.
2. Resin rods were used instead of brackets. Brackets are design to debond from its composite base prior to the composite debonding. Manufacturers design orthodontic brackets in this way to prevent enamel fractures by having the failure occur at the composite bracket interface rather than at the tooth composite interface. However, in this study, it was determined that the etchant needed to be evaluated at the enamel-adhesive level. To enhance the evaluation, a resin rod bonded directly to the facial surface would remove the variability the bracket would introduce.

3. Shear bond strength was tested instead of tensile bond strength. It seemed logical that most occlusal forces on composite and bracket systems used in orthodontics comes from shear forces. For this reason, it was determined that a shear bond strength test would be most appropriate for this study design.
4. The tooth surface was flattened on the bovine teeth due to the variance in enamel tooth presentation. Bovine central incisors are flat and have deep fissures on the facial surface. However, the lateral bovine teeth differ in that they have a relatively smooth, conical surface. In order to eliminate variance among testing samples, the tooth surface of all teeth were sanded with a bur and sandpaper to standardize the surface for the resin rod attachment.

The results of the two-way ANOVA with a Tukey's post hoc test found that Opal Etch had significantly greater bond strengths to enamel compared to Dentsply ($P < 0.001$) with no significant interaction ($p=0.32$). Ninety seconds of etching time resulted in the greatest bond strength, but was not significantly different than 30 seconds. One-hundred-twenty-seconds had the lowest bond strength to enamel, but was not significantly different than 15 seconds for both etchant materials.

A one-way ANOVA with a Tukey's post hoc test was completed to compare bond strengths between the Opal etchant and the Dentsply etchant. The shear bond strength of Opal Etch was significantly stronger in every test group compared to that of Dentsply ($p=0.007$). Additionally the shear bond strength between etchants both behaved the same. The shear bond strength for both Dentsply and Opal etch decrease significantly between 90 seconds and 120 seconds. If Opal etch had been truly self-limiting it would have been expected that the bond strength would have remained constant from 90-120 second intervals.

Non-parametric data was collected based on the fracture mode of the resin rods. A comparison was completed and tested similarly with respect to the mode of fracture with no significant difference (Table 4-13).

These results from the two-way ANOVA, one-way ANOVA and the fracture mode analysis do not support the claim that Opal Etch is self-limiting as it performed similar to the control etchant (Dentsply) in shear bond strength patterns and fracture mode. Based on this data, the null hypothesis was accepted.

VI. CONCLUSION

1. The null hypothesis was accepted in that Opal Etch ® is not self-limiting and will continue to etch the tooth if left on the enamel surface past 60 seconds.
2. Opal etchant had significantly greater bond strengths to enamel compared to the Dentsply etchant ($P < 0.001$) with no significant interaction ($p=0.32$).
3. Ninety seconds of etching time resulted in the greatest bond strength, but was not significantly different than 30 seconds.
4. 120 seconds had the lowest bond strength to enamel, but was not significantly different than 15 seconds.
5. The recommendation to clinicians is to continue following literature guidelines. For phosphoric acid etchant strengths of 30-40% and initial etchant time of 15-30 is recommended. Adjust the etchant time as needed for individual teeth. This study provides no data indicating that etching times greater than 60 seconds provides any added benefit. Although bond strengths in this study were highest at 90 seconds, possible fracture modes in the enamel may be detrimental to the tooth. Further studies would be needed in this area to change current literature recommendations.

VII. LITERATURE CITED

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