

THE EFFECT OF PHOSPHORIC ACID ETCH IN CONJUNCTION WITH SELF-
ETCHING PRIMER ON ENAMEL SURFACES AND RESULTANT
ORTHODONTIC BRACKET BOND STRENGTH

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MICHAEL BRUCE OLSEN

B.S., University of Lethbridge, 2006
D.D.S., West Virginia University, 2011

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Michael Bruce Olsen, Candidate for the Master of Science Degree

University of Missouri-Kansas City, 2013

ABSTRACT

This study examined the methods of preparing the enamel surface for orthodontic bracket adhesion in overcoming pellicle and possible fluid contamination from prebonding cleaning techniques and the effect of these treatments on the enamel surface and resultant bond strength. Shear bond strength testing, Raman linear mapping, and scanning electron microscopy was used to evaluate shear bond strength, depth of resin interface, and surface and cross-sectional characteristics respectively.

Based on an analysis of variance ($\alpha=.05$), there was a significant effect of pretreatment enamel conditioning prior to self-etching primer application on shear bond strength. This difference was found to exist solely between the pumice prophylaxis group and the 34% phosphoric acid etch group. No difference was found to exist across all groups with regards to depth of resin penetration. Scanning electron microscopy showed no significant differences in enamel surface or resin/enamel interface characteristics.

Results suggest that enamel cleaning techniques prior to SEP application are unnecessary and even deleterious to bond strength and resin penetration.

APPROVAL PAGE

The faculty listed below, appointed by the Dean of the School of Dentistry, have examined a thesis titled “The Effect of Phosphoric Acid Etch in Conjunction with Self-etching Primer on Enamel Surfaces and Resultant Orthodontic Bracket Bond Strength” presented by Michael Bruce Olsen, candidate for the Master of Science degree, and certify that in their opinion it is worthy of acceptance.

Supervisory Committee

Jeffrey Nickel, D.D.S., M.Sc., Ph.D., Committee Chair
Department of Orthodontics and Dentofacial Orthopedics
Department of Oral and Craniofacial Sciences

Mary P. Walker, D.D.S., Ph.D.
Department of Oral and Craniofacial Sciences

Yong Wang, Ph.D.
Department of Oral and Craniofacial Sciences

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CHAPTER 1

INTRODUCTION

With the advent of enamel bonding agents and fixed orthodontic appliances, finding a balance of efficiency in placement while achieving sufficient bond strength is a concern for orthodontists who strive to minimize bond failures. The untimely failure of bracket bonding during treatment leads to extra office visits, subsequent increased overhead costs, and potentially longer treatment times. In an attempt to minimize bond failures, orthodontists and manufacturing companies have sought out ways to ensure adequate bond strengths while still maintaining an efficient procedure with the least invasive nature of the bonding procedure.

Enamel Bonding

Enamel Structure

Dental enamel constitutes the outermost layer of a tooth that can be seen clinically when looking into the oral cavity. It consists of inorganic matrix of hydroxyapatite crystals making up about 96 % of the content with the remaining 4% made up of water and organic proteins such as amelogenin, ameloblastin, and enamelin. The basic unit making up the enamel structure is termed the enamel rod. During formation of the tooth ameloblasts migrate away from the dentinoenamel junction forming rod-shaped structures. Each enamel prism (rod) is composed of tightly packed hydroxyapatite crystals measuring about 4-8 μm in diameter arranged perpendicular to the underlying tooth structure (Ross et al. 2006). These enamel rods can be demineralized with acid to create a porous external surface which can be penetrated by dental resins to create a micromechanical bond (Buonocore 1955). Currently phosphoric acid in the range of 32%-37% is being used to etch the enamel surfaces.

Dental Resins

Resin use in dentistry as a restorative bondable material became more practical in the 1960's as dental composites (Rueggeberg 2002). A dental composite consists of resin-based monomers, as well as inorganic filler particles such as silicon dioxide (Gioka et al. 2005). The addition of a high-molecular weight, difunctional monomer, such as bisphenol A-glycidyl dimethacrylate (bis-GMA), aided in the development of dental composites and the addition of inorganic fillers (Rueggeberg 2002). The vinyl end unit of C=C double bonds of bis-GMA undergoes addition polymerization which form cross-links amongst the monomer units to create a more rigid polymer structure. Two common monomers used in orthodontic adhesive resins were bisphenol A-glycidyl methacrylate (bis-GMA) and triethylene glycol dimethacrylate (TEGDMA), which is less viscous. Current adhesives contain both bis-GMA and bisphenol A-bis (2-hydroxyethyl ether) dimethacrylate. The fillers are made of ground quartz or silica glass and are coated with silane to allow the hydrophilic nature of the inorganic fillers to be incorporated into the hydrophobic polymerizing resin matrix. The addition of inorganic filler particles increases the strength of the resin matrix and reduces the contraction due to polymerization. As such, much of the development of composite materials focused on these two areas of interest: polymerization method and filler materials (Rueggeberg 2002).

Currently most orthodontic brackets are bonded to the enamel surface using photopolymerization, or "light-curing". Radiant addition polymerization of the resin usually occurs with the presence of a photoinitiator, usually camphorquinone, after exposure to wavelengths of light between 400 and 500nm (Rueggeberg 2002). When camphorquinone is exposed to the appropriate wavelength of light, it converts to a high energy excited state and

creates free radicals when in contact with amine groups of the monomer. These free radicals have the ability to link the monomer molecules initiating the process of polymerization. The ability to initiate the polymerization reaction when activated by light source has allowed clinicians the convenience of a longer working time promoting greater precision and convenience of bracket placement.

As per filler content, most orthodontic resins used are low in filler content compared to restorative dental composites. The low filler content allows the orthodontic bracket to be more easily compressed against and integrated into the etched enamel surface yet still provide adequate strength and workability. However, low filler content has the disadvantage of increased polymerization shrinkage causing microleakage and a weaker bond (James et al. 2003).

Enamel/ Resin Interface

Direct bonding to enamel was first instituted in dentistry in 1955 by Buonocore when he used 85% phosphoric acid to etch enamel to enhance retention for pit and fissure sealants (Buonocore 1955). He concluded that a significant mechanical advantage of preparing the enamel surface could be obtained when using an acid to demineralize the outermost layer. Early studies have shown that the use of phosphoric acid can dissolve the enamel prisms to a depth of 5-25 μm with a diameter of 5-6 μm allowing for resin penetration with micromechanical bonding (Reynolds 1975). A later study by Ramesh Kumar et al. demonstrated that a resin penetration depth of 53.9 μm was obtained with the use of 37% phosphoric acid with an application time of 30 seconds (Ramesh Kumar et al. 2011). There are three types of patterns formed by acid-etching described by Silverstone et al. (1975) and Summit et.al. (2006). Type 1 is a pattern in which the enamel rods are dissolved,

compared to type 2 is a pattern in which the area around the enamel rods are dissolved. Type 3 is a pattern in which there is no evidence left of any enamel rods. The explanation for the different etching patterns has been attributed to the enamel rod orientation when exposed to acid. Type 1 would be the most ideal etch pattern because it would allow the most penetration of the resin, whereas type 3 would be the least favorable (Summit 2006). With greater resin tag formation or penetration, a resultant increase in bond strength would be expected. Because of the hydrophobic nature of resins, controlling moisture is of utmost importance to allow the resin adhesive to penetrate into the enamel rods securing a strong bond (Xie et al. 1993).

Conventional Bonding

Conventional bonding of orthodontic brackets includes multiple steps. A traditional first step involves cleaning of the tooth with pumice to remove surface debris and expose a clean enamel surface. The second step is the application of 30-50% phosphoric acid for 15-60 seconds followed by rinsing and drying the enamel surface (Barkmeier et al. 1985; Holtan et al. 1995; Osorio et al. 1999). A priming agent is applied to overcome residual moisture followed by the bracket with adhesive resin loaded on the base. Many studies have evaluated this method to test the retention and bond strength of brackets bonded in this conventional manner (Lindauer et al. 1997; Dorminey et al. 2003; Ireland et al. 2003). Reynolds and von Fraunhofer made a suggestion that clinical shear bond strengths of 5.9-7.9 MPa was adequate to withstand the forces of orthodontic treatment (Reynolds and von Fraunhofer 1976). Sargison demonstrated an application of 37% phosphoric acid for 15 seconds produced bond strengths of 64.7N (~7.1MPa) (Sargison et al. 1999). Consistently higher values were obtained by using phosphoric acid with application times of 30 seconds (Dorminey et al.

2003; Scougall Vilchis et al. 2009). Lindauer et al. looked at the effect pumicing had on orthodontic bracket bonding and reported that pumicing did not improve bond strength, etched enamel characteristics, or bracket retention rates (Lindauer et al. 1997). It was concluded that adequate clinical bond strength was achievable implying micromechanical interface of resin and enamel without the first step of pumicing. Others researchers have noted though that care should be taken not to exceed the cohesive shear bond strength within enamel itself, which was reported to be about 14 MPa (Bowen and Rodriguez 1962; Retief 1974). With one goal of orthodontics being to provide treatment in the least invasive manner with regards to enamel loss (Campbell 1995), studies have shown that etching with phosphoric acid produces greater loss of enamel (Hosein et al. 2004). As well, absolute enamel loss is greater with etch times of 60 seconds as opposed to 15 seconds (Osorio et al. 1999). The varied results, as well as the potential for undesirable excessive loss of enamel from etching techniques and a cumbersome multiple step application process, has motivated clinicians and manufacturing companies to pursue alternative materials and methods to prepare teeth for bonding. As a result, numerous studies have been carried out to look at shear bond strengths when using alternative materials and methods such as 10% maleic acid, sandblasting, and self-etch primers as means to prepare the enamel for resin bonding (Bishara et al. 1998; Sargison et al. 1999)

Self-etching Primers

Self-etching primers (SEPs) were developed to minimize the need for a multiple step process of etch, rinse, prime, then bond. The acid component is combined with a hydrophilic primer to be applied in one step. The component makeup of a commonly used SEP includes a methacrylate ester derivative, water, photoinitiator, and dipotassium hexafluorotitanate (3M

Unitek). A study by Banks and Thiruvengkatachari concluded that bonding using SEP was significantly faster than conventional techniques (Banks 2007). One advantage of using a SEP is the elimination of rinsing with water after the acid etching step. Rikuta et al. has shown that moisture contamination from high intraoral humidity and residual water contamination after acid etch rinsing may negatively affect bond strength (Rikuta et al. 2008). SEPs improve the monomer penetration into the enamel surface and wettability of the resin (Attar et al. 2007). Another benefit is that SEPs are more conservative with respect to degree of enamel demineralization (Cal-Neto and Miguel 2006) and produce weaker etch patterns and less dissolution of enamel of both primary and permanent teeth compared to acid etched samples (Cehreli and Eminkahyagil 2006).

Current manufacturer's guidelines for using SEPs include first cleaning the teeth with pumice, followed by a 3-5 second application time making sure to rub the SEP into the enamel surface. This is followed by a 1-2 second burst of air to thin the material and followed by placement of the bracket loaded with resin (Unitek 2008). With the advent of SEPs, specific studies have been involved to assess the application of SEPs with regard to bond strength, pH, preapplication tooth preparation, clinical bond failures and enamel finishing after debonding (Bishara et al. 2001; Torii et al. 2002; Arhun et al. 2006; Cehreli and Eminkahyagil 2006; Attar et al. 2007; Banks 2007; Ostby et al. 2007; Lill et al. 2008; Ostby et al. 2008; Parrish et al. 2011). According to a study by Parrish et al., manufacturers suggest rubbing the SEPs for 3-5 seconds against the enamel surface to produce the highest bond strength when compared to 0 and 10 seconds of application time (Parrish et al. 2011). This has been confirmed by Ostby et al. who reported that increasing the application time from 3 to 15 seconds did not significantly increase the shear bond strength (Ostby et al.

2007). Numerous studies have reported lower, but supposedly clinically acceptable bond strengths when using SEPs. Bishara et al. concluded that using an SEP produced a significantly lower shear bond force ($7.1 \pm 4.4\text{MPa}$) compared to conventional techniques using 37% phosphoric acid ($10.4 \pm 2.8\text{MPa}$) (Bishara et al. 2001). A separate study by Al-Saleh and El-Mowafy supported this conclusion except when using Esthetic Cement™ system with ceramic brackets (Al-Saleh and El-Mowafy 2010). These results may suggest that lower bond strengths will lead to more bond failures. Ireland et al., however, conducted a clinical split mouth study comparing SEPs to conventional etch techniques and concluded that there is only weak evidence to suggest that bond failures will be greater with SEPs (Ireland et al. 2003). This result was supported as well in another study (Dorminey et al. 2003).

Common variations amongst many studies are how the teeth are prepared prior to bonding. Manufacturers' guidelines recommend that prior to application of SEP the tooth should be cleaned with pumice to rid the surface of plaque, calculus, and salivary pellicle (Unitek 2008). However, from past research, the question may arise that if many studies show that SEPs are comparable to conventional etch in bond retention, and pumicing may not be necessary with conventional etch, application of SEPs directly to non-pumiced enamel surfaces may obtain the same results. A few studies have been carried out with split mouth clinical trials of pumicing and not pumicing the teeth prior to SEP application to assess its impact on bond failures. Lill et al. conducted a split mouth study of 30 orthodontic patients looking at pumicing and non-pumicing prior to SEP application and concluded that a significantly lower bond failure rate occurred using Transbond Plus™ SEP after pumice prophylaxis (Lill et al. 2008). This indicates that SEP's may not have adequate strength to

penetrate the enamel pellicle and etch the enamel surface adequately. The enamel pellicle may need to be removed prior to SEP application.

Pellicle

The acquired enamel pellicle was described by Dawes et al. as a structureless and essentially bacteria free film that is deposited on the tooth surface post-eruption (Dawes 1963). Meckel concluded that the pellicle consists of modified salivary mucoproteins that form a layer of 1 to 10 μm thickness above the enamel surface (Meckel 1965). Through compositional analysis, Armstrong supported this conclusion that the acquired pellicle was formed from a mixture of salivary mucoproteins, with the addition of bacterial cell wall materials (Armstrong 1968). He explained that the pellicle differed from plaque in its composition and ability to be removed from the enamel surface. Plaque could be removed by brushing with non-abrasive material, whereas the pellicle could only be removed with dental prophylaxis using abrasives to expose the underlying enamel prisms and crystallites (Armstrong 1968). However, Main et al., when studying ways to improve procedures for application of pit and fissure sealants, found that acid etching alone was sufficient to remove the acquired pellicle and that pumicing was not an essential step. This was confirmed when they compared the scanning electron microscopy and shear bond strength tests and found no differences of teeth with and without pellicle prior to being etched.

According to current manufacturer guidelines when using either conventional or SEP techniques, a clean and exposed enamel surface is ideal to obtain maximum bonding efficacy. This would support the use of some means of removing the pellicle prior to enamel etching. The issue arises when during prophylaxis, gingival stimulation occurs and the resultant gingival crevicular exudate contaminates the enamel surface. After etching, if the

enamel is contaminated with saliva, the organic pellicle begins to reform and will not rinse away with water (Silverstone et al. 1985). The organic pellicle covers the enamel pores, decreasing resin penetration and reducing the micromechanical bond (Silverstone et al. 1985). Contamination by fluids when bonding negatively impacts the bond strength and allows for microleakage at the resin tooth interface (Xie et al. 1993). In general, dental adhesives and resins are hydrophobic in nature. In the presence of moisture there is less resin penetration and increased likelihood of bond failure. However it is suggested that some moisture is tolerable with the Transbond Plus SEP (Unitek 2008). As an alternative, some recommendations have been made to bring the patient in for dental prophylaxis one week prior to bonding to rid the enamel of most of its plaque and calculus to improve gingival health on the day of bonding. However, this introduces another step and cost of inconvenience and chair time which many orthodontists are not willing to accept. Subsequently, other methods of removing the pellicle have been proposed. This includes using conventional etch first for 15 seconds as a pellicle removing and initial etching agent. After phosphoric acid etch application followed by rinsing and drying, a self-etching primer is rubbed on the surface for 5 seconds to further etch and prime the tooth for bracket bonding. This method has been coined the term “double-etch” technique.

Preparing enamel for bonding orthodontic brackets with light-curable resins is currently being carried out using conventional techniques of etch, prime, and adhesive, or the use of self-etch primers. A point of variation among many clinicians is what they do to prepare the tooth prior to application of self-etching primers. Manufacturers’ guidelines recommend pumicing prior to either technique; however, many clinicians and even the representatives of those companies suggest avoiding pumicing so as to prevent unwanted

fluid contamination from gingival crevicular fluids or blood. However there is currently lack of evidence to support this claim. As an alternative they recommend using phosphoric acid first, followed by a SEP to avoid unwanted fluid contamination yet penetrate the pellicle layer. This technique is an attempt to ensure adequate bond strength based on the assumption that the phosphoric acid will remove the pellicle and the SEP will etch the enamel. A study by Torii et al. determined that the use of phosphoric acid before application of SEP on bovine enamel resulted in an increased tensile bond strength (Torii et al. 2002). Although no current evidence supports it, the combination of acid etch, followed by an SEP, may create severe permanent effects to the enamel surfaces that otherwise may be avoided. This is of importance because the remaining surface is potentially more susceptible to environmental insult and developing carious lesions.

Problem Statement

The purpose of this study was to examine the methods of preparing the enamel surface for orthodontic bracket adhesion in overcoming pellicle and possible fluid contamination from prebonding cleaning techniques and the effect of these treatments on the enamel surface and resultant bond strength.

Hypotheses

1. There will be a significant difference in the self-etching primer/adhesive bracket bond strength as a function of the enamel pretreatment protocol using phosphoric acid, pumice prophylaxis, or a tap water rinse.
2. If there is a significant difference in shear bond strength, there will also be a significant difference in the depth of resin penetration as a function of the enamel pretreatment protocol.
3. There will be a relationship between the self-etching primer/adhesive bracket bond strength and the depth of enamel resin penetration.
4. There will be a qualitative difference in etched enamel surface pattern related to the enamel pretreatment protocol.

CHAPTER 2

MATERIALS AND METHODS

Specimen Preparation

Information and study details were submitted to the University of Missouri-Kansas City (UMKC) Adult Health Science Institutional Review Board (AHSIRB). It was determined that the study did not qualify as human subject research as per 12-07-NHSR.

A collection of 30 previously extracted, intact, non-identifiable, human maxillary 3rd molars were obtained from the UMKC Oral Surgery Clinic and randomly divided into three treatment groups of 10 specimens in each group. No patient identifiers were associated with the teeth and all patients signed waivers permitting use of their teeth for research at UMKC School of Dentistry. The teeth were collected in individual plastic vial containers filled with phosphate buffered solution without sodium azide. Once collected and debrided of soft tissue remnants with a dental scaler¹, they were placed in a new, clean plastic vial containing PBS solution with 0.002% sodium azide and placed in the 4°C refrigerator for storage until testing. The teeth to be included in the study were inspected for a sound and intact buccal and lingual surface with no evidence of carious lesions, demineralization, abfraction, restorations, fluorosis, anomalous morphology, or any defective surface created on the areas of interest when the tooth was extracted. The storage medium of PBS was made by mixing 9.6 grams of powder² with 1 L of sterile, deionized (DI) water. The powder was composed of 83% sodium chloride, 12% anhydrous sodium phosphate, 2% potassium phosphate and 2% potassium chloride.

¹ Dental Scaler, BPS2, Hu-Friedy, 3232 N. Rockwell, Chicago, IL 60618-5982

² Dulbecco's Phosphate Buffered Saline, Sigma-Aldrich, 3050 Spruce St., St. Louis, MO 63103

PBS solution without azide

1 Liter Autoclaved Nanopure H₂O

1 Container Dulbecco's Phosphate Buffered Saline²

PBS solution with 0.002% azide

1 Liter Autoclaved Nanopure H₂O

1 Container Dulbecco's Phosphate Buffered Saline²

0.02 grams sodium azide³ (NaN₃)

There was two testing sessions with fifteen specimens tested during each testing session. The fifteen specimen were randomly assigned to one of the three testing groups per testing session totally five specimen per treatment group per testing session.

Specimen Mounting

Fifteen specimens were individually mounted with acrylic resin⁴ embedding the roots up to, but not covering the cemento-enamel junction with the crown exposed using a mounting ring⁵ and jig (Figure 1). The teeth were oriented in the ring and jig so that the surface receiving the bracket was centered in the middle of the ring and the labial surface of tooth crown being perpendicular to the surface of acrylic resin. The acrylic resin was mixed and the tooth seated into position and allowed to cure until cooled (~30 min.), after which the acrylic was separated from the mounting ring and available for testing. For test locating purposes when examining under scanning electron microscope, each mounted tooth had the

³ Sodium Azide Sigma-Aldrich, 3050 Spruce St., St. Louis, MO 63103

⁴ Biocryl #040-016, Great Lakes, 200 Cooper Ave., Tonawanda, NY 14150

⁵ Item #20-8180, Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044

lingual testing surface designated by creating two surface score marks with a fine tipped dental carbide bur at a distance of ~4mm apart. Once prepared, a plastic open-ended cylinder was then secured in place over the top of the embedded tooth with the use of intraoral clear orthodontic rope wax⁶ to allow the tooth to be bathed in saliva for 14 hours. Fresh human saliva was collected from one experimenter, over an interval of 30 minutes prior to specimen mounting, and stored in a closed container and refrigerated at 4°C until poured into open-ended cylinder to begin 14-hour bath of specimen surfaces to form acquired pellicle and minimize any microbial growth. After the 14-hour saliva bath, the enamel surfaces had an acquired pellicle on the surface to simulate the intraoral environment.

⁶ Orthodontic wax, DynaFlex P.O. Box 99 St Ann, MO 63074-0099



Figure 1. Mounting jig centered on mounting ring

Immediately following removal from the 14-hour saliva bath, tooth specimen in the 3 experimental groups were prepared for and bonded under controlled oral environmental simulated lab settings of 75% (+/- 2%) humidity and 33°C (+/- 2°) temperature (Figure 2). Both the mesiobuccal and mesiolingual enamel surfaces were prepared in the same manner prior to bracket bonding on the buccal surface only. Specimen were assigned to and prepared according to one of the three treatment protocols described below.

1. 3 second tap H₂O rinse, 5 seconds oil-free air drying, 5 second scrub of Transbond Plus Self Etching Primer⁷.
2. Pumice⁸ prophylaxis, 3 second tap H₂O rinse, 5 seconds oil-free air drying, 5 second scrub of Transbond Plus Self Etching Primer⁸.
3. 15 second 34% phosphoric acid etch⁹, 3 second tap H₂O rinse, 5 seconds oil-free air drying, 5 second scrub of Transbond Plus Self Etching Primer⁸.

⁷ Transbond Plus Self Etching Primer™, 3M Unitek Orthodontic Products, 2724 South Peck Road, Monrovia, CA 91016

⁸ Whip Mix Preppies™, Pearson Dental Supply Company, 13161 Telfair Ave., Sylmar, CA 91342

⁹ 34% Tooth Conditioner™, 3M Unitek Orthodontic Products, 2724 South Peck Road, Monrovia, CA 91016

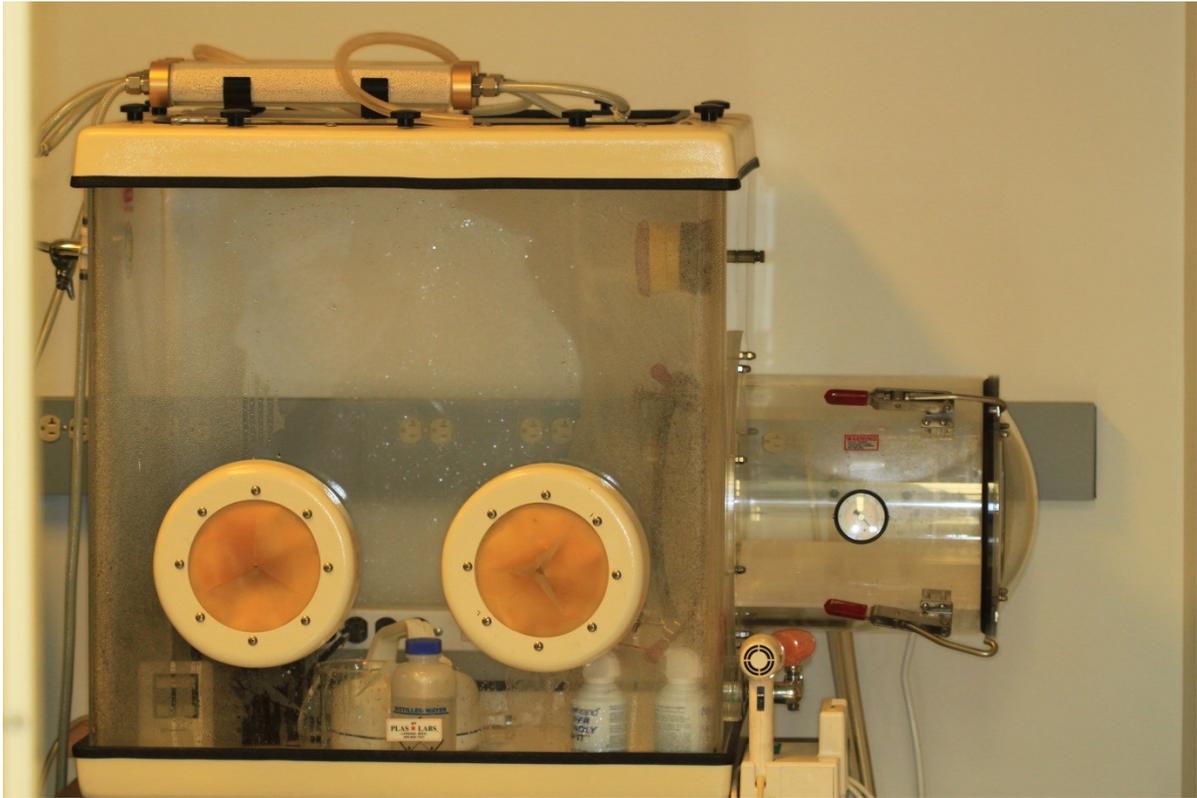


Figure 2. Environmental Chamber

Materials

The orthodontic brackets¹⁰ used were standard edgewise maxillary first premolar stainless steel brackets with 0.022-inch slot and concave bracket base. Maxillary 3rd molar specimen were initially obtained and various brackets were adapted to the mesiobuccal surfaces to determine the best fit. The maxillary first premolar bracket was selected because it adapted well and it is universal for both right and left sides.

The self-etching primer⁸ used in all treatment groups was comprised mainly of a mixture of methacrylate ester derivative making up 75-85% by weight. The remaining contents included water (15-25%), DL-camphorquinone (<1.0%), and dipotassium hexafluorotitanate (<0.2%).

The orthodontic resin adhesive¹¹ used in this study was composed of 70-80% silane-treated quartz, 10-20% bis-GMA, 5-10% Bisphenol A Bis (2-hydroxyethyl ether) dimethacrylate, less than 2% silane-treated silica, and less than 0.2 % diphenyliodonium hexafluorophosphate.

¹⁰ Victory Series/MBT, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

¹¹ Transbond XT™, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

Experimental Design

The effects of one independent variable with three levels of treatment (enamel pretreatment) on the resultant orthodontic bracket shear bond strength, resin depth penetration, and enamel etch patterns of was examined in this study. The enamel pretreatment protocols entailed removing the pellicle from the enamel surface with three different methods prior to self-etching primer application and subsequent orthodontic bracket bonding. The three methods tested of pellicle removal prior to self-etching primer application were: 1) tap water rinse; 2) pumice prophylaxis; 3) 34% phosphoric acid etch. All treated specimens then had a self-etching primer/adhesive applied and subsequent orthodontic bracket bonded. This design potentially produced three different prepared enamel surfaces for the bonding to be carried out on and subsequently tested and analyzed (Table 1).

TABLE 1
RESEARCH DESIGN

Enamel Pretreatment Protocol (N= 10 teeth/protocol)	Shear Bracket Bond Strength (MPa)	Depth of Resin Penetration (Raman) (μm)	Surface Etch Pattern (Type 1,2,3)
Distilled H ₂ O Rinse			
Pumice Prophylaxis			
Phosphoric Acid Etch			

The first group of specimen were treated with a tap water rinse and self-etching primer. Upon removal from the saliva bath, the teeth were rinsed with tap H₂O, dried gently with a stream of oil free air for 5 seconds followed by an immediate 5 second scrub application of Transbond Plus self-etching primer (SEP), according to manufacturer guidelines (Unitek 2008). The SEP was thinned lightly with air following which an orthodontic bracket (Unitek 0.022 Victory Series premolar bracket¹²) was loaded with Transbond XTTM¹³ and applied to the mesiobuccal enamel surface orienting the bracket base perpendicular to the base of the resin block and was fully depressed with a Hollenbeck¹⁴ carver with the excess resin cleaned from around the bracket (Figure 5). The resin was light polymerized for a total of 20 seconds, 10 seconds from the gingival and 10 seconds from the occlusal with a LED resin curing light¹⁵ of recorded wavelength using a radiometer¹⁶. The same procedure was administered to the mesiolingual surface of the same specimen less the bonding of the bracket.

The second group of specimen were subjected to prophylaxis with non-fluoride containing pumice for 5 seconds, followed by rinsing with tap water for 5 seconds, air drying lightly for 5 seconds, and then application of Transbond Plus self-etching primer (SEP) scrubbing the enamel surface for 5 seconds (Unitek 2008). Following SEP application a light

¹² Victory Series/MBT, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

¹³ Transbond XTTM, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

¹⁴ Hollenbeck Carver, CVHL 1/2, Hu-Friedy, 3232 N. Rockwell, Chicago, IL 60618-5982

¹⁵ Ortholux™ LED Curing Light, 3M Unitek Orthodontic Products, 2724 South Peck Road, Monrovia, CA 91016

¹⁶ OrthoLux LED radiometer, 3M Unitek, 2724 South Peck Rd., Monrovia, CA 91016

stream of air was blown over the enamel surface to thin the SEP and then one orthodontic bracket (Unitek 0.022 Victory Series premolar bracket) was loaded with Transbond XTTM and applied to the mesiobuccal enamel surface orienting the bracket base perpendicular to the base of the resin block and fully depressed with the Hollenbeck carver. The excess resin was then cleaned off around the bracket with the same carver and the resin was polymerized by exposure to LED curing light of recorded specified wavelength for a total of 20 seconds, 10 seconds from the gingival and 10 seconds from the occlusal aspect of the bracket. The same procedure was administered to the mesiolingual surface of the same specimen less the bonding of the bracket.

The third test group was subjected to phosphoric acid etch followed by SEP application. The specimen were removed from the saliva storage and immediately exposed to 34% phosphoric acid application to the bonding area for 15 seconds. They were then rinsed for 5 seconds with tap H₂O and dried for another 5 seconds with oil-free air. Following this sequence they were immediately subjected to a 5 second scrub of the SEP (Transbond Plus, 3M Unitek). Once again a light stream of air was blown over the enamel surface to thin the SEP and a bracket (Unitek 0.022 Victory Series premolar bracket) loaded with Transbond XTTM was applied to the mesiobuccal enamel surface orienting the bracket base perpendicular to the base of the resin block and fully depressed with a Hollenbeck carver. The excess resin was cleaned off around the bracket and the resin was cured for a total of 20 seconds, 10 seconds from the occlusal and gingival aspects of the bracket, with the LED light of recorded specified wavelength. The same procedure was administered to the mesiolingual surface of the same specimen less the bonding of the bracket.

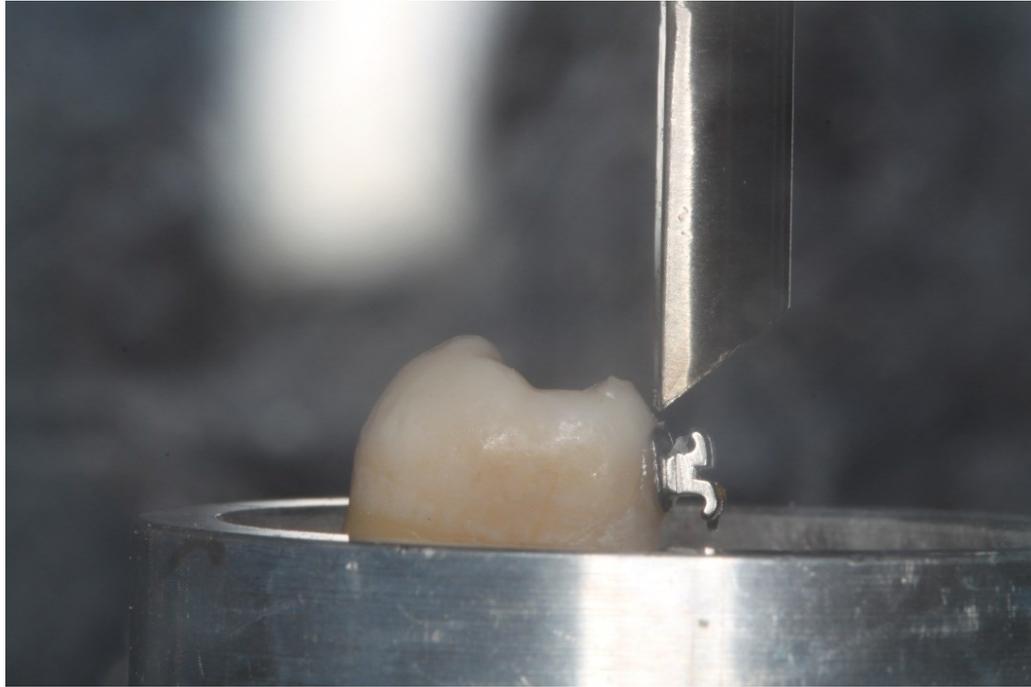


Figure 3. Specimen mounted for shear bond testing. Bonded bracket to the MB cusp of maxillary 3rd molar perpendicular to the resin base.

Instrumentation and Data Collection

Once all of the specimen were bonded during a testing session they were loaded for shear bond strength, following which they were stored in dry containers in the refrigerator until further testing via scanning electron microscopy and Raman mapping. The time between bond and debond was recorded for to ensure consistency.

Shear Bond Strength

Shear bond strength was tested using a universal testing machine¹⁷ (Figure 6) with the brackets being subjected to a crosshead speed of 1mm/min with an occlusogingival load direction along the bracket tooth interface. The stainless steel blade was manually brought to within 1mm of the occlusal edge of the bracket base and then the test initiated (Figure 5). The maximum load to debond the bracket in Newtons (N) was recorded using commercial digital software¹⁸. The shear bond strength was then calculated as following using the bracket base surface area of 10.83mm² as per caliper measurement of the brackets:

$$\text{Shear Bond Strength (MPa)} = \frac{\text{Debond Force (N)}}{\text{Bracket Base Area (mm}^2\text{)}}$$

Statistical analysis to determine if a significant difference exists among debond strengths between groups was carried out. If no significant difference existed, representative specimen from each of the three test groups would have been randomly selected to undergo Raman mapping and SEM to look at resin depth penetration and surface etch pattern characteristics. Specimen from each test group were sectioned with a slow speed diamond

¹⁷ Model 5967, Instron Corporation, 825 University Ave., Norwood MA 02062-2643

¹⁸ Bluehill software, Instron Corporation, 825 University Ave., Norwood MA 02062-2643

saw¹⁹ first in a mesiodistal direction to obtain the bonded (buccal) and unbonded (lingual) surfaces. Next the saw was used to make three cuts in a buccolingual direction across the debonded buccal site to produce two specimens in cross-section. One specimen produced three areas for analysis; one for SEM of the enamel surface (mesiolingual site), one for SEM in cross-section after debonding (mesiobuccal site), and one for Raman mapping in cross-section after debonding (mesiobuccal site).

¹⁹ 41 Buehler Isomet Slow Speed Diamond Saw, Waukegan Road Lake Bluff, Illinois 60044, USA

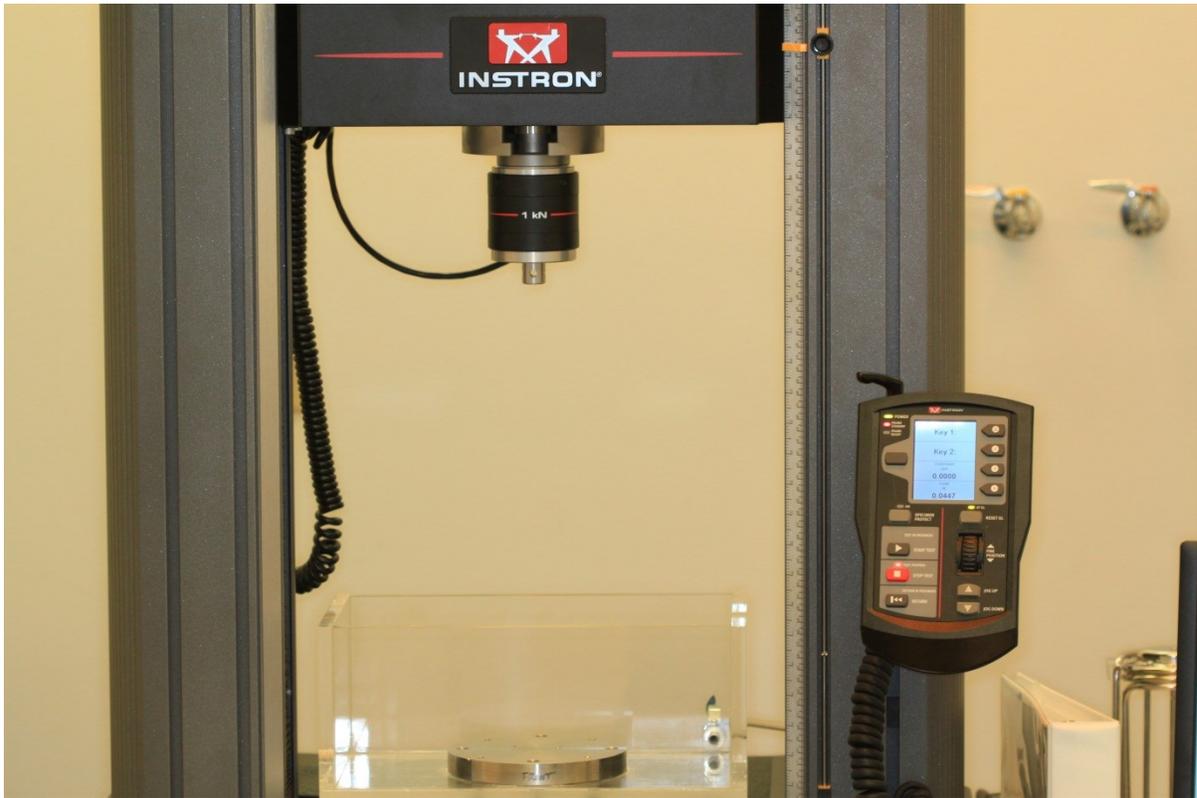


Figure 4. Universal tester used for shear bond strength testing.

Raman Mapping

Raman mapping was used to evaluate resin penetration into enamel. Since a significant difference in bond strength as a function of enamel pretreatment protocol was noted, selected specimen had the interface of the enamel and resin mapped using Raman mapping. Tooth specimens were sectioned with a slow speed diamond saw²⁰ first in a mesiodistal direction to obtain the bonded (buccal) and unbonded (lingual) surfaces. Next the saw was used to make three cuts in a buccolingual direction across the debonded buccal site to produce two specimens in cross-section. One specimen produced three areas for analysis; one for SEM of the enamel surface (mesiolingual site), one for SEM in cross-section after debonding (mesiobuccal site), and one for Raman mapping in cross-section after debonding (mesiobuccal site).

One half of the buccal sectioned specimen was subjected to Raman mapping to generate Raman spectrum for the bonding site in cross-section. Raman mapping in cross-section allowed quantification of the depth of penetration of the adhesive resin. The depth of resin penetration may subsequently coincide with greater micromechanical retention and increased shear bond strength of the orthodontic bracket.

Prior to each selected specimen being subjected to Raman mapping, the specimen was mounted on a glass slide and resin located using 50x lens on a light microscope. The spectrometer was calibrated by using a silicon sample at 520nm to control for daily humidity variations and to allow comparisons across samples. The red ion laser of 632.8nm wavelength focused through 100x lens was used with the range of spectra collection set at

²⁰ 41 Buehler Isomet Slow Speed Diamond Saw, Waukegan Road Lake Bluff, Illinois 60044, USA

400-2000 cm^{-1} . The spatial resolution and spectral resolution was set at 1.0 μm and 2.5 cm^{-1} respectively. Once the specimen was loaded and oriented with the resin/enamel interface in a vertical orientation on the monitor, the site to be analyzed was determined and confirmed by Raman mapping at 2 sites, one in the resin and one in the enamel, to confirm correct specimen orientation and location of the interface. Once the interface had been accurately located, two additional sites were tested by moving the mapping acquisition a linear measurement of 10 μm both above and below the initial test site. This gave three mapping sequences per specimen to give a mean depth of resin penetration per specimen. Each linear mapping across the resin/enamel interface was carried out to scan at 1 μm intervals across a 25 μm range. The resultant mapped peaks were formatted and refined by NGSLabSpec® digital software²¹ to eliminate extraneous peaks and noise using baseline correction and smoothing. The resultant compiled mapping spectra were copied to PowerPoint²² for viewing and depth of resin penetration scoring.

Scanning Electron Microscopy

Three representative tooth specimen sections from each group were imaged with a scanning electron microscope²³ (SEM) at various magnifications to qualitatively assess the penetration of the resin to the underlying enamel surface from the different preparation techniques. To prepare the cross-section specimens for imaging, the surface was subjected to a 30 second acid etch with 5M HCl to demineralize the enamel leaving the resin tags exposed. The specimen were mounted and coated with gold-palladium. Images of the

²¹ GRAMS/AI v7.02, Galactic Industries Corp., 395 Main St., Salam, NH, 03079

²² PowerPoint 2010, One Microsoft Way, Redmond, WA 98052-6399

²³ Philips XL30, Philips Electron Optics, 5350 NE Dawson Creek Drive, Hillsboro, OR 97124

remaining resin tags were then qualitatively evaluated in terms of structure and appearance. Similarly, the same representative tooth specimen lingual halves were used to qualitatively evaluate the surface enamel characteristics of the etch pattern on the mesiolingual surface. No prior processing was required for these specimens.

Resultant qualitative differences in depth of resin penetration and etch pattern between the enamel pretreatment protocols was noted.

Data and Statistical Analysis

All quantitative values from shear bond strength testing was analyzed with a statistical software program to determine significance²⁴. A one-factor ANOVA ($\alpha = 0.05$) was used to determine if there was a significant effect of enamel pretreatment on bracket bond strength. A Tukey Kramer post hoc test was utilized to determine where the difference existed.

Since there was a significant difference in shear bond strength, Raman mapping data was also analyzed with a 1-factor ANOVA and a Tukey's post hoc. The SEM qualitative evaluation of enamel resin penetration was used as a complementary confirmation to the Raman mapping. There was also a qualitative SEM evaluation of the etch pattern on the lingual surface.

²⁴ SPSS Inc., 233 S. Wacker Drive, Chicago, IL 60606-6307

CHAPTER 3

RESULTS

Shear Bond Strength

Extracted non-identifiable human maxillary third molars were collected and assigned to one of the three treatment groups according to the inclusion protocols established in Chapter 2. Ten molar specimens were randomly assigned to each of the three test groups. Specimens were mounted in acrylic and bathed in collected human saliva for 14 hours in refrigerated conditions 4°C. The specimens were then removed from the saliva bath for immediate bracket bonding as per bonding protocols in Chapter 2. The resultant shear bond force after bonding according to one of the three bonding protocols of H₂O rinse, pumice prophylaxis, and 34% phosphoric acid etch prior to self-etching primer were recorded (Table 2). Debond forces were recorded in Excel® software with the corresponding shear bond strength calculated according to the equation: Shear Bond Strength (MPa) = Debond Force (N)/Bracket Base Area (mm²) (Table 2). The average time between bonding and debonding was 42.73 ± 3.30 minutes. Resin/tooth interface was characterized using 2.5x dental loupes to note where the bond failure occurred and categorize the fracture as a type 1,2,3,4, or 5 based on where the separation occurred (Table 2). If the failure was a cohesive failure within the resin it was denoted a type 1 fracture. An adhesive failure between the resin and the enamel surface it was assigned a type 2 failure. If the failure was between the resin and the bracket it was a type 3 failure, and if it was a mixed adhesive and cohesive failure it was assigned a type 4. Two specimens had an intraenamel failure where enamel was removed with the bracket and this was labeled a type 5 enamel cohesive failure. The H₂O rinse group showed a wide variance in type of fracture with specimens showing type 2, 3, 4, and 5

fractures. The pumice prophylaxis group showed mostly adhesive fractures, a type 2, between the enamel and resin interface, with two specimens having a type 4, mixed fracture. The 34% phosphoric acid etch group showed mostly a type 3 adhesive fracture, with one specimen undergoing a type 4 fracture and one a type 5 fracture.

Utilizing SPSS® software, the mean shear bond strength and standard deviation was calculated and recorded in Table 2 and shown in Figure 5. The mean shear bond strength of the H₂O rinse group was 13.836 ± 5.899 MPa. The mean shear bond strength of the pumice prophylaxis group was 11.138 ± 4.863 MPa. The mean shear bond strength of the 34% phosphoric acid etch group was 17.828 ± 3.428 MPa.

Utilizing a one-way analysis of variance (1-way ANOVA) with SPSS® software, a significant difference in shear bond strength with a value 0.016 was determined to exist among the treatment groups ($p < 0.05$). Tukey Kramer post hoc testing located the difference in pre-treatment protocols to exist between the pumice prophylaxis and the 34% phosphoric acid etch groups. No significant difference was found to exist between the H₂O rinse and the pumice prophylaxis group, or between the H₂O rinse and the 34% phosphoric acid group.

Raman Mapping

Three selected specimens from each test group were prepared for Raman mapping according to protocols outlines in Chapter 2 and the resin/enamel interface was mapped at 1 micron increments across a 25 μ m interface. Verification of enamel and resin on either end of the mapping site was first determined with an initial spectrum being collected to verify enamel and resin. The mapping was acquired perpendicular to the interface at three sites and the resultant peaks were formatted and stacked using NGS LabSpec® software. Mean depth

of resin penetration with standard deviation of the interface for each test group was recorded (Table 3).

Descriptive statistics calculated both the mean shear bond strength and the mean depth of resin penetration for the selected specimens (Table 3 and Figure 6). The mean shear bond strength of the H₂O rinse group was 15.629 ± 6.126 MPa. The mean shear bond strength of the pumice prophylaxis group was 12.715 ± 6.613 MPa. The mean shear bond strength of the 34% phosphoric acid etch group was 19.761 ± 0.283 MPa. The mean depth of resin penetration of the H₂O rinse group was 8.777 ± 2.870 μm . The mean depth of resin penetration of the pumice prophylaxis group was 3.890 ± 1.837 μm . The mean depth of resin penetration of the 34% phosphoric acid etch group was 7.667 ± 2.400 μm .

One-way ANOVA showed no significant difference to exist between the subset data mean scores of all treatment groups in regards to both shear bond strength (0.320) and depth of resin penetration (0.103) with $p < 0.05$. However, a large percentage of the effect of shear bond strength and depth of resin penetration on bond strength was attributable to enamel surface preparation method as indicated from the partial eta squared values of 0.316 and 0.531 respectively.

SEM Analysis

Surface topography of selected specimen from each of the test groups were evaluated from the lingual surfaces that were prepared with the three protocols of etching. Specimens were coated with a gold/palladium alloy and images taken at 30X, 100X, 1500X, 3000X and 6000X magnification. The surface characteristics were noted and etch pattern was compared to etch patterns described as being Type 1, 2 or 3 (Silverstone, 1975). All specimen showed non-distinct etch patterns most closely resembling a Type 3 etch pattern. The enamel surface

from the H₂O group showed depressions that were somewhat uniform, the pumice group showed distinct scratch lines possibly from the abrasiveness of the pumice, and the 34% etch group showed similar appearance to the H₂O group with possibly more pitted morphology (Figure 7).

The cross-sectioned specimens were also imaged at the same magnifications and the underside of the resin was characterized. The SEM imaging table was tilted 30-75° to view the underside of the resin. The H₂O test group showed a consistent network of resin tags of apparent uniform size and structure (Figure 8a,b). The pumice group showed less defined resin tags as well as a replication of the surface scratches noted from the surface SEM analysis (Figure 8c,d). The 34% phosphoric acid etch group resin undersurface had less uniform display of resin tags compared to the H₂O group; however, it appeared to have some larger protrusions of resin mixed with the smaller tags (Figure 8e,f). Overall the resin undersurface tags in each groups seem to replicate the surface etch characteristics with reasonable consistency.

TABLE 2. Debond Force, Shear Bond Strength, Time and Fracture Interface Type for Tested Specimen

Enamel Pretreatment Protocol (N= 10 teeth/protocol)	Specimen	Debond Force (N)	Shear Bracket Bond Strength (MPa)	Bond to Debond Time (min.)	Interface Fracture Site Type*	Mean Shear Bracket Bond Strength (MPa)
Distilled H ₂ O Rinse	1-1	135.35	12.501	36.00	2	13.84 ± 5.90
	1-2	165.05	15.244	37.00	4	
	1-3	78.45	7.245	39.00	2	
	1-4	253.92	23.451	47.00	5	
	1-5	115.93	10.707	44.00	2	
	1-6	111.97	10.341	43.00	2	
	1-7	126.66	11.698	42.00	4	
	1-8	245.64	22.687	40.00	4	
	1-9	193.58	17.879	41.00	2	
	1-10	71.54	6.607	35.00	3	
Pumice Prophylaxis**	2-1	121.13	11.187	45.00	2	11.14 ± 4.86
	2-2	204.33	18.871	44.00	2	
	2-3	65.14	6.016	46.00	2	
	2-4	61.76	5.704	42.00	2	
	2-5	61.99	5.725	42.00	2	
	2-6	95.69	8.838	39.00	2	
	2-7	171.76	15.863	41.00	4	
	2-8	179.89	16.614	40.00	4	
	2-9	97.54	9.009	41.00	2	
	2-10	146.7	13.549	45.00	2	
Phosphoric Acid Etch	3-1	164.41	15.184	44.00	3	17.83 ± 3.43
	3-2	196.88	18.183	43.00	5	
	3-3	212	19.580	44.00	3	
	3-4	192.07	17.739	45.00	3	
	3-5	142.55	13.166	46.00	3	
	3-6	217.5	20.088	47.00	3	
	3-7	268.15	24.766	46.00	3	
	3-8	170.44	15.741	47.00	3	
	3-9	212.39	19.616	46.00	3	
	3-10	153.91	14.215	45.00	4	

* Interface Fracture Type: 1= cohesive (intraresin), 2= adhesive (resin/tooth), 3= adhesive (resin/bracket), 4= mixed (2&3), 5= cohesive (intraenamel)

** There was a significant decrease in shear bond strength when pumice was utilized prior to SEP application as compared to the use of 34% phosphoric acid etch.

TABLE 3. Mean Values of Selected Specimens from Pre-treatment Groups

Enamel Pretreatment Protocol	Mean Shear Bond Strength (MPa)	Mean Depth of Resin Penetration (Raman) (μm)	Surface Etch Pattern (Type 1,2,3)
Distilled H ₂ O Rinse	15.629 \pm 6.126	8.777 \pm 2.870	3
Pumice Prophylaxis	12.715 \pm 6.613	3.890 \pm 1.837	3
Phosphoric Acid Etch	19.761 \pm 0.283	7.667 \pm 2.400	3

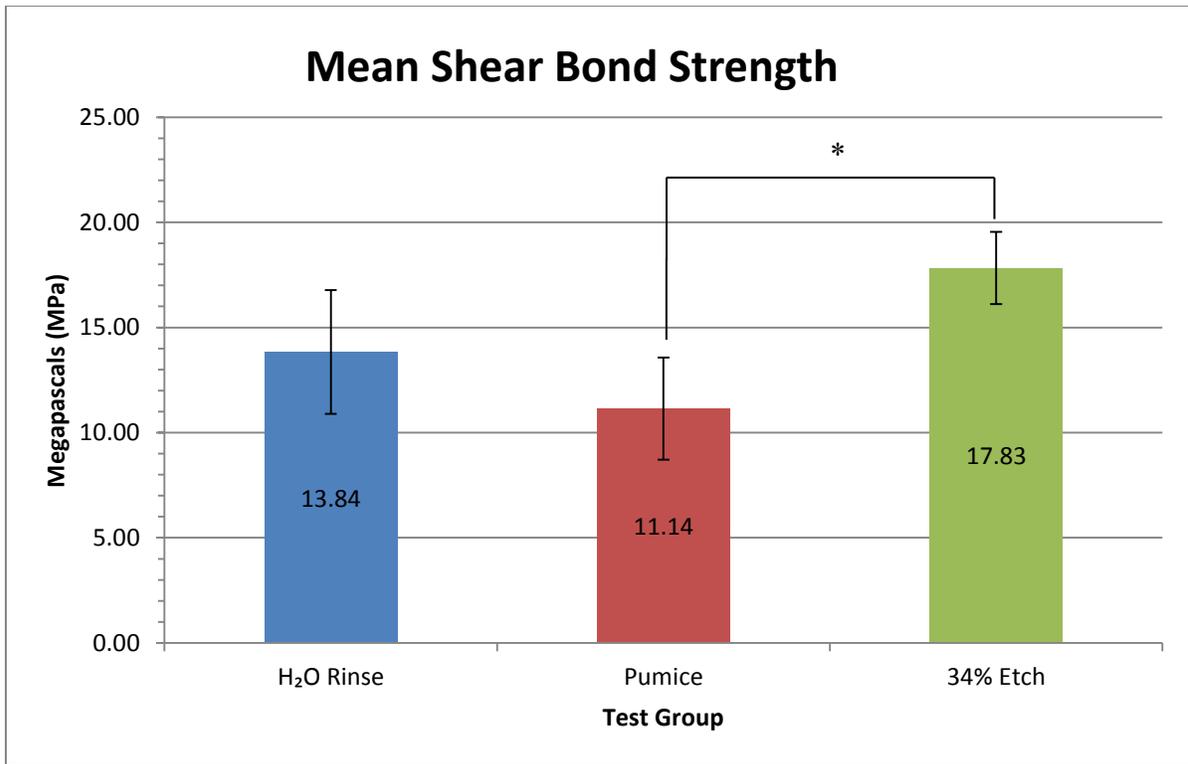


Figure 5. Mean shear bond strength. Shear bond strength values for the three experimental groups with standard deviation. * Tukey Kramer post-hoc ($p < 0.05$).

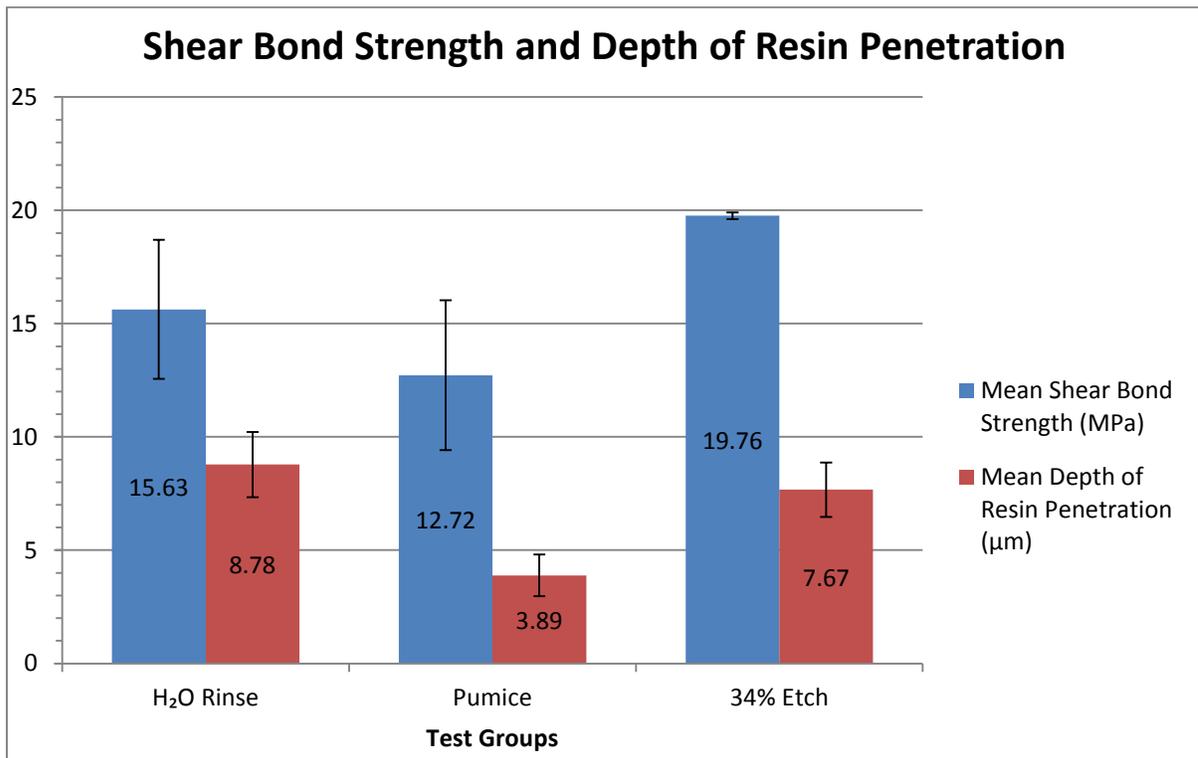


Figure 6. Mean shear bond strength and depth of resin penetration. Mean values of selected specimen from each of the three experimental groups with standard deviation. No significant difference exists across all treatment groups ($p < 0.05$).

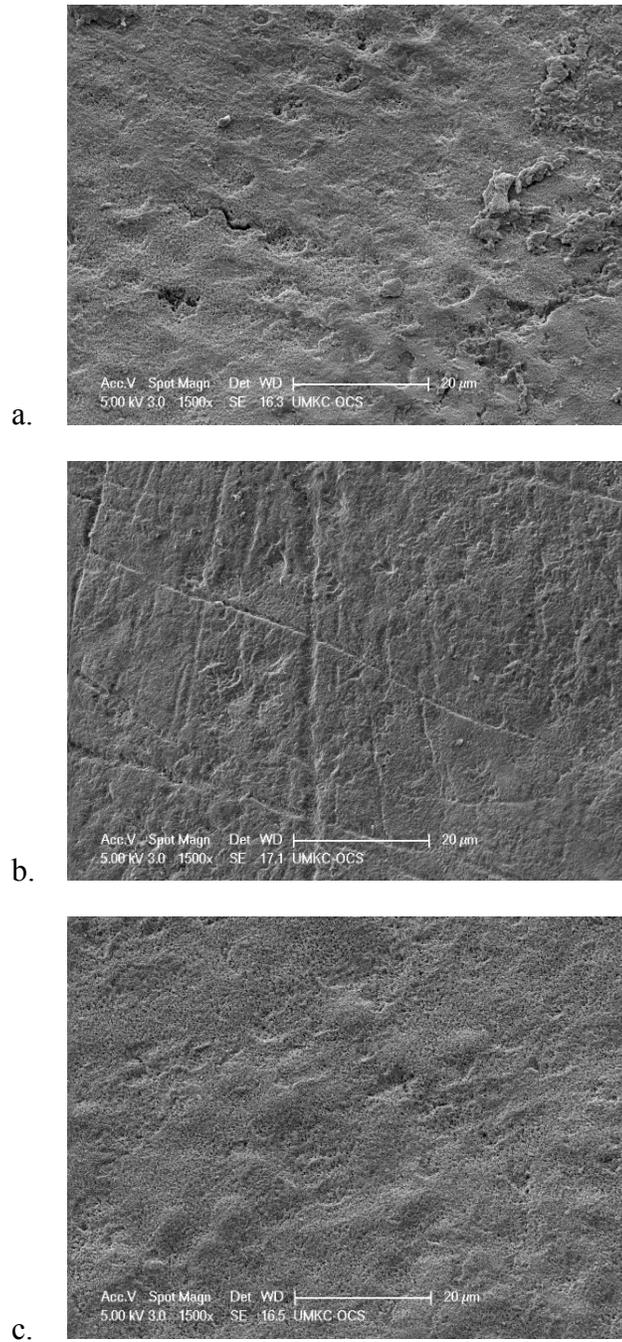


Figure 7. Enamel surface scanning electron microscopy. *a.* H₂O rinse group (1500X magnification); *b.* Pumice group (1500X magnification); *c.* 34% phosphoric acid etch group (1500X magnification).

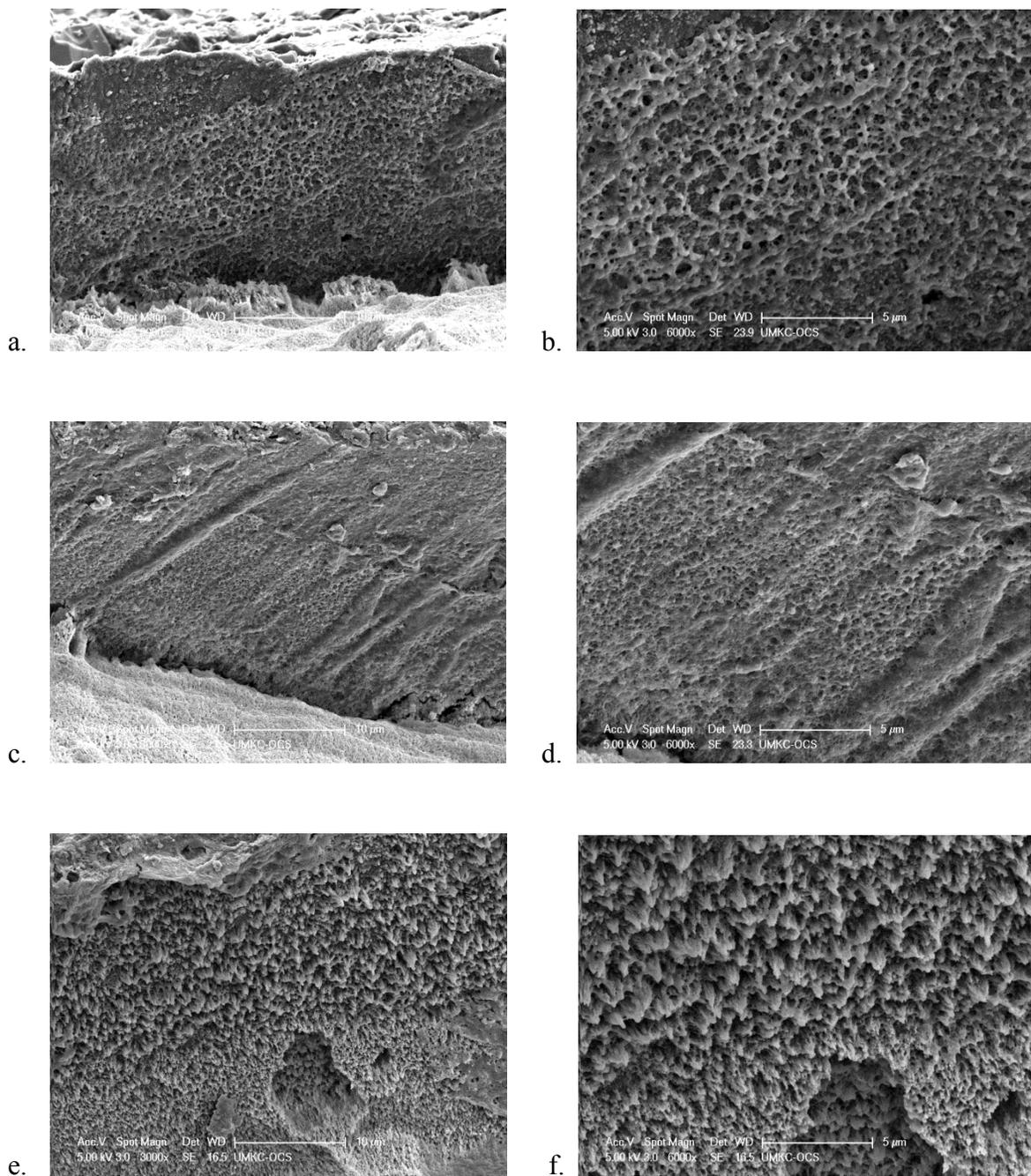


Figure 8. Cross-sectional scanning electron microscopy. *a,b*, SEM of resin tags from resin/enamel interface of H₂O specimen after dissolution of enamel (3000X and 6000X magnification); *c,d*, SEM of resin tags from resin/enamel interface of pumice specimen after dissolution of enamel (3000X and 6000X magnification); *e,f*, SEM of resin tags from resin/enamel interface of 34% etch specimen after dissolution of enamel (3000X and 6000X magnification).

CHAPTER 4

DISCUSSION

The use of self-etching primers when bonding orthodontic brackets is widely used among practicing orthodontist with varying preparation of enamel surfaces prior to its application. These differences in application protocol, or treatment of the enamel surface prior to SEP application, was the motivating factor behind this study being undertaken. According to manufacturer guidelines on their website, it is recommended that the tooth be cleaned prior to SEP application through the act of pumice prophylaxis. This is in contrast though to what the representatives from those companies direct orthodontists to do in practice. They recommend not pumicing the day of bonding but to apply 34% phosphoric acid etch as the tooth cleaning agent prior to SEP application. To date many studies have evaluated the effect of 34% phosphoric acid etch and SEP independently, however there are no studies evaluating the combined effects of these etchants on human teeth in simulated oral environment. The purpose of this study was to examine the methods of preparing the enamel surface for orthodontic bracket adhesion in overcoming pellicle and possible fluid contamination from pre-bonding cleaning techniques and the effect of these treatments on the enamel surface and resultant bond strength. The combinatory effects of phosphoric acid etch and self-etching primer application on shear bond strength and depth of resin penetration as compared to pumicing or rinsing the enamel surface with H₂O prior to SEP application was studied. The unknown effect on the enamel surface from application of multiple etchants is not well known and was qualitatively described in this study.

Based on the results of this study, there was significantly decreased shear bond strength of brackets that were bonded after the enamel surface was pumiced as compared to

enamel surfaces that were either rinsed with water or 34% phosphoric acid etched. From the selected specimen used to represent the three tests groups, no difference was found to exist in regards to depth of resin penetration. The pumice prophylaxis group did show a trend to have less depth of resin penetration; however, this was not statistically significant.

Shear Bond Strength

The shear bond strength values obtained in this study ranged from 5.704 to 24.766 MPa with the mean values from each of the test groups being 13.84, 11.14, and 17.83 MPa. These values are well above the suggest ideal that Reynolds and von Fraunhofer made that clinical shear bond strengths of 5.9-7.9 MPa were adequate to withstand the forces of orthodontic treatment (Reynolds and von Fraunhofer 1976). It needs to be noted that these values were only opinion based and not supported by results from a specific study. Higher values have consistently been recorded ranging from 11.3 to 19.0 MPa when using phosphoric acid-etch (Dorminey et al. 2003; Scougall Vilchis et al. 2009). A 35% phosphoric acid application for 15 and 30 seconds resulted in bond strengths of 15.3 ± 5.5 MPa and 17.6 ± 5.4 MPa respectively (Holtan et al. 1995). Torii et al. (2002) also showed resulting bond strengths of 11.2 MPa and 14.3 MPa after application of SEP alone and 35% etch plus SEP respectively (Torii et al. 2002). The noted difference in SEP application alone compared phosphoric acid etch in combination with SEP from the study by Torii et al. (2002) were supported with the trends that this study demonstrated, however, statistical significance was not achieved. According to Bishara et al., SEPs do produced a significantly lower shear bond force (7.1 ± 4.4 MPa) compared to conventional techniques using 37% phosphoric acid (10.4 ± 2.8 MPa) (Bishara et al. 2001). One possible cause of the higher values obtained in this study may be due to the application technique of the SEP. The study by Bishara stated

that the SEP was placed on the tooth for 15 seconds and thinned with air. This is different to how the SEP was applied in this study as it was scrubbed on the enamel surface for 5 seconds, as supported by current online recommendations and recent literature (Otsby 2007, Parish 2011).

Lindauer et al. looked at the effect pumicing had on orthodontic bracket bonding and reported that pumicing did not improve bond strength, etched enamel characteristics, or bracket retention rates (Lindauer et al. 1997). These results were supported in this study as the treatment protocol of pumicing the enamel prior to SEP application produced significantly weaker shear bond strengths. However this may be clinically insignificant as the mean value of 11.14 MPa for this treatment group is still above the suggested adequate shear bond strength.

The highest values of shear bond strength in this study were obtained from the 34% phosphoric acid etch plus the SEP group. This may be a concern given that the high values may cause enamel fracture on debond and thusly higher bond strengths than what the practitioner may desire. One specimen from this group did suffer an intraenamel failure at 18.183 MPa, however, one specimen from the H₂O rinse plus SEP group also suffered the same fate at 23.451 MPa. Both of these shear bond values are quite high and would indicate that shear bond strengths reaching this magnitude may be undesirable. The fact that a specific treatment protocol would create these undesirable bonding strengths is inconclusive from this study.

Resin/Enamel Interface

The mean depth of resin penetration, or the width of resin/enamel interface, in this study were 8.777 ± 2.870 , 3.890 ± 1.837 , and 7.667 ± 2.400 for the H₂O, pumice, and 34%

phosphoric acid-etch groups. These results coincide with previous reports that the use of phosphoric acid can dissolve the enamel prisms to a depth of 5-25 μm with a diameter of 5-6 μm (Reynolds 1975). Other literature has quoted the depth of resin penetration to range from 5-50 μm . The width of resin/enamel interface in this study did not approach that high of values or the 53.9 μm depth of resin penetration reported by Ramesh Kumar et al. with the use of 37% phosphoric acid or 40.5 μm when using a SEP (Ramesh Kumar et al. 2011). These differences may be due to the methods that were utilized to measure the interface. The current study used Raman spectroscopy and linear mapping acquisition to analyze the chemical compositions at 1 μm intervals across the resin/enamel interface. The study by Ramesh Kumar utilized confocal microscopy and rhodamine B dye incorporated into the resin. The major benefit of confocal microscopy is that the specimen isn't altered by processing; however, the stain is non-specific for resin and the depth of resin penetration may be overestimated due to the amount of the fluorescence shown. Another difference in the increased depth of resin penetration could be attributed to the length of phosphoric acid etch time, which was 30 seconds compared to 15 seconds in the current study.

The results of the current study did show a trend that with decreased width of resin/enamel interface, decreased shear bond strength could be anticipated. The pumice prophylaxis groups showed both the lowest shear bond strength and the smallest resin/enamel interface. Also almost all the bond failures in this group were along the resin/enamel interface. The SEP group and 34% etch group showed no statistical significance in regard to depth of resin penetration or bond strength. Increasing the specimen numbers tested may show differences between the groups.

SEM Topographical Analysis

The surface characteristics of all groups in this study did not show the same distinct uniform etching that was described to have occurred in previous studies using 37% phosphoric acid or SEP (Cal-Neto and Miguel 2006). Cal-Neto reported Type I etch patterns whereas the current study showed Type III, indistinct etched surfaces. These differences may be caused by the additional SEP application in our study further denuding the surface enamel. It was anticipated that either the H₂O rinse or pumice prophylaxis group surfaces more similarly reproduce the results obtained by Cal-Neto (2006), but this was not the case. The pumice group in our study did show surface scratches as a result of the abrasiveness of the pumice which is similar to the results found by Lindauer et al. (1997). Lack of pumicing did not significantly decrease the bond strength in the study by Lindauer et al. (1997) and led to their conclusion that there was not any clear support for pumice prophylaxis as a prerequisite for achieving adequate enamel etching and bond strength. Our study showed significantly lower bond strength after pumice prophylaxis and these surface scratches may have contributed the resultant decreased bond strength. Both studies findings may influence the clinician to reconsider the use of pumice prior to orthodontic bracket bonding.

Study Limitations

This study was not without limitations. The first and most obvious limitation would be the small sample size tested. Increasing the number of specimens in each of the test groups could lead to a greater probability of finding significance between the H₂O/pumice and the H₂O/34% etched groups. Increasing the specimen tested in cross-section could also lead to a significant difference being noted in the depth of resin penetration as in this study only selected samples from each group were chosen. More importantly, by subjecting all

specimens to both shear bond testing and Raman mapping, a correlation coefficient could be calculated to determine if any correlation exists between shear bond strength and depth of resin penetration.

Another potentially limiting factor in this study was the use of extracted maxillary third molars as specimen for bonding in an in-vitro setting. The molars were collected from patients prior to their eruption into the oral cavity. This most likely led to the specimen having minimal exposure to topical fluorides as well as minimal environmental insult from the oral cavity. These factors may have contributed a more susceptible enamel surface and resultant higher shear bond strengths. An environmental chamber was used to mimic bonding in the intraoral environment in an attempt to control both the temperature and the humidity when bonding; however, this is not an exact replica of intraoral conditions and each patient will present with some variance in condition.

This study used a crosshead speed of 1mm/minute when testing the shear bond strength. As the crosshead speed increases, bond strength increases (Finnema et al. 2010). Thus the values reported in this study may actually be less than clinical debond forces if clinical shear forces exceed 1mm/minute. However, 1mm/minute was used in the current study to allow comparisons to be made to other literature as this is the rate of shear force application which is most commonly applied in shear bond strength testing. An increased velocity of the shearing force would most likely lead to better performance of the resin and possible increased levels of shear bond forces (Finnema et al. 2010).

Clinical Significance

The focus of this study was to examine the effects of enamel surface cleaning techniques prior to self-etching primer application when bonding orthodontic brackets and the resultant bond strength and depth of resin penetration. The results from this study imply that pumicing the teeth the day of bonding can actually reduce the strength of the bond as compared to rinsing the tooth with water or applying 34% phosphoric acid etch for 15 seconds. There are patients that present prior to “bonding” with a significant amount of plaque and materia alba present which do require mechanical debridement of the enamel surfaces prior to etching. In these cases it is recommended that pumice prophylaxis take place a week prior to the bonding appointment to allow the intraoral tissues to recover and minimize fluid contamination.

The results from this study do not support the current recommendations from orthodontic company representatives that “double-etching” the enamel surface produces significantly stronger bond strength. Our study shows that while the bond strengths are larger in the double-etch group, it is not statistically significant from just rinsing the surface with H₂O. Even though the double-etch group mean shear bond strength was higher than the H₂O group, the H₂O rinse group specimen still produced more than adequate clinical bond strengths. This would be advantageous to both the orthodontist and the patient as fewer costs would be incurred from using fewer products, and the enamel surface of the patient would be exposed to less acid challenge. Excess orthodontic etching, especially beyond the bracket base limits, could result in a patient being more susceptible to white spot lesions and greater depth of lesion penetration when coupled with poor oral hygiene (Knosel et al. 2012).

One of the most important clinical applications of enamel preparation techniques is the contribution that enamel surface preparation techniques has on the resultant shear bond strength and depth of resin penetration. The results from this study show only significant difference between the pumice and 34% etch groups in shear bond strength. This may lead the clinician to believe that the type of enamel surface preparation technique really does not matter prior to SEP application. However, this is not the case as the partial eta squared values showed that a large percentage of the effect of shear bond strength and depth of resin penetration is attributable to enamel surface preparation method.

Future Investigations

Due to the convenience sample size used in this study, it would be of benefit to continue this current investigation adding more specimens to the test groups as well as complete Raman mapping of more specimens to test correlation between shear bond strength and depth of resin penetration. Even though the sample size was small, this study did account for 23 of the recommended 27 test conditions to be included in a study of orthodontic bond strength testing (Finnema et al. 2010). Continuing to look at the effects that double-etching has on the enamel surface and quantifying the enamel lost etching could potentially reduce patients being subjected to unnecessary enamel loss. Also future investigations could include a newly developed pre-loaded bracket where the resin is expressed only after compression against the enamel surface without producing any “flash”. Ideally these testing protocols could be transitioned to a split-mouth clinical study as opposed to in vitro testing.

CHAPTER 5

CONCLUSIONS

1. There was a significant difference in the self-etching primer/adhesive bracket bond strength as a function of the enamel pretreatment protocol. This difference existed only between the pumice prophylaxis and the 34% phosphoric acid-etch groups. No difference was found to exist between the pumice/H₂O and the H₂O/34% acid-etch groups.
2. There was no significant difference in depth of resin penetration across all three test groups. Trends showed less resin penetration with the pumice prophylaxis group; however, these results were not statistically significant.
3. The trends showed that with more depth of resin penetration that bond strength was better. More importantly, the pre-treatment protocol had a strong contribution to the resultant depth of resin penetration.
4. No obvious qualitative difference existed between groups with the surface etch patterns obtained. The pumice prophylaxis pretreatment group did show significant surface scratches whereas the other groups did not.

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VITA

NAME:

Michael Bruce Olsen

DATE AND PLACE OF BIRTH:

September 1st, 1981

EDUCATION:

5/1999	Diploma	Kelowna Senior Secondary Kelowna, B.C., Canada
5/2006	BSc	University of Lethbridge Lethbridge, AB, Canada
5/2011	D.D.S.	West Virginia University School of Dentistry Morgantown, West Virginia
12/2013	M.S. Oral & Craniofacial Sciences	University of Missouri – Kansas City School of Dentistry Kansas City, Missouri

INTERNSHIP AND/OR RESIDENCIES:

2011-2013	Orthodontic Residency	University of Missouri – Kansas City School of Dentistry Kansas City, Missouri
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PROFESSIONAL ORGANIZATIONS:

American Association of Orthodontists
Canadian Association of Orthodontists
American Dental Association
Canadian Dental Association
American Student Dental Association
Omicron Kappa Upsilon

HONORS:

Graduated 1st in class. West Virginia University School of Dentistry (5/2011)
Omicron Kappa Upsilon Dental Honor Society Induction (5/2011)
WVU Senior Dental Student Award (5/2011)

WVU Dental Research Student Competition Award (2/2011)
William S. Kramer Award of Excellence- Alpha Beta Chapter of Omicron Kappa Upsilon
Dental Honor Society (5/2010)
Basic Sciences Award from Omicron Kappa Upsilon Alpha Beta Chapter of West Virginia
University- highest cumulative grade point average in clinical sciences (5/2009)
West Virginia School of Dentistry Alumni Association Instrument Award (11/21/2008)
University of Lethbridge Academic Scholarship (2005)
Canadian Millennial Excellence Award for Academics (2004)
Provincial Academic Scholarship from Ministry of Education of British Columbia (2003)