Original Article

Tensile bond strength: Evaluation of four current adhesive systems in abraded enamel and deep dentin

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This study aimed to evaluate the tensile bond strength of adhesive systems in abraded enamel and deep dentin of the occlusal surface of forty human molar teeth. Enamel surfaces as well as the rest of the teeth were coated with epoxy resin and regularized and polished with silicon carbide sandpapers. The 40 teeth were randomized into eight groups of five teeth per group. Four groups were assigned to have deep dentin as the dental substrate and the other four had abraded enamel as the substrate for the adhesives to be tested. The adhesives being tested were the total etching Single Bond: SB, the self-etching Clearfil SE bond: CSEB, self-etching One Up Bond F: OUBF and the self-etching Self-Etch Bond: SEB adhesives. The samples (teeth) were restored with composite resin and subjected to a traction assay. The results were statistically analyzed using the ANOVA and TUKEY tests. The total etching SB adhesive system had the greatest bonding strength of all the adhesives tested, on both dental substrates (20.1 MegaPascals (MPa) on abraded enamel and 19.4 MPa on deep dentin). Of the self-etching dental adhesives tested, CSEB had the greatest bonding strength on both substrates (14.6 MPa on abraded enamel and 15.4 MPa on deep dentin). Both OUBF (11.0 MPa for enamel, 13.1 MPa for dentin) and SEB (10.2 MPa for enamel, 12.6 MPa for dentin) showed comparable bonding strengths without any significant differences for either substrate Thus, the total etching SB adhesive system had better bonding strength than the other self-etching adhesives used, regardless of the dental substrate to which the adhesives had been bonded.

Key words: Dental enamel, dentine, tensile strength

Dental adhesives were developed in order to provide a strong bonding interface between the tooth substrate and the restorative material being used in restorative dentistry.[1] Sometimes, the interface fails to bond the tooth and the restorative material if the two possess different mechanical properties.

Dentin has been described as a dynamic heterogeneous substance which is an unfavorable substrate for bonding.[2,3] On the other hand, enamel is an easier substrate for adhesive systems. The quality of the dentin-adhesive bond may be affected by the degree of demineralization and of impregnation of resinous monomers into the demineralized region. When the depth of demineralization exceeds the depth of monomer diffusion into the dentine, some collagen fibrils in the dentine layer remain unhydrolyzed. Thus, a zone of unstable collagen is set which becomes susceptible to hydrolysis.[4-6]

Bond strength is an important feature, which may show the ability of the adhesive system to provide a strong adhesive interface which offers biological protection against microleakage in the tooth / restoring interface. Several authors have described the formation, quality and morphology of the resin / dental substrate.[7] The formation of this hybrid layer is still a controversial mechanism although the importance of the adhesive interface in providing biological protection has been shown.[8,9]

Acid monomers of self-etch adhesive systems have been one of the main benefits of adhesive dentistry. Unlike total-etching adhesives, self-etching adhesives do not require the removal of the smear layer, thus, infiltrating the demineralized dentin completely to form a hybrid layer which includes the dissolved smear layer. Due to the retention of the smear layer, self-etching adhesive systems involve fewer steps and are simpler to use.[2,10–14]

Dental adhesives display different bonding strengths on different substrates and in different conditions on the same substrate. This *in vitro* study evaluated the

tensile bond strengths of a total-etching adhesive system and three self-etching adhesive systems on enamel and deep dentine substrates. The null hypothesis tested was that there is no difference in the bonding of self-etching and total-etching adhesives to either enamel or dentine.

MATERIALS AND METHODS

40 third molar teeth used in this *in vitro* study were obtained from young patients who needed to extract them as part of their dental treatment. The teeth were cleaned of debris and kept in saline solution at 5ºC until their use in this study. In order to evaluate tensile bond strength (TBS), the buccal surfaces were sectioned obliquely and the occluded surfaces were sectioned transversally at the half coronal with a low-speed diamond saw Isomet 1000 (Buchler, Lake Bluff, IL, USA) [Figure 1].

The enamel buccal surface fragment and the rest of the dentine were included in epoch resin after being randomized into four groups [Table 1].

The exposed occluded dentine and the buccal surface of enamel were polished with 600-grit carbide paper for 20 seconds in order to create a standardized smear layer in the dentine and an abraded surface on the enamel.

After polishing, all the included fragments were delimited by placing a piece of adhesive paper with holes of 4 mm diameter. The dental adhesives were bonded to the substrates according to the manufacturers' instructions. Light intensity of an Ultralux (Dabi Atlante Industrias Medico-Odontologicas, Ribeirao Preto, SP, Brazil) was measured periodically with a Gnatus Radiometer (Gnatus Industria e Comercio de Equipamentos Medico-Odontologicos, Ribeirao Preto - SP, Brazil) and it ranged from 450-500 milli Watts (mW)/cm^2.

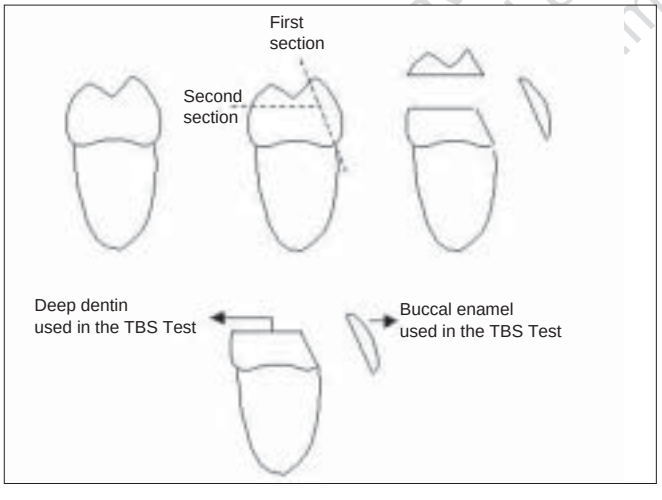

Figure 1: Section of the buccal enamel and deep dentine

A 4 mm diameter conical cylinder and metallic matrix were installed at the top of the specimen to receive a composite resin restoration in four oblique increments, polymerized for 20 seconds each, with Filtek Z 250 composite resin (3M ESPE, St. Paul MN, USA, batch number 2NE) in order to exclude the influence of different restorative resins on the test. The specimens were kept in distilled water at room temperature and after 24 hours, they were submitted to the tensile bond strength test in an MTS 810 machine (Material Test System, Edemprairie, MN, USA) at a speed of 0.5 mm/min until the specimens fractured. The tensile bond strength of each specimen was registered in MPa and the results were submitted to ANOVA and TUKEY tests at 5% level of significance.

RESULTS

ANOVA did not show any significant interaction between adhesive systems and dental substrates but did indicate that while the performance of a dentin bonding agent (DBA) did not vary with the substrate, there was a difference in the bonding of the various DBAs to a specific substrate [Table 2].

The means in Table 2 which are followed by the same letter (subscript letters down and subscript letters across) did not show any significant difference in Tukey's test (0.05%).

The total-etching SB adhesive system showed the greatest tensile bond strength to both substrates (dentin and enamel) of all the systems tested.

Among the self-etching DBAs, CSEB had the greatest TBS while OUBF and SEB had comparable TBS with slightly higher though not significantly different TBS on dentin compared to enamel.

DISCUSSION

Since the introduction of acid etching by Buonocoke[15] and the reporting of the mechanism of adhesion to dentin by Nakabayashi,[16] the main objective of the adhesive process is to get a good infiltration of resin monomers into the hard acid-etched tissues.

The surface enamel is mineral-rich and adhesion to

Table 1: Bonding agent used in the experimental groups

Single bond	Clearfil SE bond	One up bond F	Self-etch bond
(3M ESPE-St. Paul, MN, USA)	(Kuraray Co. Ltd. Osaka, Japan)	(Tokuyama Corp, Tokyo, Japan)	(Vigodent S/A, Rio de Janeiro, Brazil)
Batch number:1FH	Batch number:51205	Batch number: 000231E	Batch number: 001/03

Table 2: Means of tensile bond strength (in MPa) measured for four dentin bonding agents and two dental substrates

Adhesive systems	Substrates	
	Abraded enamel	Deep dentine
Single bond	20.1_{a}^{a}	19.4_{a}^{a}
Clearfil SE bond	14.6_{b}^{a}	15.4_{b}^{a}
One up bond F	11.0_{c}^{a}	13.1_{bc}^{a}
Self-etch B	10.2_{c}^{a}	12.6_{c}^{a}

The means followed by the same letter (subscript letters in the vertical and superscript letters in the horizontal) did not show statistically significant differences by Tukey`s test (0.05%).

it requires a definite protocol. Acid-etchingpromotes the removal of the interprismatic enamel creating micromechanical retentive porosities that will then be occupied by resinous monomers.

Proper adhesion is ideally characterized by high tensile bond strength and consequently, a high resistance to microleakage from the cavity edges of the enamel and dentin. The discrepancy between the depths of demineralization and of monomer resin infiltration has led to the development of adhesive agents which do not require the removal of the smear layer by acid-etching.[9]

The problem posed by etching of dentin before application of the adhesive / primer is that due to the phosphoric acid in dentin, besides removing the smear layer, etching also demineralizes 3-5 µm of the dentin layer. This exposes the collagen fiber networks which may collapse, thus hampering the restorative process by preventing permeation of resinous monomers into the dentin.17]

The highest TBS of the total-etching SB adhesive among all the DBAs tested can be attributed to the fact that total etching removes the smear layer completely, exposing the collagen fibers as mentioned above. The resinous monomers of the SB can now permeate into the demineralized and cleaned enamel / dentin substrate. Depending on the action of the demineralizer agent and tissue composition, penetration of the resin into the demineralized substrate can occur to various degrees. Abraded enamel does not have much superficial mineral content whereas the deep dentin layer has more tubules.. The difference between deep and superficial dentin has been studied[9,10] as has the performance of several adhesive systems on enamel and dentin.[1,7,17]

CSEB presented intermediate results of TBS in both substrates, having more TBS than the other self-etching DBAs and less TBS than the total-etching SB adhesive. CSEB contains methacryloyloxydecyl dihydrogen phosphate (MDP) which acts as the acid phosphate resin monomer, the pH being around 1.9. The depth of demineralization effected by this agent is the same as the thickness of the hybrid layer (THL). Mean THL of CSEB was 3.85 µm in superficial dentin, while mean THF of OUBF that used 11-methacryloxy-1,1-undecandedicarboxylic acid (MAC-10) as the monomer agent (pH around 1.3) was 3.34 µm.[17] According to studies of shear bond strength by Toledano *et al.*,[9] the self-etching adhesive Clearfil SE Bond (CSEB) had higher shear bond strength on enamel than Scotchbond Multipurpose (SBMP), a total-etching adhesive system (SBMP = 14.8 MPa, CSEB = 19.6 MPa). When these adhesive systems were compared in superficial and deep dentine, the self-etching adhesives showed higher, statistically significant TBS values.

When the authors compared each adhesive system in superficial and deep dentine, the results were not statistically different in either substrate.

Studies in bonding strength of dental adhesives are mainly important because of the information obtained from their relative values as numerical comparisons are not always possible. Although the self-etching adhesives get into both the deeper and superficial dentin, the intrinsic features of the deep dentine promote a different pattern of demineralization and infiltration of resin monomers.[2,10,18]

Although the total-etching SB adhesive shows the highest TBS of all adhesives tested, it has a higher binding strength to enamel than to dentin. This is because of the higher mineral content of enamel as compared to dentin, which requires more effective conditioning which the process of complete removal of the smear layer by total-etching adhesives is able to accomplish. On the other hand, self-etching agents show greater TBS on dentin as compared to enamel because of the stronger infilitration into the demineralized dentin to form the hybrid layer (without complete removal of the smear layer) which is not the case with enamel. However, the generally greater bonding of total-etching DBA as compared to that of the self-etching DBAs in this study, can be explained by the fact that total-etching SB completely removes the smear layer to demineralize the dentin while self-etching DBAs don't achieve the same depth of demineralization due to the intact smear layer.

CONCLUSIONS

Total-etching adhesive systems showed greater tensile bond strength than the self-etching adhesive systems and had greater TBS on enamel than on dentin in this study. However, self-etching adhesive systems had greater TBS on dentin than on enamel. Of all the self-etching DBAs tested, CSEB had the greatest tensile strength on both enamel and dentin substrates.

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