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Bonding Properties of Newly Erupted and Mature Human Premolars

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BONDING PROPERTIES OF NEWLY ERUPTED AND MATURE HUMAN PREMOLARS

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at Virginia Commonwealth University.

by

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Abstract

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By Diana M. Almy, D.D.S.

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Following eruption of a tooth into the oral cavity, enamel is thought to continue to calcify. The continued calcification and maturation of enamel is described as "post-eruptive enamel maturation." It is believed that an observed decrease in enamel pore size and increase in the calcification of enamel matrix over time can be attributed to this process. In recent years, there has been a significant increase in the number of adult patients seeking orthodontic treatment. Since orthodontic attachments are bonded directly to the etched enamel using composite resin, post-eruptive enamel maturation may affect the bonding process. The purpose of this study was to determine whether there were differences in bond strength between mature and newly erupted teeth when using both conventional and self-etching primer techniques for

bonding orthodontic appliances. The nature of adhesive bond failure among the groups was also compared using an adhesive remnant index (ARI). Etched surfaces were examined under scanning electron microscopy (SEM) and representative photomicrographs were taken. Human premolars were collected and bonded randomly with either the conventional or self-etching technique. Brackets were debonded using an Instron testing machine in shear-testing mode. There were no statistically significant differences in the bond strengths between the self-etching primer and conventional etching groups. ARI scores showed differences between mature and newly erupted teeth. Mature teeth had more cohesive bond failures whereas newly erupted teeth had more adhesive failures at the enamel-composite resin interface. Scanning electron micrographs (SEMs) of self-etched enamel revealed smooth areas of resin with filler particles. Conventionally etched enamel had rougher surfaces. There were no differences in etch pattern of new versus mature enamel. Further research may be needed as new bonding materials and techniques become available to determine the effects, if any, of post-eruptive enamel maturation on their success.

Introduction

Odontogenesis, a series of events taking place from bud formation stage until the completion of calcification and maturation of the tooth, is a complex and misunderstood process. Odontogenesis was once assumed to end as soon as the erupting tooth reached the oral cavity. However, today it is known to continue even after the tooth is fully erupted. This phenomenon is explained as "post-eruptive maturation".[1]

Upon eruption the outermost layer of enamel is immature and not fully calcified. This outer layer then begins to calcify due to the effects of salivary minerals. Calcification continues in the bottom of the subsurface inner layer and continues until it is complete.[1] At the time of eruption, human enamel contains a high amount of mineral. However, it has been shown that mineralization is incomplete in the surface of the enamel due to porosity.[2] The porosity has also been shown to fluctuate seasonally with enamel porosity measured in the Fall to be less thick than in the Spring.[3] Steifel and Binus[4] reported that the process of mineralization takes more than five years to complete after eruption of a tooth into the oral cavity.

During aging, hydroxyapatite crystals increase in size due to the incorporation of ions from the surrounding saliva. This in turn results in a decrease in the enamel pore size.[5] Briner and Rosen[6] attributed this to the mineralization of calcium and phosphate ions taken up from saliva and bacterial acid products. In their work with rats, they showed that fluoride increased post-eruptive maturation of enamel while another known

inhibitor of hydroxyapatite crystal growth, disodium ethane-1-hydroxy-1-diphosphonate, caused a decrease in the rate of maturation. Therefore, immature young enamel with large pores in its superficial layers could be readily calcified by ions derived from the saliva, food and drink.

Previous studies on post-eruptive maturation and its effects on caries development have shown a decrease in the incidence of caries in individuals as age increased, presumably due to continuous mineralization and maturation of the enamel. The reduction was especially marked shortly after eruption. This is not surprising as demineralization by the bacterial acids and subsequent caries formation would be less likely to occur on a highly mineralized surface than a less mineralized one.[7]

One SEM study showed that lingual and buccal surfaces of mature teeth have a prismless enamel structure. However, the same areas of newly erupted teeth are prismatic.[8] Since there are structural differences between mature and newly erupted teeth, it is logical to expect differences in bond strengths of brackets bonded to mature teeth versus newly erupted teeth.

In 1955, Buonocore[9] introduced the direct bonding concept to dentistry. The direct bonding technique consists of adhering the brackets onto teeth with composite resin after etching the enamel surface with phosphoric acid. In this technique, enamel preparation with acid etching creates a porous surface which provides a mechanical bond between the bracket and tooth. By the early 1980’s, bonding orthodontic brackets with

the acid etching technique became the gold standard in the orthodontic profession. Bond strengths of 6-8 MPa are reported to be adequate to withstand the forces generated by orthodontic wires while also allowing for the removal of brackets without causing damage to the enamel at the end of the treatment during the debonding procedure.[10]

In the 1990's, self-etching primers were introduced for use in orthodontics. The main advantage of using self-etching primers is to save chairside time for the clinician as the etching and primer application steps are combined into one step. These products consist of phosphoric acid and hydroxyethylmethacrylate (HEMA). Upon application, hydrophilic HEMA molecules penetrate into the deeper layers of the enamel and phosphoric acid etches the surface of the tooth. Phosphoric acid does not need to be rinsed off as in the conventional systems because, after its penetration into the enamel, it is neutralized. The calcium ions that are dissolved by etching do not need to be rinsed away because they are incorporated into the primer matrix.

Numerous *in vitro* studies have shown that some self-etching primer systems exhibit lower bond strengths than the conventional phosphoric acid products do while other self-etching primer systems exhibit higher bond strengths.[11,12,13] Other *in vitro* studies have shown no difference between conventional and self-etching techniques when both are used as recommended by the manufacturer.[14] Although some reported bond strengths are lower for the self-etching systems, most values fall within the clinically acceptable range needed for the orthodontic attachments to be able to withstand the

forces generated by the wires. When compared clinically in a split mouth study over a period of six months, the self-etching primer group was found to have statistically less bond failures than the conventionally etched group.[15] Recently, these products have become very popular in orthodontics because they provide an acceptable range of bond strengths in addition to substantially decreasing chair time during bracket bonding.

Studies measuring bond strengths of orthodontic brackets bonded using the conventional acid etching technique show that newly erupted permanent teeth produce more retentive conditions with 15 seconds of etching time. However, mature permanent teeth require 60 seconds of etching to provide similar retentive conditions. [16] This may be due to the greater calcium content of the mature teeth. Therefore, since the newly erupted teeth are less mineralized, less etching time may be needed to create the surface irregularities required for bonding, or more time is needed when etching the mature teeth.

SEM studies of conventional phosphoric acid etch patterns of unerupted and erupted teeth show that both groups have similar microstructures suggesting no differences in etching.[17] Oliver[18] compared bond strengths of newly erupted and unerupted teeth using conventional etching techniques and found no difference in bond strengths. In the literature, there are many studies which evaluated bond strength of conventional and/or self-etching primer systems; however, none of them considered the possible effects of the maturation level of enamel on the etching quality. Studies that took enamel maturation into account used conventional etching techniques.

Recently, there has been a substantial increase in the number of adult patients seeking orthodontic treatment. For such patients, it may be necessary to etch the enamel longer than the suggested time due to the effects of post-eruptive maturation. The aims of this study were: 1) to determine the effects of post-eruptive maturation on the etching and bond strength of orthodontic brackets using both conventional and self-etching primer techniques on mature and newly erupted teeth, 2) to determine the mode of bond failure by examining the bracket and tooth surfaces using an adhesive remnant index (ARI), and 3) to analyze the differences in etching patterns between mature and newly erupted teeth using scanning electron microscopy.

Materials and Methods

Bond Strength Analysis

For this study, maxillary and mandibular human premolars extracted for orthodontic treatment were collected from young patients 9 through 14 years of age to serve as the newly erupted group and adult patients older than 21 to serve as the mature group. Following extraction, teeth were individually stored in 10% formalin at room temperature until the experiment began. Only healthy teeth with no apparent defects on their buccal and lingual surfaces were included in the study. Teeth were randomly assigned to the conventional etch (N=16) and self-etch primer bonding groups (N=16). Due to the difficulty in collecting sound adult premolar teeth, both buccal and lingual surfaces of the teeth were used for bonding. Previous studies have shown that there are no significant differences in the bond strength between buccal and lingual surfaces.[19,20] The four experimental groups were as follows: Mature/Conventional Etch, Mature/Self-Etching Primer, Newly Erupted/Conventional Etch, and Newly Erupted/Self-Etching Primer.

A single operator performed all of the bonding procedures. Initially, the buccal and lingual surfaces of the teeth were cleaned with a rubber prophy cup and a fluoride free pumice to eliminate any contaminants. The teeth were then thoroughly rinsed. For enamel surface preparation, the following techniques were used in the conventional and SEP groups, respectively. Teeth in the conventional group were etched with 37%

phosphoric acid gel (3M Unitek, Monrovia, CA) for 30 seconds, rinsed with water for 10 seconds and air-dried using an oil free air-water syringe until surfaces had a frosty white appearance as recommended by the manufacturer. Adhesive primer (Moisture Insensitive Primer, 3M Unitek, Monrovia, CA) was applied to the etched surface of the specimens and gently air dried. Teeth in the self-etch groups were etched using the self-etching primer (3M Unitek, Monrovia, CA) also following the manufacturer's instructions. Adhesive pre-coated brackets (APC II Mini Twin Bicuspid, 3M Unitek, Monrovia, CA) were positioned in the center of the crown and firm pressure was applied. Any excess composite was removed. The adhesive was light-cured for three seconds from the mesial and three seconds from the distal aspects as recommended by the manufacturer using a plasma arc visible-light curing unit (Ortholite, 3M Unitek, Monrovia, CA).

Following bonding, the teeth were individually embedded in phenolic rings using cold cure acrylic covering the root surface up to the cementoenamel junction. Teeth were stored in deionized water at 37°C for at least 7 days.

All of the specimens were debonded on the same day by the same operator. During testing, the specimen was seated on a custom-made attachment that could be positioned at different angulations. The sample holder was tilted until the bracket slot was parallel to the upper member of the Instron to ensure parallelism between the bracket surface and the testing machine as seen in Figure 1.

Figure 1: Picture of specimen in testing machine. Blade is parallel to the bracket slot.

Specimens were debonded in the shear/peel mode using a crosshead speed of 0.02 in/min. The force required to debond the bracket was recorded. The shear strength was calculated by dividing the force by the bracket base area ($0.01671 in^2$).

Adhesive Remnant Index Measurement

Following debonding, specimens were examined under a stereomicroscope at 10X magnification to determine the location of the bond failure using an adhesive remnant index (ARI).[21] Each tooth was given a score of one through five based on the amount of composite left on the tooth.

Table I: Adhesive Remnant Index

1	**2**	**3**	**4**	**5**	
All adhesive on enamel	*More than 90%*	*10%>90%*	*Less than 10%*	*No adhesive on the enamel*	*Enamel Fracture*

Statistical Analyses

The mean shear bond strength and standard deviation for each test group was calculated and a two-way ANOVA was used to determine differences among the four groups. T-tests were used for statistical analyses when comparing two groups (e.g. conventional vs. self-etching primer groups). A chi-square test was used to determine if there were significant differences in the ARI scores. Data was also analyzed using Weibull analysis.

Weibull Analysis

Clinically, a mean bond strength value alone cannot be taken as the indicator of the quality of the bond as there may be significant factors contributing to the mechanical behavior of the tested specimen. In brittle materials, inevitable flaws that are present in the material would cause a considerable variation in the bond strength values of the tested specimens. Additionaly, improper alignment on the testing machine, the surface characteristics of the enamel and other factors may affect the mechanical behavior of the

tested specimen. Weibull analysis is the choice of method when dealing with such problems. This method provides a “weakest-link-in the-chain” distribution and is therefore interested in the first failure. For example, if a failed material could be re-tested, one would expect higher strength values as the weakest flaw has already been eliminated from the specimen. Finally, the primary advantage of the Weibull analysis is that it provides reasonably accurate failure analysis and failure forecasts even with extremely small samples.

The equation for the Weibull cumulative distribution function is given by:

$F(t) = 1 - e$ ^ - {[(t - γ)/η] ^ β} with $t > \gamma$.

Where: e = natural log base 2.718.

t = parameter of interest, or x value. In the present study this is the debonding stress.

γ (Gamma) = x value shift, threshold, location parameter, or Weibull 3rd parameter. Gamma is the location parameter that moves the plot right or left to get a better fit of the data to a straight line. Gamma is the debond stress value below which the probability of failure is zero. Its value moves the plot left for $\gamma > 0$ or right for $\gamma < 0$. For a two parameter plot gamma equals zero.

η = Characteristic level, life scale parameter. It is the characteristic life or Weibull “mean” strength. It is treated as a ‘mean’ for the plot and corresponds to the 63.2% of the median rank on the straight line: $0.632 = 1 - 1/e$.

β = Slope, Weibull modulus, shape factor. It is the slope which correlates with precision. This factor indicates the type of probability distribution, e.g.:

β =1 for an exponential distribution.

β < 2.6 for a positive skewed distribution with a right tail.

2.6 < β <3.7 for a zero coefficient of skewness with no tail, or a normal distribution.

β > 3.7 for a negative skewed distribution with a left tail.

Scanning Electron Microscopy

For the scanning electron microscopic studies, four mature and four newly erupted teeth were randomly etched on the buccal and lingual surfaces with either the conventional etching technique or self-etching primer as previously described. Teeth were air dried and sputter-coated with a thin gold-palladium alloy to prevent electrical charging during the SEM examination. The surfaces were examined under SEM and representative photomicrographs were taken at various magnifications to obtain information on the etching patterns.

Results

Bond Strength Analysis

Bond strength values were compared by race, gender, arch and surface. No significant differences were found based on a t-test. The mean shear bond strength values and standard deviations were as follows: African-American (N=12) 13.26 ± 8.36 MPa, Asian (N=16) 14.52 ± 3.74 MPa, and Caucasian (N=36) 10.89 ± 5.55 MPa. The mean shear bond strength values and standard deviations were as follows: Male (N=40) 13.26 ± 6.51 MPa and Female (N=24) 10.55 ± 4.43 MPa. There was a trend toward males having higher bond strengths than females. However; there were no statistical differences in the bond strength values (p=0.08). When bond strength values were compared between maxillary and mandibular arches, no significant differences were found (p=0.28). The mean shear bond strength for the mandibular arch (N=24) was 11.20 ± 6.40 MPa. The mean shear bond strength for the maxillary arch (N=40) was 12.87 ± 5.62 MPa. Therefore, all groups were combined for further testing.

The mean shear bond strength, standard deviation, and range for the buccal and lingual surface groups are shown in Table II and illustrated in Figure 2. The mean shear bond strength for the buccal surface group was 13.12 ± 6.54 MPa. The mean shear bond strength for the lingual surface group was 11.37 ± 5.20 MPa. No significant differences

(p=0.24) were found between the two groups based on a t-test. Therefore, buccal and lingual surfaces were grouped together for further testing.

Table II: Bond Strength in MPa for the buccal and lingual surface groups.

	Mean ± SD (MPa)	**Range (MPa)**
Buccal Surface	13.12 ± 6.54	2.06-27.85
Lingual Surface	11.37 ± 5.20	2.27-18.86

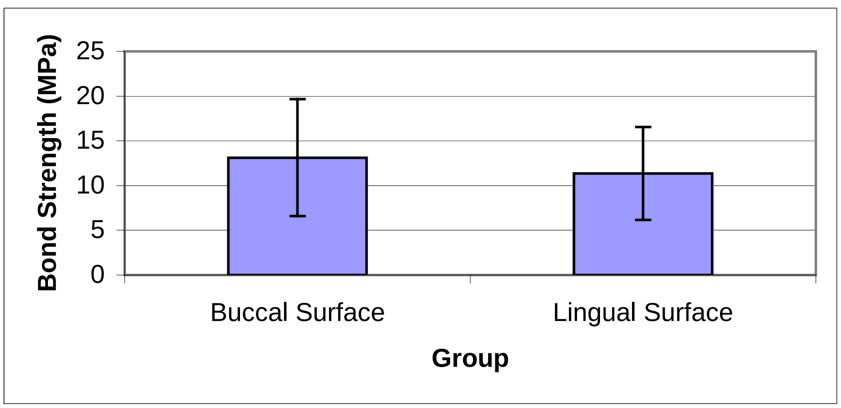

Figure 2: The mean shear bond strength for the buccal and lingual surfaces (± standard deviations) (p=0.24).

Data were also analyzed using Weibull distribution. The findings from this statistical analysis method agreed with those of the t-test and ANOVA; there were no differences in the strength values between the groups. The two parameter Weibull distribution plot can be seen in Figure 3. Data set one is buccal data and data set two is lingual data. Since the 95% Confidence Limits (CLs) overlap, lingual and buccal data are not different.

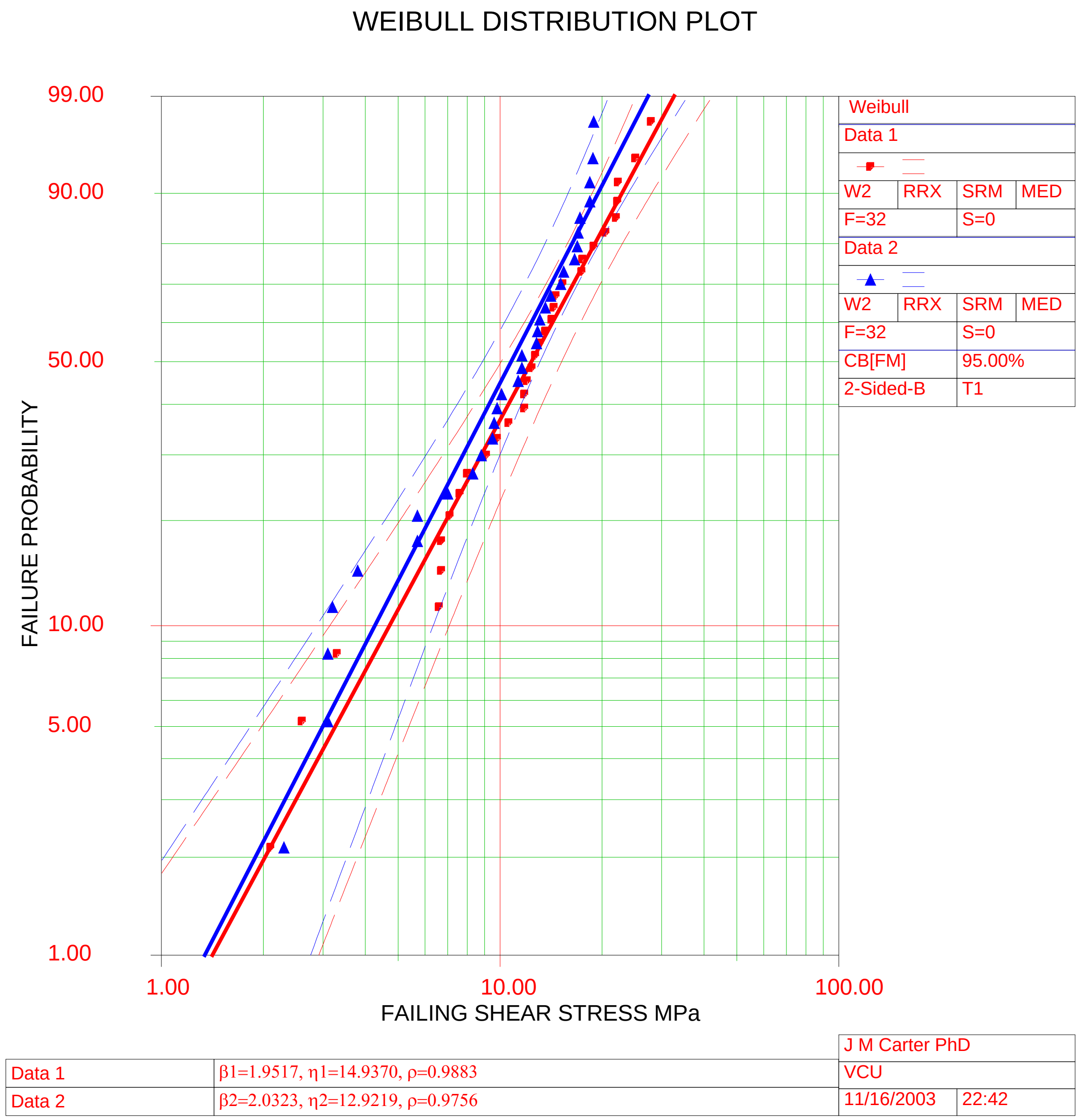

Figure 3: Two-Parameter Weibull distribution plot. Data set one is the buccal data and data set two is lingual data. Since the 95% CLs overlap, lingual and buccal data are not different.

The Weibull parameters for the data are listed underneath the X-axis. BETA (β) 1 and 2 represent the slopes of the fitted lines. The slope values or β were 1.95 for buccal and 2.03 for lingual. The greater the slope value, the smaller the dispersion in the strength values, which indicates greater reliability in the bond/alignment/and other variables in the experiment. The ETA value, A.K.A the scale parameter or Weibull characteristic strength, corresponds to a strength value with a failure probability of 63.2%. A horizontal line is drawn at 63.2 on the Y axis to cut the regressed plot line, and then dropped to the X-axis for the strength value. Rho (ρ) represents the correlation coefficient, and is a measure of how well the linear regression model fits the data. The closer to one, the better the linear fit. A zero coefficient would indicate data that are randomly scattered. Both data sets have values very close to one indicating a highly linear fit.

The contour plot of buccal and lingual bond strength data is seen in Figure 4. Since the plots overlap, there is no difference between the two groups.

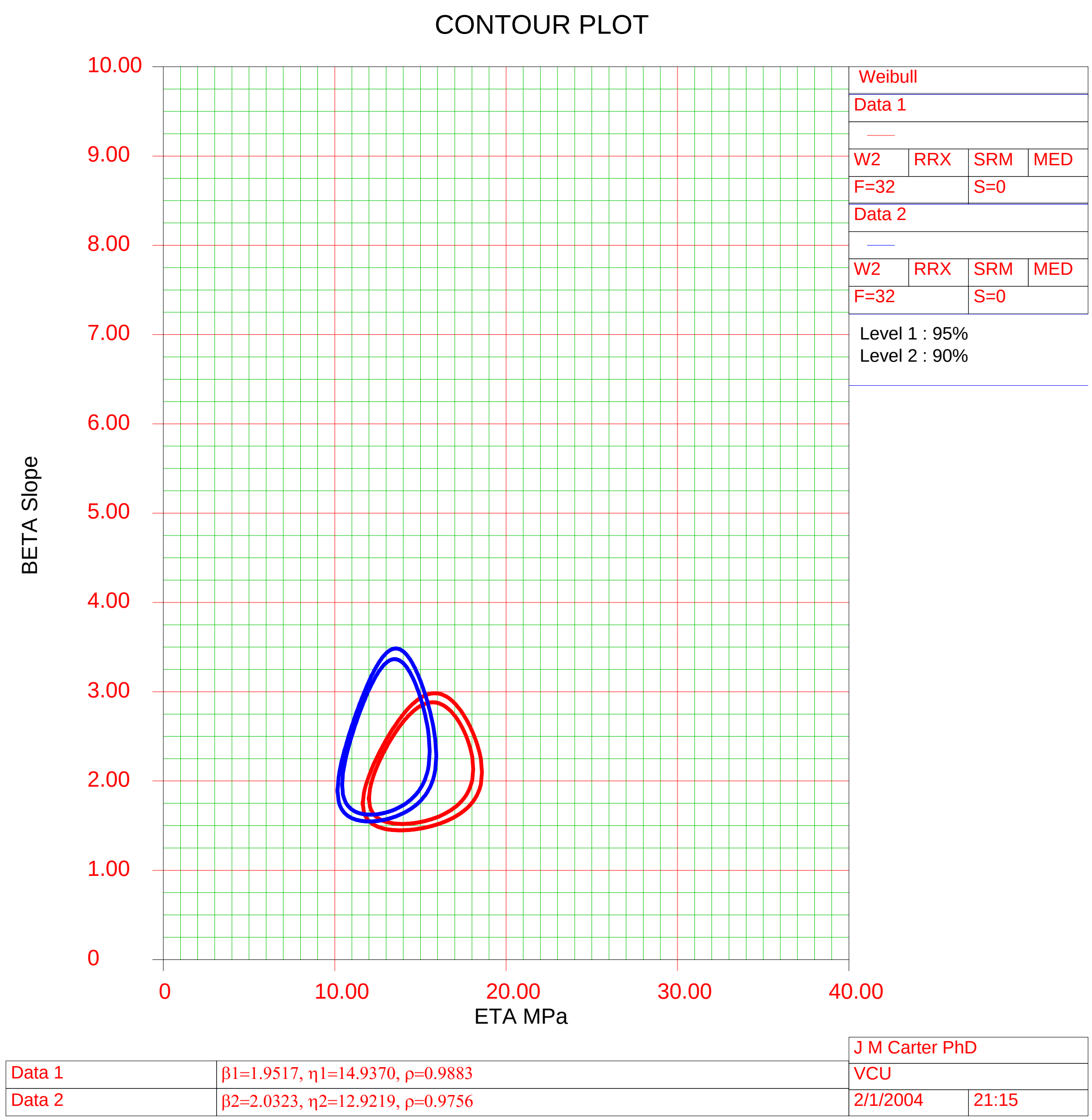

Figure 4: Contour plot of buccal vs. lingual bond strengths. Data set one is the buccal data and data set two is the lingual data. Since the plots overlap, there is no difference between the two groups.

It was of special interest to see whether there were differences between the lingual and buccal surfaces of the teeth. As mentioned earlier, both teeth surfaces were used in order to provide enough samples for the study since it was difficult to collect teeth from adult patients.

The mean shear bond strength, standard deviation, and range for the four sample groups (Mature/Conventional Etch, Mature/Self-Etch, Newly Erupted/Conventional, Newly Erupted/Self-Etch) are shown in Table III and illustrated in Figure 5. The mean shear bond strength for the groups were as follows: Mature/Conventional Etch 13.63 ± 4.75 MPa, Mature/Self-Etch 12.16 ± 5.49 MPa, Newly Erupted/Conventional 13.42 ± 7.74 MPa, and Newly Erupted/Self-Etch 9.77 ± 5.00 MPa. No significant differences ($p=0.23$) were found among the groups based on two-way analysis of variance (ANOVA) (Figure 5).

Table III: Bond Strength in MPa for the four sample groups.

	Mean ± SD (MPa)	**Range (MPa)**
Mature/Conventional Etch	13.63 ± 4.75	6.70-22.30
Mature/Self-Etch	12.16 ± 5.49	2.30-20.50
Newly Erupted/Conventional Etch	13.42 ± 7.74	2.10-27.90
Newly Erupted/Self-Etch	9.77 ± 5.00	2.60-17.40

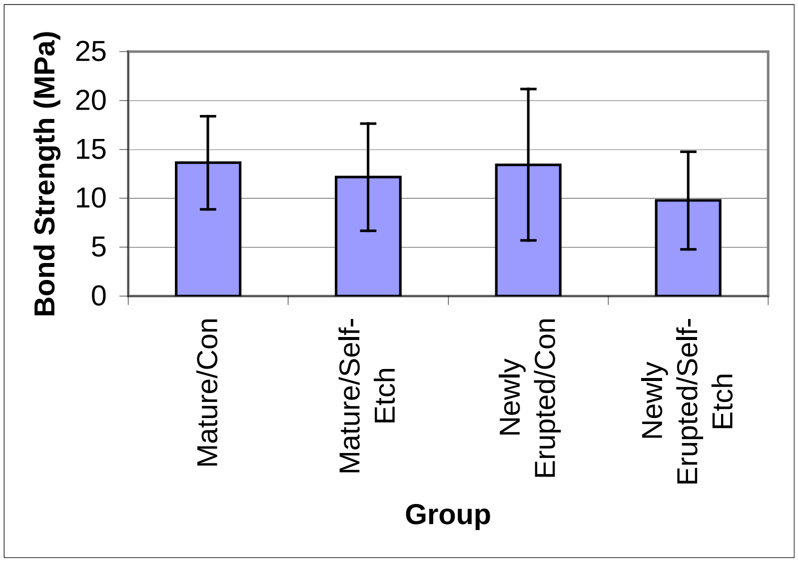

Figure 5: The mean shear bond strength of each group.

The mean shear bond strength, standard deviation, and range for the groups divided into mature and newly erupted sample groups disregarding etching procedure are shown in Table IV and illustrated in Figure 6. The mean shear bond strength for the mature group was 12.89 ± 5.10 MPa. The mean shear bond strength for the newly erupted group is 11.59 ± 6.67 MPa. No significant differences (p=0.38) were found between the groups based on a t-test (Figure 6).

Table IV: Bond Strength (MPa) for the mature and newly erupted groups.

	Mean ± SD (MPa)	**Range (MPa)**
Mature	12.89 ± 5.10	2.30-22.30
Newly Erupted	11.59 ± 6.67	2.10-27.90

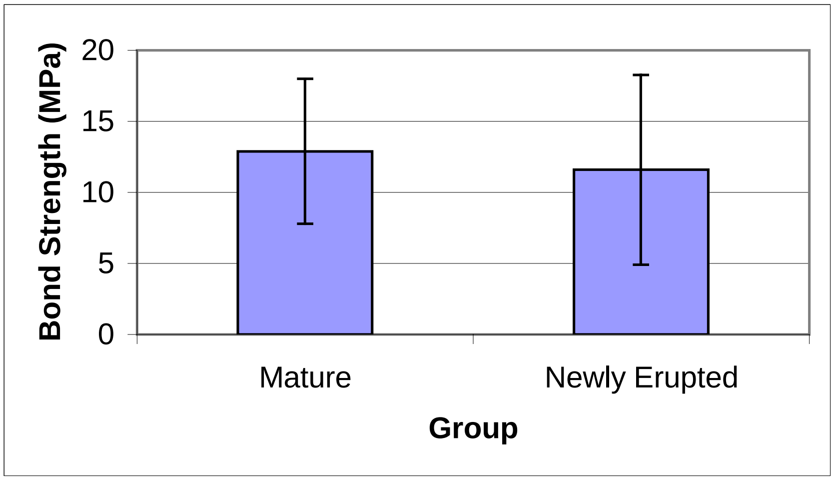

Figure 6: The mean shear bond strength of each age group.

Data were also analyzed using Weibull distribution. The findings from this statistical analysis method agreed with those of the t-test and ANOVA; there were no differences in the strength values between the groups. The two parameter Weibull distribution plot can be seen in Figure 7. Data set one is mature teeth data and data set two is newly erupted teeth data. Since the 95% Confidence Limits (CLs) overlap, mature and newly erupted data are not different.

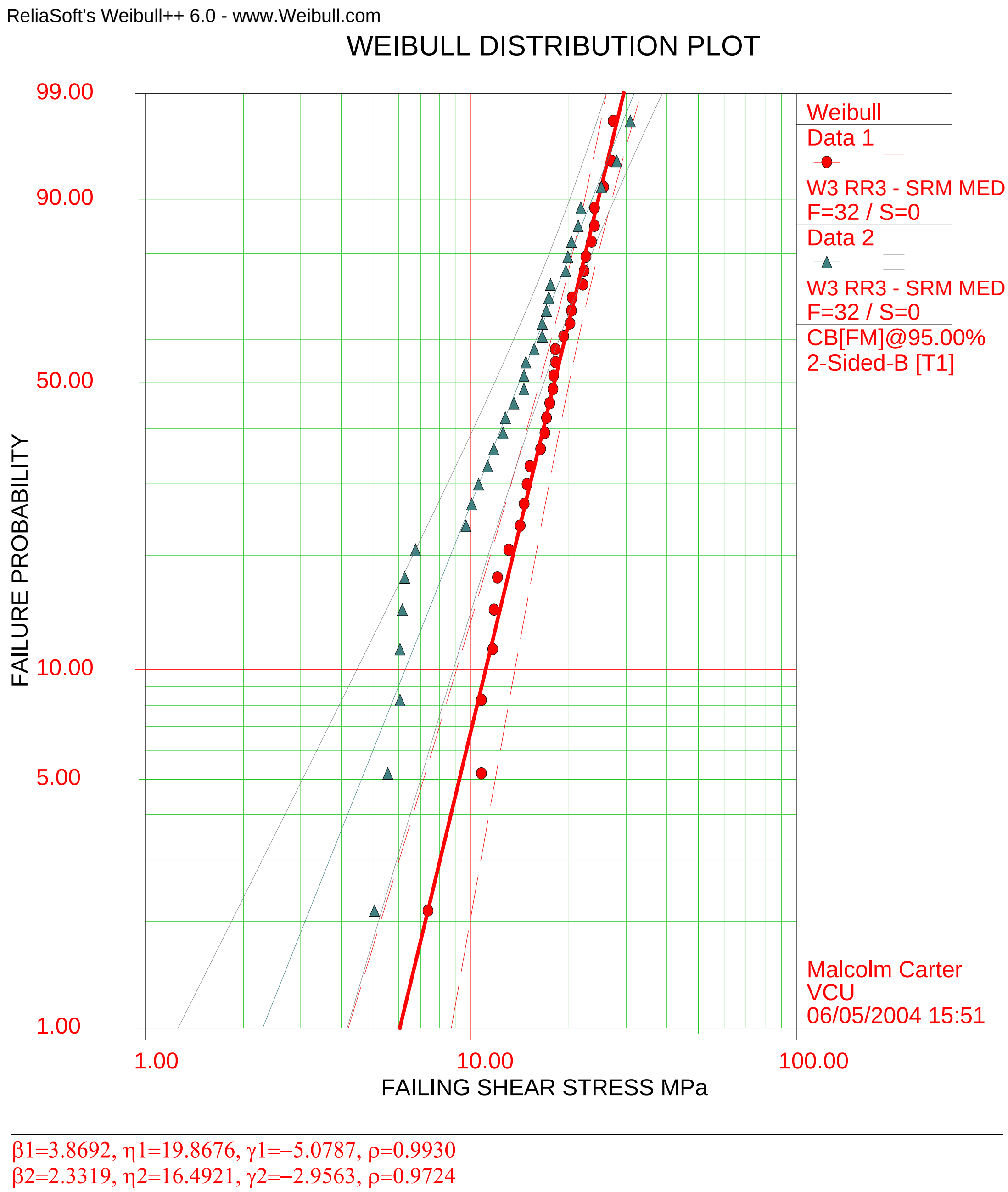

Figure 7: Two-Parameter Weibull distribution plot. Data set one is the mature data and data set two is newly erupted data. Since the 95% CLs overlap, mature and newly erupted data are not different.

The Weibull parameters for the data are listed underneath the X-axis. BETA (β) 1 and 2 represent the slopes of the fitted lines. The slope values or β were 3.87 for the mature data and 2.33 for the newly erupted data. The greater the slope value, the smaller the dispersion in the strength values, which indicates greater reliability in the bond/alignment/and other variables in the experiment. The rho (ρ) value indicates both data sets have values very close to one indicating a highly linear fit.

The contour plot of mature and newly erupted bond strength data is seen in Figure 8. Since the plots overlap, there is no difference between the two groups.

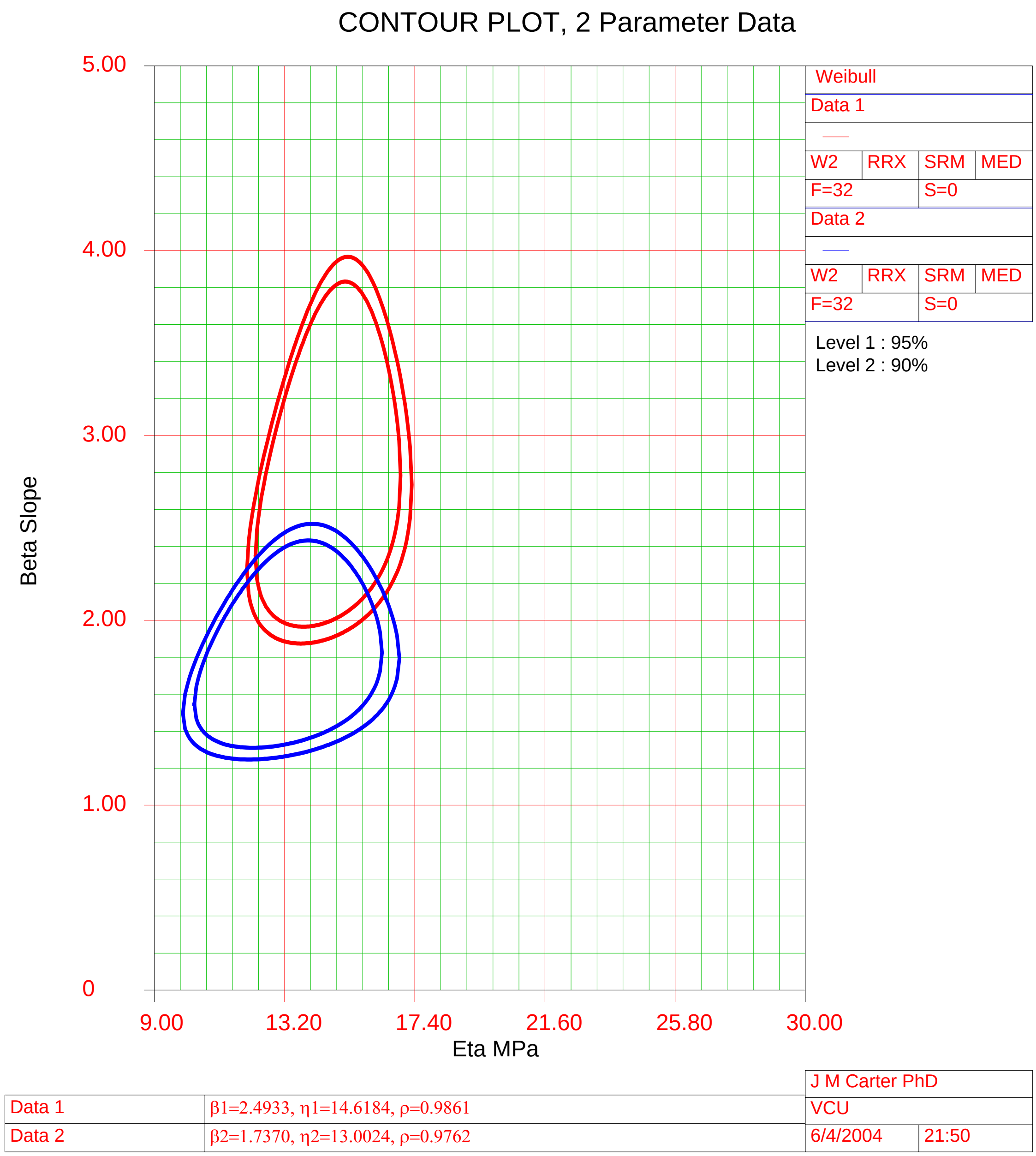

Figure 8: Contour plot of mature vs. newly erupted bond strengths. Data set one is the mature data and data set two is the newly erupted data. Since the plots overlap, there is no difference between the two groups.

The mean shear bond strength, standard deviation, and range for the groups divided into self-etch and conventional etch sample groups disregarding maturity are shown in Table V and illustrated in Figure 9. The mean shear bond strength for the self-etch group was 10.96 ± 5.30 MPa. The mean shear bond strength for the conventionally etched group was 13.53 ± 6.32 MPa. Although there was a tendency for the self-etch group to have slightly lower bond strength values, no significant differences were found between the groups based on a t-test (p=0.08) (Figure 9).

Table V: Bond Strength in MPa for the two etching technique groups.

	Mean ± SD (MPa)	**Range (MPa)**
Conventional Etching	13.53 ± 6.32	2.10-27.90
Self-Etching	10.96 ± 5.30	2.30-20.50

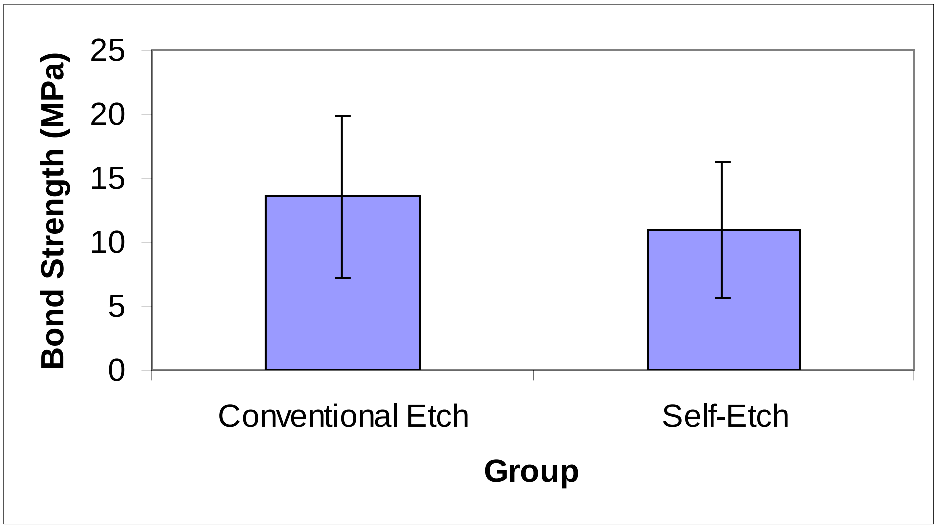

Figure 9: The mean shear bond strength of the conventional and self-etch groups.

Data were also analyzed using Weibull distribution. The findings from this statistical analysis method agreed with those of the t-test and ANOVA; there were no differences in the strength values between the groups. The two parameter Weibull distribution plot can be seen in Figure 10. Data set one represents SEP data while data set two represents conventional etching data. Since the 95% Confidence Limits (CLs) overlap, SEP and conventional etching data are not different.

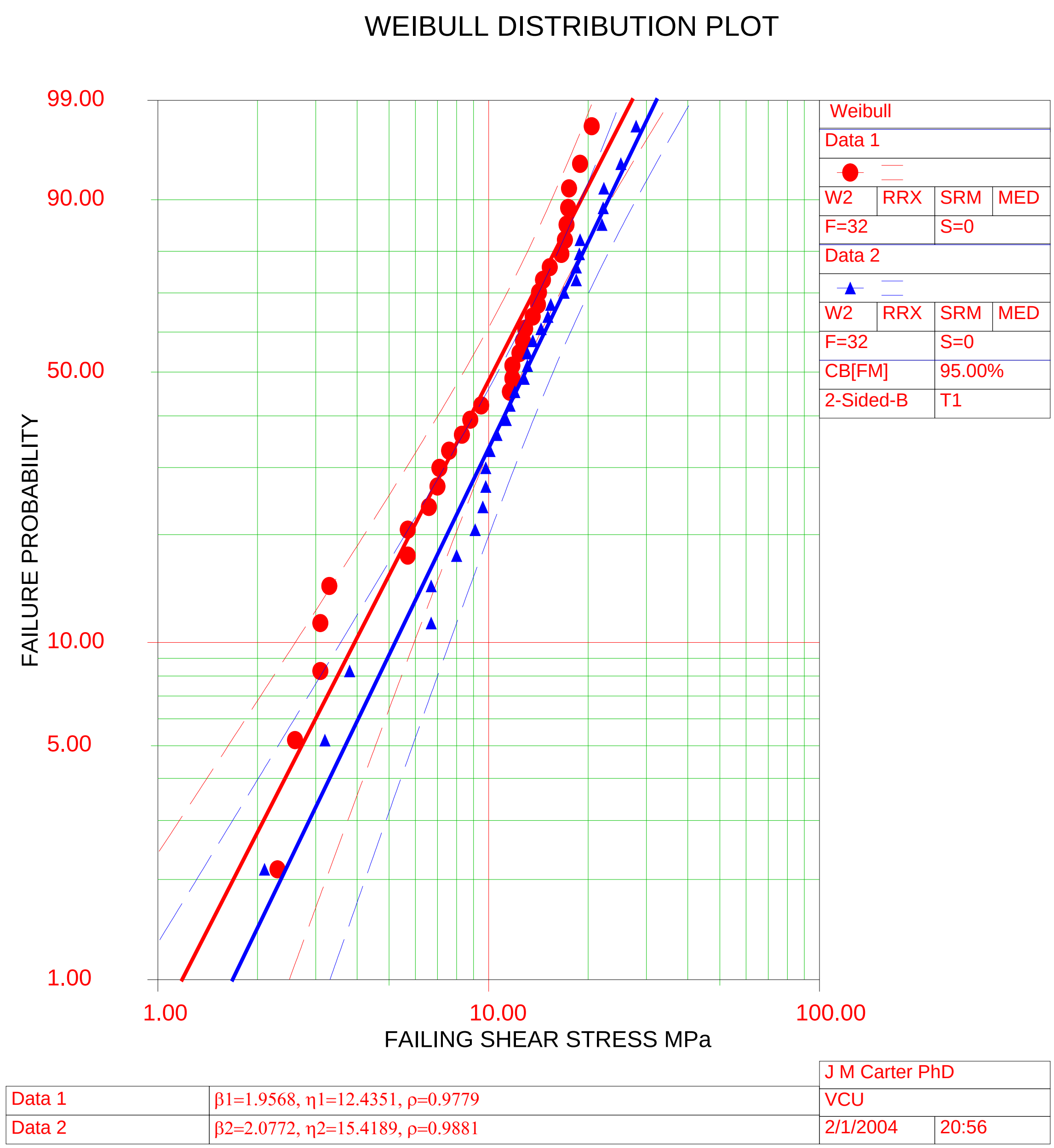

Figure 10: Two-Parameter Weibull distribution plot. Data set one is the SEP data and data set two is the conventional etching data. Since the 95% CLs overlap, SEP and conventional etching data are not different.

The Weibull parameters for the data are listed underneath the X-axis. BETA (β) 1 and 2 represent the slopes of the fitted lines. The slope values or β were 1.96 for the SEP group and 2.08 for the conventionally etched group. The greater the slope value, the smaller the dispersion in the strength values, which indicates greater reliability in the bond/alignment/and other variables in the experiment. Rho (ρ) values indicate that both data sets have values very close to one indicating a highly linear fit.

The contour plot of SEP and conventional etching bond strength data is seen in Figure 11. Since the plots overlap, there is no difference between the two groups.

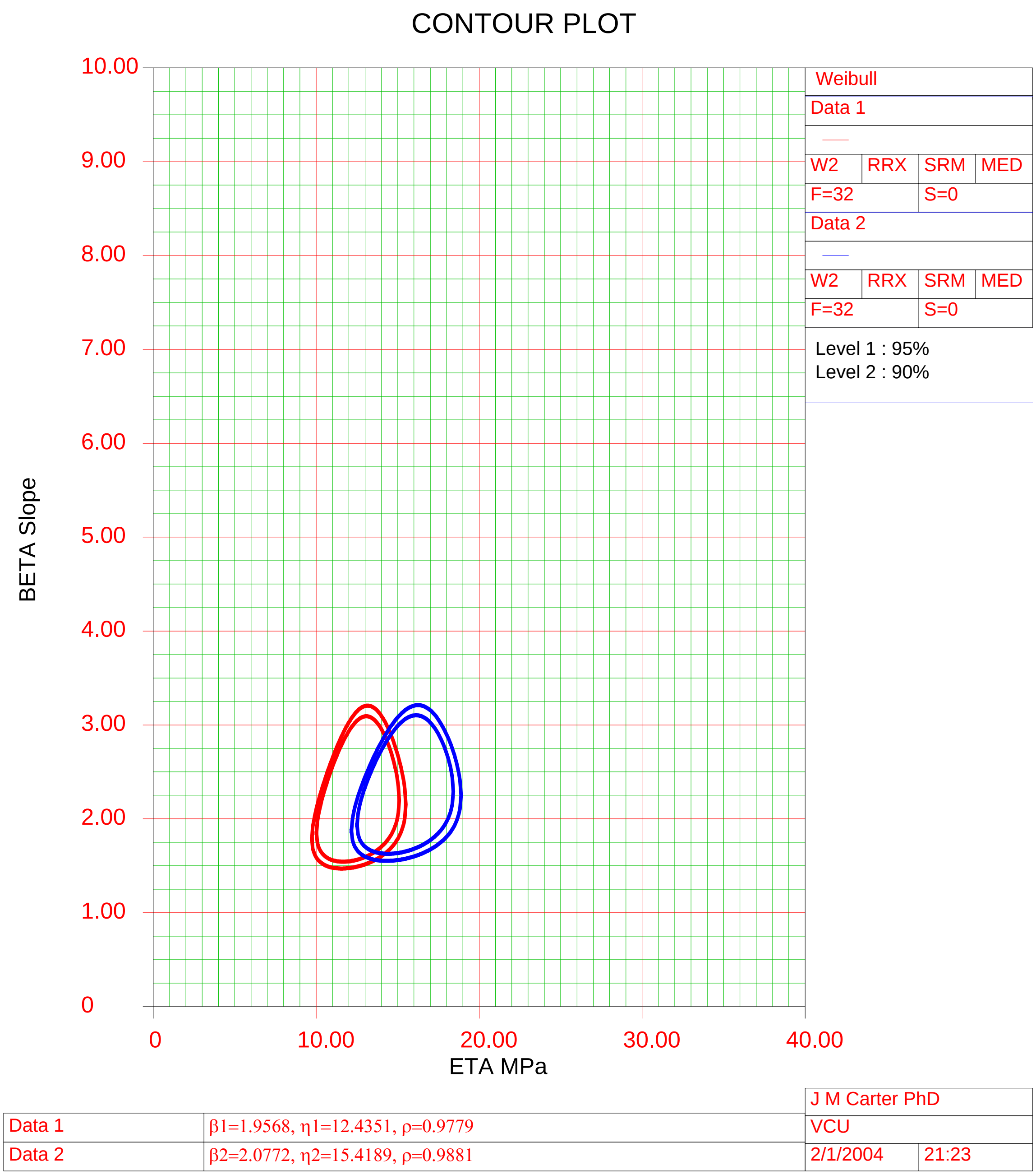

Figure 11: Contour plot of SEP vs. conventional etching bond strengths. Data set one is the SEP data and data set two is the conventional etching data. Since the plots overlap, there is no difference between the two groups.

Adhesive Remnant Index Analyses

Chi-square analyses showed that there were no significant differences in adhesive remnant index between buccal and lingual surface groups (p=0.710). When ARI scores for the maxillary and mandibular arch groups were compared, no significant differences were found (p=0.72).

Chi-square analysis of pooled data showed no significant differences in ARI between the conventional and SEP groups (p=0.68).

ARI scores for comparison between age groups are seen in Table VI and Figure 12. Chi-square analysis of pooled data for differences according to age showed significant differences between the groups (p=0.0002). It was found that mature teeth had more cohesive bond failures; whereas, newly erupted teeth had more adhesive bond failures.

Table VI: Residual adhesive ratings according to the ARI for each age group.

ARI Score	**1**	**2**	**3**	**4**	**5**	
	All adhesive on enamel	*More than 90%*	*10%>90%*	*Less than 10%*	*No adhesive on the enamel*	*Enamel Fracture*
Mature	0 (0%)	2 (3.5%)	14 (25%)	11 (19%)	1 (1.75%)	5
Newly Erupted	0 (0%)	1 (1.75%)	5 (9%)	8 (14%)	15 (26%)	4

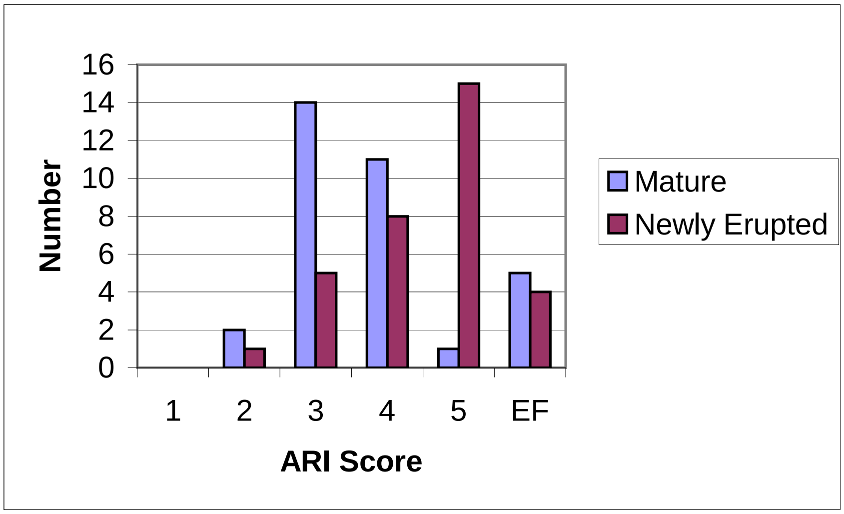

Figure 12: Percentage of bracket failure mode (ARI) for each group.

Results of the Adhesive Remnant Index Scores for the four tests groups (i.e. Mature/Conventional Etch, Mature/Self-Etch, Newly Erupted/Conventional, Newly Erupted/Self-Etch) also revealed significant differences (p=0.002). The difference was most likely due to the age group differences.

Results of the Adhesive Remnant Index (ARI) scores comparing samples by race (African-American, Asian, and Caucasian) were not statistically significant (p=0.15). When the ARI scores for the male and female groups were compared using a chi square test, no significant differences were found (p=0.98).

Scanning Electron Microscopy

Photomicrographs of enamel surfaces etched with the self-etching technique are provided in Figures 13 and 14. Self-etched enamel surfaces had a smooth appearance with no distinct dissolution. There were no differences in the enamel surfaces of young vs. mature and buccal vs. lingual samples.

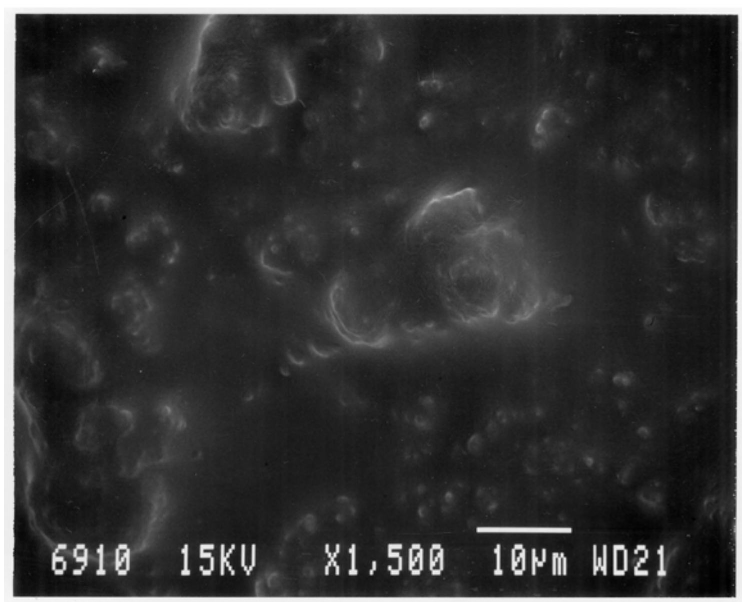

Figure 13: Scanning electron micrograph of the buccal surface of a newly erupted premolar etched using the self-etching primer technique (X1500).

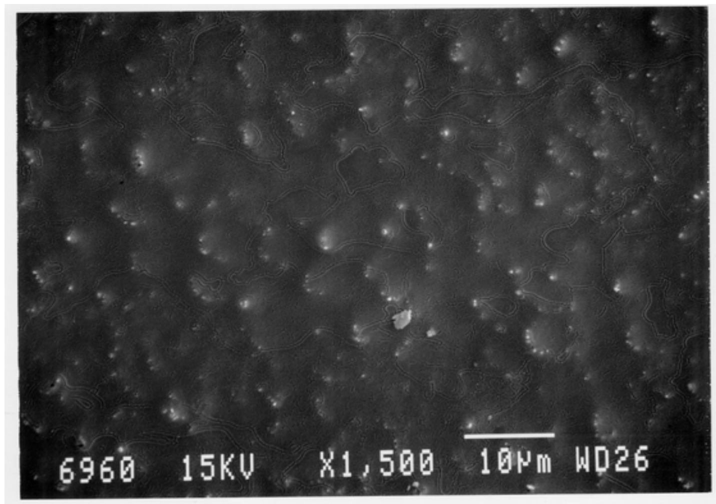

Figure 14: Scanning electron micrograph of the lingual surface of a mature premolar etched using the self-etching primer technique (X1500).

Figures 15 and 16 are from the specimens with enamel surfaces etched with phosphoric acid. Etched surfaces showed enamel rods with microporosity due to the dissolution of enamel prism cones. The pore sizes ranged from 5 to 10 microns. The newly erupted teeth seemed to have an enamel surface with larger pores compared to the mature teeth when the conventional phosphoric acid etching technique was used.

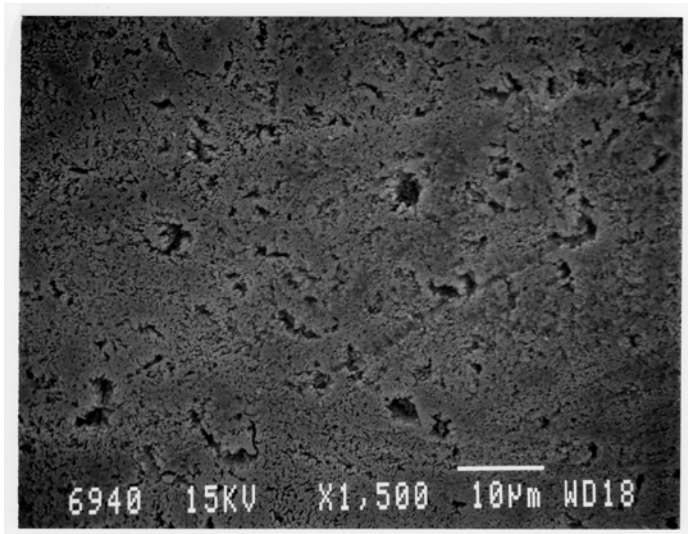

Figure 15: Scanning electron micrograph of the buccal surface of a mature premolar conventionally etched (X1500).

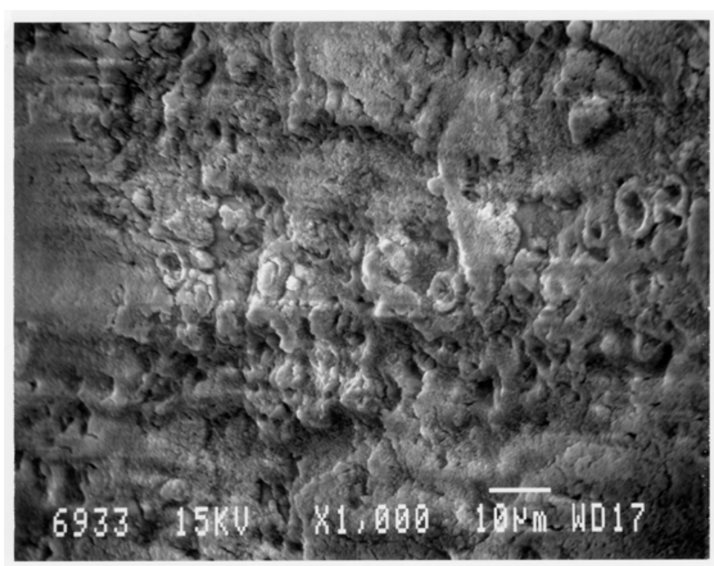

Figure 16: Scanning electron micrograph of the lingual surface of a newly erupted premolar conventionally etched (X1000).

Discussion

This study evaluated bond strengths of brackets bonded on mature and newly erupted teeth using either the conventional etch or self-etching primer techniques. Sample surfaces were also examined to determine the mode of failure using an ARI index. SEM studies were performed on the etched surfaces and representative micrographs were taken.

Since both buccal and lingual surfaces of the premolars were used for shear bond strength testing, it was of special interest to compare bond strength values between the two groups. Statistical analyses showed that the differences were not significant ($p=0.24$). Results of the Weibull analysis also agree with this. When using a two-parameter analysis at 95% confidence levels there were no significant differences between the buccal and lingual surfaces. Both sets of data also had rho (ρ) values close to one indicating a highly linear fit. The slope values or β were 1.95 for buccal and 2.03 for lingual. The greater the slope value, the smaller the dispersion in the strength values, which indicated greater reliability in the bond/alignment/and other variables in the experiment. The results of this study agree with those obtained in previous studies.[19,20] Therefore, one may conclude that using both buccal and lingual surfaces of teeth in bonding studies is a viable resource for increasing sample sizes when there are difficulties in collecting teeth.

To rule out any possible variable error, maxillary and mandibular arches were analyzed for bond strength differences. There were no statistically significant differences between the two groups (p=0.28).

Race and gender were also analyzed for bond strength differences. When shear bond strength values for race were examined, no significant differences were found among the race groups (p=0.10), although Asians tended to have higher bond strengths than African-Americans who tended to have higher bond strengths than Caucasians. Unfortunately, there is no data in the literature on the bond strength differences related to race. Possible bonding differences among races should be pondered by the practitioner as new bonding materials become available. When the mean shear bond strengths were compared by gender, no significant differences were found between the groups (p=0.08). There was a trend toward males having higher bond strengths, although the results were not significantly different. Differences in the salivary mineral content among races and/or sexes and diet may have contributed to this trend. Further research may be needed to compare the worth doing to compose the enamel makeup among races and sexes.

Due to possible effects of the post-eruptive enamel maturation process, the mature and newly erupted age groups were analyzed for bond strength differences. No significant differences (p=0.38) were found between the groups. Results of the Weibull analyses also agree with this. When using a two -parameter analysis at 95% confidence levels there were no significant differences between the mature and newly erupted

groups. Both sets of data also had ρ values close to one indicating a highly linear fit. The slope values or β were 3.87 for the mature data and 2.33 for the newly erupted data. The greater the slope value, the smaller the dispersion in the strength values, which indicated greater reliability in the bond/alignment/and other variables in the experiment. This finding agrees with previous work which also found no differences in bond strengths between newly erupted and unerupted teeth.[17] However, another study reported that more retentive surfaces were obtained when conventionally etching newly erupted teeth for 15s and mature teeth for 60s.[16] That study was done using a subjective measurement of etching patterns when viewed under SEM. From the results of the current study, it appears that the post-eruptive enamel maturation process may have little effect on bond strength values when etched either conventionally or with 3M Unitek self-etching primer.

The two etch groups were also compared for differences between the two products. No significant differences (p=0.08) were found between the groups; however, the self-etching primer groups had a trend toward having lower bond strengths. Results of the Weibull analysis also agree with this. When using a two-parameter analysis at 95% confidence levels there were no significant differences between the SEP and conventional etching groups. Both sets of data also had ρ values close to one indicating a highly linear fit. The slope values or β were 1.96 for the SEP group and 2.08 for the conventionally etched group. The slope values, showed a small dispersion in the strength values, which indicated good reliability in the bond/alignment/and other variables in the experiment.

This finding agrees with previous studies that have reported lower bond strength values with self-etching primers. Nevertheless, these values were within a clinically acceptable range.[12] This is somewhat controversial as Buyukyilmaz [13] reported higher bond strengths with the 3M self-etching primer. The findings from the current study do agree with Dorminey[14] who found no difference between conventional etching and the 3M self-etching primer when used as per the manufacturers instructions. Therefore, the 3M self-etching primer appears to produce differences in bond strengths when used correctly according to the manufacturers' instructions.

When the effects of the two etch techniques on the mature and newly erupted teeth were compared, no significant differences were found (p=0.23). Based on these results, one may conclude that the bond strength produced using the 3M Unitek self-etching primer does not seem to be affected by the post-eruptive enamel maturation process. This finding is promising in the light of the increasing numbers of adult patients seeking orthodontic treatment.

Differences in the amount of adhesive remaining on the enamel surfaces following debonding were not significant except when mature and newly erupted teeth groups were compared. These results indicate that the mode of bond failure on mature teeth is usually cohesive (within the resin); whereas, the bond failure mode on newly erupted teeth is adhesive (at the enamel resin interface). These results may suggest the presence of some differences between the age groups.

Results of the Adhesive Remnant Index (ARI) scores comparing buccal and lingual surface groups, maxillary and mandibular arch groups, etch group, race groups, and male and female groups revealed no significant differences in the amount of adhesive remaining on the tooth surface. The only finding from previous literature regarding ARI scores and self-etching primers was regarding differences in etch groups. Bishara[12] found that the self-etching primer group left more adhesive on the teeth. Other studies claimed that self-etching primer groups left less adhesive on the teeth in the SEP group versus those in the conventional group.[22] Other studies found variation in ARI scores among other brands of self-etching primers.[13] The conflicting results are actually due to the fact that ARI scoring is a highly subjective analysis unless standardized methods of evaluation are used.

In the literature, it has been stated that it is desirable to have resin remnants on the tooth surface after bracket debonding to prevent enamel crazing or fracturing.[10] Adhesive removal after debonding from teeth surfaces can be a difficult and time consuming process which may cause enamel damage. The ideal adhesive should exhibit bond strengths strong enough to withstand orthodontic forces and abuse from the patient’s oral environment, yet provide easy removal after treatment without causing enamel damage. The ARI scores in this study indicated that newly erupted teeth had less resin remaining on the enamel following debonding.

Scanning electron microscopy studies of the self-etched enamel of mature and newly erupted teeth revealed similar results of enamel surfaces without a distinct dissolution pattern. Enamel surfaces etched with SEP were smoother than enamel surfaces etched with phosphoric acid. The inclusions observed on the self-etched enamel surfaces are thought to be filler particles of the resin. These particles may also be calcium and other minerals that recrystallized after enamel dissolution which subsequently became embedded in the primer matrix. Teeth surfaces were prepared according to the manufacturers' instructions for bonding. Therefore, smooth resin surfaces are expected as self-etching primer products work by simultaneously etching and wetting the tooth surfaces. Self-etched surfaces had a characteristic smooth appearance as the tubules were wetted with the primer. Conventionally etched mature and newly erupted enamel surfaces had a rough appearance with the micropores ranging from 5 to10 microns in size as expected from previous SEM studies. The etching pattern appeared to be Type 1 or 2 in nature depending on the sample. Newly etched teeth had pores which appeared larger.

Since Buonocore's introduction of direct bonding in the 1950's to the advent of self-etching primers, the pursuit of quick and efficient bonding is becoming more of a reality. Self-etching primers appear to exhibit acceptable values of effective bond strengths for orthodontics and allow for easy debonding while saving time during bonding procedures. This study sought to determine if one self-etching primer product had the same efficacy on mature and newly erupted teeth. It was found that the self-

etching primer used in this study had similar bonding properties to conventional etching and can be reliably used for bonding orthodontic attachments to both young and adult patients' teeth.

Conclusions

The important findings of the present study can be summarized as follows:

- Self-etching primer and conventional etching techniques do not differ in the bond strengths they produce when comparing them on mature and newly erupted enamel.
- ARI scores differ between mature and newly erupted teeth in that mature teeth have more cohesive bond failures and newly erupted teeth have more adhesive failures at the enamel-composite junction.
- Scanning electron micrographs of the enamel prepared with self-etching primer reveal smooth surfaces with resin covering the tubules. Resin filler particles were also seen occasionally. Conventionally etched enamel surfaces had a rough appearance with the micropores ranging from 5 to10 microns in size with newly etched enamel appearing to have larger pore size.

As orthodontic bonding techniques continue to advance and become more efficient, their effects on enamel of various ages due to the post-eruptive maturation process must be taken into account. The self-etching primer techniques appear to be a viable alternative to conventional etching even with a slightly lower, yet clinically acceptable, bond strength. As more adults seek orthodontic treatment, it is important to be mindful of their mature enamel with higher calcified content which may lead to different bonding

properties. Further research needs to be done in order to examine the effect of various bonding techniques on enamel with different inorganic compositions.

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VITA

Diana M. Almy was born in Fredericksburg, Virginia in 1976. She attended public school in Stafford County, Virginia and graduated in 1994 from Stafford Senior High School. She received a Bachelor of Science from Randolph-Macon College in 1998 with a double major in Biology and Chemistry. She proceeded to Virginia Commonwealth University School of Dentistry and graduated in 2002 with a Doctor of Dental Surgery degree. She is currently a postgraduate resident in the Orthodontics program at Virginia Commonwealth University and will receive a certificate in Orthodontics and a Master of Science degree. Upon graduation, Diana will start a private practice in her hometown of Fredericksburg, Virginia. She is married with one son.

APPENDIX A (Raw Data)

Tooth Number	Surface	Tooth Age	Etch	Pt Age	Race	Gender	Arch	Force (lbs)	Force (PSI)	ARI
1	Buccal	Old	Self	41	Asian	Male	Max	49.8	**20.5**	**EF**
2	Buccal	Old	Self	41	Asian	Male	Mand	30	**12.4**	**2**
3	Buccal	Old	Self	41	Asian	Male	Max	45.7	**18.9**	**4**
4	Buccal	Old	Self	41	Asian	Male	Mand	42.3	**17.5**	**EF**
5	Buccal	Old	Self	23	Asian	Male	Max	34.5	**14.2**	**3**
6	Buccal	Old	Self	23	Asian	Male	Mand	28.7	**11.8**	**3**
7	Buccal	Old	Self	23	Asian	Male	Max	37	**15.3**	**4**
8	Buccal	Old	Self	23	Asian	Male	Mand	16	**6.6**	**3**
9	Lingual	Old	Self	35	White	Male	Max	41.1	**17.0**	**4**
10	Lingual	Old	Self	35	White	Male	Mand	5.5	**2.3**	**4**
11	Lingual	Old	Self	35	White	Male	Max	31.3	**12.9**	**3**
12	Lingual	Old	Self	35	White	Male	Mand	16.9	**7.0**	**3**
13	Lingual	Old	Self	25	White	Female	Max	23	**9.5**	**4**
14	Lingual	Old	Self	25	White	Female	Max	41.8	**17.2**	**4**
15	Lingual	Old	Self	35	White	Female	Max	13.9	**5.7**	**3**
16	Lingual	Old	Self	35	White	Female	Max	13.9	**5.7**	**3**
								Mean	***12.2***	***3.4***
								STD	***5.3***	***0.6***
9	Buccal	Old	Conventional	35	White	Male	Max	54	**22.3**	**4**
10	Buccal	Old	Conventional	35	White	Male	Mand	23.8	**9.8**	**3**
11	Buccal	Old	Conventional	35	White	Male	Max	53.2	**22.0**	**3**
12	Buccal	Old	Conventional	35	White	Male	Mand	16.3	**6.7**	**2**
13	Buccal	Old	Conventional	25	White	Female	Max	19.3	**8.0**	**4**
14	Buccal	Old	Conventional	25	White	Female	Max	31.8	**13.1**	**4**
15	Buccal	Old	Conventional	35	White	Female	Max	22	**9.1**	**3**
16	Buccal	Old	Conventional	35	White	Female	Max	29.1	**12.0**	**3**
1	Lingual	Old	Conventional	41	Asian	Male	Max	31.7	**13.1**	**3**
2	Lingual	Old	Conventional	41	Asian	Male	Mand	24.5	**10.1**	**3**
3	Lingual	Old	Conventional	41	Asian	Male	Max	37.3	**15.4**	**3**
4	Lingual	Old	Conventional	41	Asian	Male	Mand	45.7	**18.9**	**EF**
5	Lingual	Old	Conventional	23	Asian	Male	Max	36.7	**15.1**	**4**
6	Lingual	Old	Conventional	23	Asian	Male	Mand	27.4	**11.3**	**5**
7	Lingual	Old	Conventional	23	Asian	Male	Max	31	**12.8**	**4**
8	Lingual	Old	Conventional	23	Asian	Male	Mand	44.5	**18.4**	**EF**
								Mean	***13.6***	***3.4***
								STD	***4.6***	***0.8***

Tooth Number	Surface	Tooth Age	Etch	Pt Age	Race	Gender	Arch	Force (lbs)	Force (PSI)	ARI
17	Buccal	Young	Self	14	White	Female	Mand	7.9	**3.3**	**5**
18	Buccal	Young	Self	14	White	Female	Max	18.3	**7.6**	**5**
19	Buccal	Young	Self	14	White	Female	Mand	42.2	**17.4**	**4**
20	Buccal	Young	Self	14	White	Female	Max	28.5	**11.8**	**4**
21	Buccal	Young	Self	14	White	Male	Max	17.2	**7.1**	**5**
22	Buccal	Young	Self	14	White	Male	Mand	30.8	**12.7**	**3**
23	Buccal	Young	Self	14	White	Male	Max	6.2	**2.6**	**5**
24	Buccal	Young	Self	14	White	Male	Mand	35.5	**14.6**	**4**
25	Lingual	Young	Self	13	Black	Male	Mand	33	**13.6**	**5**
26	Lingual	Young	Self	13	Black	Male	Max	21.3	**8.8**	**5**
27	Lingual	Young	Self	13	Black	Male	Max	20.2	**8.3**	**5**
28	Lingual	Young	Self	13	Black	Male	Mand	7.5	**3.1**	**5**
29	Lingual	Young	Self	13	White	Female	Max	7.4	**3.1**	**5**
30	Lingual	Young	Self	13	White	Female	Max	28.1	**11.6**	**EF**
31	Lingual	Young	Self	10	Black	Female	Max	40.2	**16.6**	**4**
32	Lingual	Young	Self	10	Black	Female	Max	34.2	**14.1**	**4**
								Mean	***9.8***	***4.5***
								STD	***4.9***	***0.6***
25	Buccal	Young	Conventional	13	Black	Male	Mand	67.5	**27.9**	**5**
26	Buccal	Young	Conventional	13	Black	Male	Max	60.8	**25.1**	**3**
27	Buccal	Young	Conventional	13	Black	Male	Max	53.8	**22.2**	**5**
28	Buccal	Young	Conventional	13	Black	Male	Mand	5	**2.1**	**4**
29	Buccal	Young	Conventional	13	White	Female	Max	32.9	**13.6**	**5**
30	Buccal	Young	Conventional	13	White	Female	Max	35	**14.4**	**3**
31	Buccal	Young	Conventional	10	Black	Female	Max	16.2	**6.7**	**3**
32	Buccal	Young	Conventional	10	Black	Female	Max	25.8	**10.6**	**5**
17	Lingual	Young	Conventional	14	White	Female	Mand	28	**11.6**	**5**
18	Lingual	Young	Conventional	14	White	Female	Max	9.3	**3.8**	**EF**
19	Lingual	Young	Conventional	14	White	Female	Mand	41	**16.9**	**3**
20	Lingual	Young	Conventional	14	White	Female	Max	23.8	**9.8**	**2**
21	Lingual	Young	Conventional	14	White	Male	Max	44.7	**18.4**	**5**
22	Lingual	Young	Conventional	14	White	Male	Mand	23.3	**9.6**	**4**
23	Lingual	Young	Conventional	14	White	Male	Max	45.6	**18.8**	**EF**
24	Lingual	Young	Conventional	14	White	Male	Mand	7.7	**3.2**	**4**
								Mean	***13.4***	***4.0***
								STD	***7.5***	***1.0***