The Effects on Microtensile Bond Strength in the Absence of Oxygen-Inhibited Layer and method of Etching.

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ABSTRACT

Objective: Dental adhesives are relied upon to provide a strong and durable bond that will last, while also resisting dissociating forces, such as those experienced during polymerization shrinkage of composite resin restorations. The aim of this study is to determine the effects of anaerobic photo-curing of dental adhesives, by eliminating the oxygen-inhibited layer, on their mechanical properties; specifically, microtensile bond strength (µTBS).

Materials and Methods: Forty permanent molars were divided among eight groups determined by a combination of one of two universal adhesive systems (Prime and Bone Elect, Scotchbond Universal), etching type (self-etch, total-etch), and curing conditions (aerobic, anaerobic). All bonded specimens were incubated in distilled water, half of which were stored for one month, and half for one year. Each specimen was cut into 1mm x 1mm in cross-section sticks, that were then used to test the µTBS of the adhesive. The specimens were subsequently evaluated under a light microscope to determine the location and type of failure.

Results: Etching technique and curing conditions had a greater effect, while adhesive type has less influence, on the µTBS of the adhesive systems. Total-etch resulted in better bond strength under anerobic conditions, but performance decreased after one year. Prime Bond Elect presented the highest mean when used in aerobic curing technique associated with total etch (40.17MPa). The lowest value was observed with Scotchbond (SB) under anaerobic conditions and self-etching pre-treatment (11.58MPa).

Conclusion: Within the limits of this study we can conclude that etching system and curing condition require further evaluation according to the adhesive system used in order to get the best adhesion results. As it stands, bond strength tends to be comparable, or better, under aerobic conditions. But improvements for better control of an anerobic environment could provide more accurate answers.

Clinical significance: The evolution of universal adhesive represents the effort of manufacturers to facilitate the clinical procedures of adherence, but through these results, the choice of pre-treatments as well the curing process should be better evaluated

INTRODUCTION

Dental adhesives play a crucial role in the performance of a restoration. It is the direct interface between the dental restoration and the tooth which allows for retention of the restoration. These adhesives are thus under frequent forces and stresses, such as polymerization shrinkage, shear, and tensile forces. An important component of the dental joint, dental adhesives are constantly being re-innovated to allow for easier application and increased durability and longevity. With advances of adhesion technology that allows for universal application as either self-etch (SE) or total-etch (TE), there is an ongoing demand to determine the best mode of application[1,2,3]

One factor that is often overlooked is the formation of the oxygen-inhibited layer (OIL) which forms during curing on the adhesive surface exposed to air[4]. This layer is often sticky to the touch and non-uniform, this is due to the low degree of conversion (DC) caused by the presence of oxygen[4]. Generally, it is believed that the formation of OIL is desired, as its formation allows for a chemical bond to form between itself and the resin restoration. Thus, it results in adequate adhesive and restoration performance[5,6].

However, when tension forces are applied, it was demonstrated that the restoration often fails at the adhesive/restoration interface; where the resin restoration bonds with the OIL of the adhesive[7]. A study by Suh showed that it is possible to achieve a good bond even in the absence of oxygen, thus absence of OIL[4]. Another recent study by Phaneuf et al. proved that when oxygen is absent during curing of the dental adhesive, there is over a 2-fold increase in the DC[8]. The same study also concluded that there is no significant difference in shear bond strength (SBS) between samples that were cured anaerobically, and those cured in presence of air[8,9].

Although current evidence show that increased DC is the only benefit of curing the dental adhesive anaerobically, it begs the question of its effect on the microtensile bond strength (µTBS) of the adhesive system. Since the failure point is often associated with the adhesive/restoration interface, ensuring high DC and better adhesive/resin restoration interaction could potentially eliminate that problem. Thus, the purpose of this study is to test the effects of anaerobic curing of two common universal dental adhesives, Scotchbond Universal (SB) and Prime&Bond Elect Universal (PB) on their µTBS performance. This study will also test the effects of the mode of application (SE vs. TE) on the µTBS under both aerobic and anaerobic conditions. The effect of anaerobic curing technique will be tested both short term (T= 1 month) and long term (T= 1 year). The Hypotheses are:

- Null hypothesis
    - No difference in microtensile bond strength between conventional and anaerobic curing techniques regardless of application mode.
- Alternative hypothesis 1
    - A significant difference in microtensile bond strength exists that is due to the method of application; self-etch vs. etch-and-rinse.
- Alternative hypothesis 2

- A significant difference in microtensile bond strength exists that is due to the presence or absence of an oxygen-inhibited layer.

MATERIALS AND METHODS

Tooth selection, mounting, and preparation

80 human permanent molars without prior restorations were selected[10]. Each tooth had the occlusal 1/3 of the crown removed and a flat surface perpendicular to its long axis was prepared with water-cooled Isomet low speed saw (Buehler, Lake Bluff, USA)[7]. The exposed mid-coronal dentin was wet-polished with 600 grit SiC paper to create a standardized surface texture and smear layer for all teeth before beginning the bonding procedure[7].

Bonding Procedure

<u>For groups testing aerobic conditions</u>

Caulk 34% Tooth Conditioner Gel (DENTSPLY, Germany) etchant was applied as per manufacturer's instructions (For groups using TE technique). Remaining water was removed by gentle blow of oil-free air. The adhesive systems were applied as per their respective manufacturer's instructions and light cured with Valo Cordless LED curing light (UP Dental GmbH, Koln-Porz, Germany) for 20 seconds according to an ongoing study by Rosenthal et al[11]. A 0.5mm layer of SureFil® SDR® flow+ (DENTSPLY, Germany) flowable was applied overtop and light cured for 20 seconds as per manufacturer's instructions. Two increments of 2mm each of TPH Spectra High viscosity composite resin (DENTSPLY, Germany) were used for the bulk of the restoration for a total of 4mm. Light curing for (20 seconds, as per manufacturer's instructions) between application of the successive increment.

<u>For groups testing anaerobic conditions</u>

34% Tooth Conditioner etchant was applied as per manufacturer's instructions (For groups using TE technique). Remaining water was removed by gentle blow of air. The adhesive systems were applied as per their respective manufacturer's instructions but were *not* cured. A 0.5mm layer of flowable was applied overtop of the non-cured adhesives. The adhesive and flowable were light cured together for 20 seconds. Two increments of 2mm each of composite resin were used for the bulk of the restoration for a total of 4mm. Light curing for 20 seconds between application of the successive increment.

Each group had half of the samples stored in distilled water for one month, while the remaining half were stored in distilled water for one year. Teeth were stored at 37C with the water replaced every two weeks until the µTBS testing

Microtensile Bond Strength testing

Each tooth was cut along the vertical axis to 1mm thick slices using a diamond saw at 100rpm with 75grams of force. Each slab was further cut into 1mm thick slices, yielding 1mm x 1mm sticks. Each stick was mounted onto the jaws of a Micro Tensile Tester (BISCO, Schaumburg, U.S.A) and secured with ZAPIT glue (BISCO, Schaumburg, U.S.A), see Figure 1. A cross head loading rate of 0.5mm/min was set and the µTBS tested[7,10]. Results were recorded as Newtons (N), and later converted to MPa using the cross-section of the sample following the formula:

$$\mu TBS\ (MPa) = \frac{Force\ (N)}{Cross - section\ (mm^2)}$$

Statistical Analysis:

The results of each group were evaluated for normal distribution to determine type of analysis that best suits the purpose. A Kruskal-Wallis test was performed on both the one-month and one-year samples. Tukey's Post-hoc tests were used to evaluate significant differences between the groups at the 0.05 level.

RESULTS

1 Month results

Mean µTBS for each group of the 1-month incubation time can be found in Table 1. Highest microtensile bond strength was demonstrated by Prime&Bond under aerobic conditions and total-etching technique (39.6MPa), while the weakest bonds were found to be by both Prime&Bond and Scotchbond under anaerobic conditions and self-etching technique (11.4MPa and 11.9MPa respectively). As some groups' results did not follow a conventional normal distribution, a Kruskal-Wallis test was used to analyze the results (see Figure 2).

Tukey's Post-hoc test shows that under anerobic conditions, TE technique results in significantly better µTBS for both universal adhesives at an $\alpha = 0.05$ confidence level. However, under aerobic conditions, significant difference in etching technique was only evident for Scotchbond, with SE yielding better bond strength (31.7MPa); Prime&Bond did not have a significant difference between etching-techniques (39.6MPa TE vs. 34.3MPa SE).

For all combinations of adhesives and etching methods, except for Scotchbond with total-etching technique, higher µTBS was observed under aerobic conditions when compared to anerobic. No statistically significant difference was observed in bond strength for Scotchbond when used in a total-etching technique in both aerobic and anerobic environments. It was also demonstrated that a significant difference between the adhesive systems used was only evident when used with total-etching technique in a conventional aerobic environment (39.6MPa PB vs 18.8MPa SB).

1 Year results

Table 2 shows the mean µTBS for all samples incubated for one year in distilled water. Best performance in bond strength was found to be by Scotchbond when used in self-etching technique and an aerobic environment (34.8MPa). The same adhesive and etching technique, but under anaerobic environment failed to provide intact samples for µTBS testing. As no readings were achieved, no value could be placed for this group. Normal distribution of results was observed only in some of the groups, and hence a Kruskal-Wallis test was performed (Figure 3).

Evaluating the µTBS results between the groups using Tukey's Post-hoc test showed that Scotchbond has better bond strength under aerobic conditions when using SE technique (34.8MPa). Etching technique had no significance for Scotchbond when used in an anerobic environment. However, TE was found to perform better under anerobic conditions for the Prime&Bond adhesive system (24.4MPa TE vs 4.6MPa SE).

When looking at how the curing environment had affected the µTBS of the universal adhesives, it was found that the greatest performance of both adhesives systems was under aerobic conditions a SE technique (34.8MPa SB and 30.6MPa PB). Although Prime&Bond did yield a greater mean µTBS in aerobic environment with TE technique when compared to the SE technique (34.0MPa TE vs 30.6MPa), it was not significantly different than when using TE technique in anaerobic environment (20.8MPa Anerobic + TE).

The type of adhesive system used had no statistical significance in an anerobic curing environment regardless of the etching technique used. When looking at a conventional aerobic curing of the adhesives, the adhesive system used mattered only when used with TE technique, where Prime&Bond resulted in significantly greater µTBS (34.0MPa PB vs. 17.8MPa SB). Both adhesives systems performed similarly as well in an aerobic environment when used with the SE technique (30.6MPa PB vs 34.8MPa SB).

DISCUSSION

The purpose of this study was to evaluate the effects of various combinations of etching techniques with curing conditions on the µTBS of universal dental adhesives. As it was shown by on existing pending studies that curing of dental adhesives under anerobic conditions lead to higher degree of conversion during polymerization, it begged the question on how that influenced the overall performance of the bond strength[8]. Adding etching technique as a factor could allow for identification of the sequence of steps of adhesive placement that will provide the optimal bond strength.

Curing environment

As the main factor at question here, curing environment is what determines the degree of conversion during polymerization of the adhesive, and subsequently whether an oxygen-inhibited layer (OIL) will form. The results indicate that initially (after 1-month incubation) the µTBS tended to be higher when cured under aerobic conditions. Specifically, for the two universal adhesive systems, SE technique tended to perform better in aerobic environment than anerobic. TE technique had yielded better results under aerobic conditions only for Prime&Bond adhesive system but had no significant difference for Scotchbond. This indicates that the anerobic environment had little influence on the bond strength of Scotchbond when using TE technique.

The samples stored for one year exhibited consistently better performance for the aerobic group vs anerobic. Specifically, when looking at Scotchbond, whose samples treated with SE technique and cured anaerobically failed to provide intact specimens for µTBS testing. These specimens all failed at the adhesive interface during specimen preparation, which indicated the significant loss of bond strength after one year. Prime&Bond had demonstrated a consistent bond strength after one year of storage when using TE technique. Both Scotchbond and Prime&Bond had significantly better results under aerobic curing conditions when using SE technique.

Results thus indicate that aerobic curing tends to provide better µTBS of the adhesives, especially when using SE technique. Although for the most part anaerobic curing showed decreased bond strength, TE technique appeared to be contributing to the longevity of the bond strength over time. Thus, a combination of anaerobic curing and SE is not recommended. It is hypothesized that the lack of OIL formation prevents adequate bonding of the adhesive to the restoration, resulting in decreased strength.

Etching Technique

Etching technique is an important factor to consider in restoration to ensure proper penetration and formation of the hybrid layer to aid in the bond strength of the universal adhesives. When looking at 1-month samples in aerobic environment only, samples treated with Scotchbond performed significantly better with SE technique. However, Prime&Bond had no difference between SE and TE and performed just as well with either etching technique. This was also true for the 1-year samples.

The differences in µTBS appeared more readily when looking anerobic bonding only. The 1-month samples show that both Scotchbond and Prime&Bond yielded better bond strength with TE technique. Utilizing SE in anaerobic conditions proved to result in significantly decreased bond strength. Under anerobic conditions, the bond strength worsened in the 1-year samples treated with Scotchbond, thus no statistically meaningful difference was found between the TE and SE. This was not the case with Prime&Bond, where TE technique in anerobic environment still produced better µTBS over SE.

These results indicate that under aerobic conditions, either etching technique is adequate for adhesive strength, except for Scotchbond, where SE yields better results over time. However, etching technique plays a crucial role in anerobic curing, where TE is preferred over SE as it provides better long-term outcome. In either etching technique though, bond strength decreases with time, and may ultimately lead to premature failure. Thus, a correct combination of adhesive system and etching technique is important for anerobic curing.

Adhesive system

From all three factors tested, the type of adhesive system used mattered the least. Both adhesive systems performed in a similar manner in both aerobic and anerobic conditions when utilizing the SE technique. The 1-year samples have demonstrated no significant difference between the adhesives either with the SE technique; both had adequate longevity of bond strength under aerobic conditions, and both had decreased bond strength under anerobic conditions.

Likewise, both adhesive systems showed similar properties under anaerobic conditions when using TE technique for both the 1-month and 1-year samples. The difference lies in the use of TE in the conventional aerobic curing of the adhesives. Prime&Bond consistently yield greater µTBS over Scotchbond in both the 1-month and 1-year samples.

Further investigation is warranted to more accurately evaluate the performance of each adhesive, but as it stands now, both adhesives behave in a similar manner under the various conditions provided, and thus the adhesive system used is of little significance, especially when using under aerobic conditions.

Given the results and their statistical analysis, it is determined that the null hypothesis is rejected, and alternative hypotheses 2 is accepted. Alternative hypothesis 1 requires further investigation in a better controlled environment to evaluate its validity, and association with curing conditions.

CONCLUSION

Through this study, it was demonstrated the µTBS of universal adhesives is greatly influenced by the etching technique as well as the environment within which the adhesive is cured. Greatest µTBS were achieved under aerobic conditions which remained stable even after one-year incubation. Anerobic curing resulted in decreased bond strength which further weakened over time. This field requires further investigation of anerobic curing as conditions and elements to aid in achieving a controlled anerobic environment could be improved. Regulating thus the degree of conversion and OIL formation could help identify optimal conditions for maximum bond strength.

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APPENDIX

Table 1: Mean µTBS for sample of each group incubated for one month in distilled water.

	Aerobic				Anerobic			
	Scotchbond		Prime&Bond		Scotchbond		Prime&Bond	
	SE	TE	SE	TE	SE	TE	SE	TE
Mean µTBS (MPa)	31.7	18.8	34.3	39.6	11.9	22.3	11.4	20.8

Table 2: Mean µTBS for sample of each group incubated for one year in distilled water.

	Aerobic				Anerobic			
	Scotchbond		Prime&Bond		Scotchbond		Prime&Bond	
	SE	TE	SE	TE	SE	TE	SE	TE
Mean µTBS (MPa)	34.8	17.8	30.6	34.0	N/A*	15.3	4.6	24.4

***** No sticks were recovered during sample preparation as all failed at the adhesive interface during cutting.

Figure 1: Mounting of specimen on the jaws of a BISCO Micro Tensile Tester and secured with BISCO ZAPIT glue.

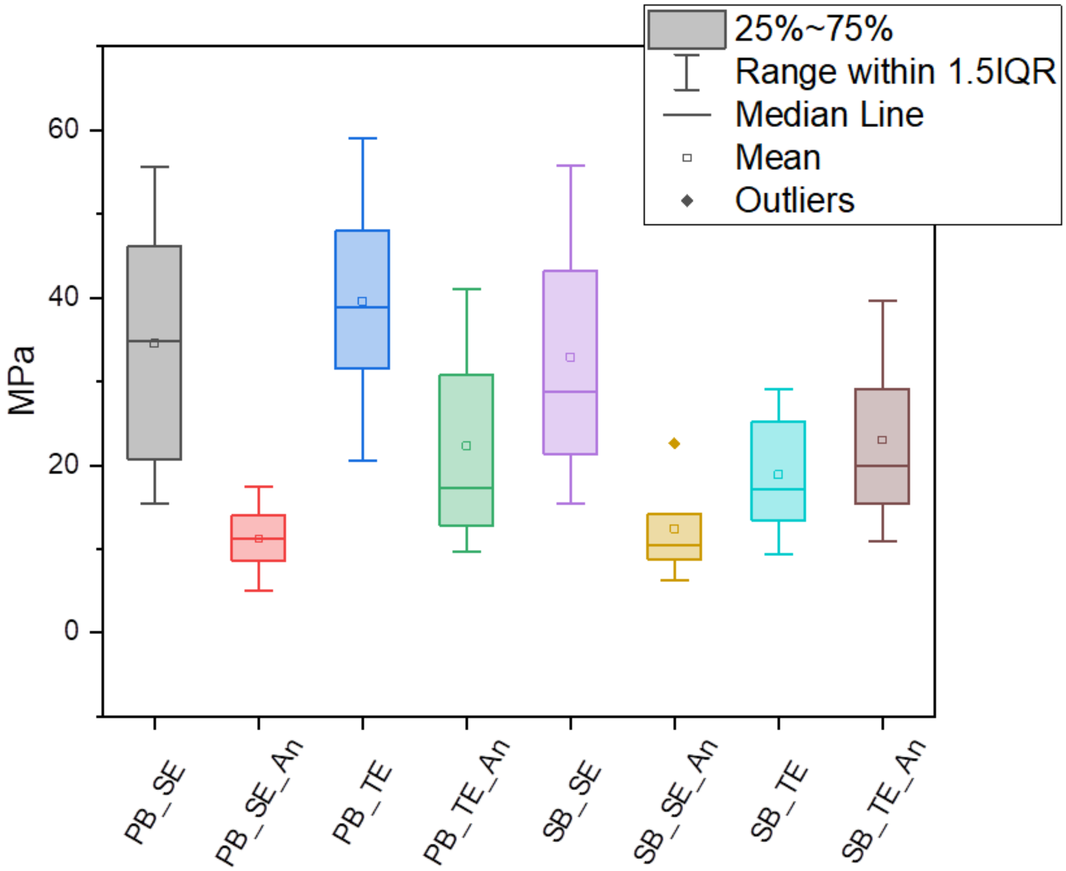

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Figure 2: µTBS of two universal adhesive systems (PB and SB) comparing curing conditions (aerobic and anaerobic) and etching technique (SE and TE) when incubated for 1 month in distilled water.

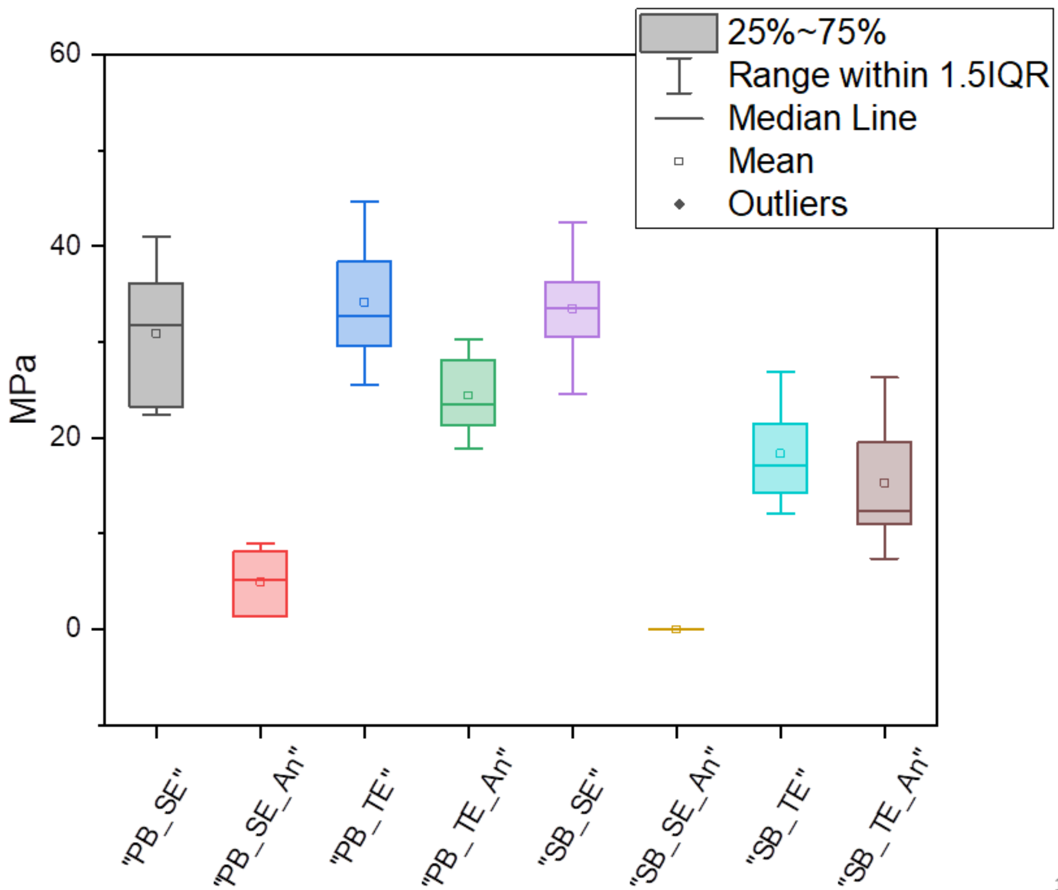

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Figure 3: μTBS of two universal adhesive systems (PB and SB) comparing curing conditions (aerobic and anaerobic) and etching technique (SE and TE) when incubated for 1 year in distilled water.