Estimation and comparison of tensile bond strengths at resin-dentin interface of total-etch and self-etch bonding systems

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Abstract

Objective: To estimate and compare the Tensile Bond Strengths and to observe the resin-dentin interface of total-etch and self-etch bonding systems to dentin of primary and permanent molar teeth.
Method: Thirty non-carious exfoliated primary human molars and thirty extracted permanent molars were randomly assigned to four groups according to adhesives used. The specimens were subjected to tensile bond strength (TBS) testing at a crosshead speed of 1 mm/min. Mean TBS was statistically analyzed with student 't' test. Interfacial morphologies were analyzed by Scanning Electron Microscopy (SEM).
Result: Etch-and-rinse adhesive Adper Single Bond 2 Total Etch® yielded high bond strength when applied to both primary and permanent dentine. For the one-step Adper Easy One® self-etching adhesive, the bond strength was relatively low regardless of the dentine type. SEM interfacial analysis revealed funnel shaped resin tags and a rough hybrid layer in specimens subjected to total etch system and cylindrical shaped and a smooth hybrid layer in specimens subjected to self etch system in both primary and permanent teeth.
Conclusion: The total-etch systems provide better adhesion to primary and permanent tooth dentine. The self etch systems though convenient to use, do not match the bond strengths of conventional total etch systems. Also, it can be concluded that both adhesives show weaker bond strengths to primary tooth dentin.

Key words: Tensile bond strength, etch, resin-dentine interface

Introduction

Dentistry has primarily been witnessing so many advancements in restorative procedures over the last few decades that earlier techniques have undergone marked variances. The increasing demand for aesthetics has transformed from the century old gold restorations to latest innovative 'nano' enhanced composites and glass ionomers in the practice of Pediatric Dentistry[1]. Principles in restoration have been continually changing over the last four decades and adhesive dentistry has steadily gained in importance. To all these advancements, dentinal bonding, which refers to micromechanical coupling of restorative materials, particularly composites, to human dentine via an intermediary adhesive resin layer has been one of the most important advances, thus enabling use of non retentive cavity preperations, and thereby, emphasizing the concepts of conservation over extension for preservation. The concept of "adhesive restoration" has been, essentially, the most noteworthy development in this ever progressing science.
Adhesive systems that are currently available can be classified according to their interaction with the smear layer. The first system, "the total etching" system, removes the smear layer and demineralises the subsurface dentine via acid etching; this is followed by the application of the bonding agent. The second system, the "self etching system" uses a self-etching acidic primer to demineralize the smear layer and subsurface dentine. Because the primer is not rinsed off, the acidic monomer penetrates into the demineralized dentine, forming the hybrid layer, which is included in the dissolved smear layer. Two generations of self etch adhesives are available today-two step adhesives that consists of an acidic primer and a hydrophobic bonding resin and- all in one adhesives in which acidic primer and bonding resin are combined into one solution. In general, adhesives work by interacting with the enamel/dentine substrate in one of the two different ways: etch-and-rinse and self-etching[2]. The two-step etch-and-rinse adhesive system in which the smear layer is removed with phosphoric acid and the primer and adhesive is combined in a bottle to elicit their functionality by "wet-bonding technique" has been widely used. The main disadvantage of etch-and-rinse is its technique-sensitiveness[3]. In contrast, in the self-etching which has been developed to meet the increasing demand for convenient application, the dentine is simultaneously etched and primed without rinsing[2]. Given that the etching, priming and bonding procedures are taking place in one solution, the one-

step self-etching adhesive system is more hydrophilic than the two-step self-etching adhesive in which separate hydrophobic bonding resins are used[4].
Bonding of an adhesive to the dentine is complex and bond strength is one of the most important performance parameters of dental adhesives[5,6]. Although it has been suggested that bond strength depends on both the type of dentine and the adhesive used due to the inherent characteristics of the dentine as well as the compositions of different adhesives[7], comprehensive evaluations of dentine type-dependent bond strength of different categories of adhesives are still lacking.
The purpose of this study was to determine and compare the tensile bond strengths and to observe the resin-dentine interface of total-etch and self-etch bonding systems to dentine of primary and permanent molar teeth.

Materials and method

The experimental groups which consisted of the 60 samples were divided as Group I (n=15, primary molars), Group II (n=15, permanent molars), Group III (n=15, primary molars) and Group IV (n=15, permanent molars). Group I (primary molars) and Group II (permanent molars) were treated with total etch bonding system (Adper™ Single Bond 2 Adhesive, trademark of 3M, U.S.A.) while Group III (primary molars) and Group IV (permanent molars) were treated with self etch bonding system (Adper™ Easy Bond Adhesive, trademark of 3M, U.S.A.)

Specimen preparation
30 exfoliated primary and 30 extracted permanent non-carious molar teeth were selected for the study after duly obtaining consent from parents of wards in case of primary teeth and adults in case of permanent teeth. The occlusal dentin up to the level of 'yellow tinge' of the samples were trimmed and the teeth were mounted up to the level of cervical constriction on commercially available PVC Cylindrical Blocks (0.95 mm diameter and 1.5 inches height), stabilized using self cure acrylic resin.

Preparation of samples
Total Etch - Group I (Primary) and II (Permanent)
The exposed dentine in each of the 15 primary molars and 15 permanent molars, were etched for a period of 15 seconds for permanent teeth and 7 seconds for primary teeth with 3M Scotch bond etchant (Scotchbond™ Universal Adhesive, trademark of 3M, U.S.A.) in a single coat according to the manufacturers' recommendations, rinsed for 10 seconds and dried for 5 seconds, using 3 way syringe. 3M Adper Single Bond bonding agent (Adper™ Single Bond 2 Adhesive, trademark of 3M, U.S.A.) was applied with an applicator tip in a single coat, gently air dried for 5 seconds to remove the excess solvent and light cured for 10 seconds according to the manufacturers' instructions. Following this application, a 2 mm layer of Z 250 light cure composite (3M™ Filtek™ Z250, trademark of 3M, U.S.A.) was cured on the prepared surface.
Self Etch - Group II (Primary) and IV (Permanent)
The exposed dentine in each of the 15 samples, was etched for a period of 20 seconds for permanent teeth and 10 seconds for primary teeth with self etch single bottle system Adper Easy One (Adper™ Easy Bond Adhesive, trademark of 3M, U.S.A.) bonding agent according to the manufacturers' recommendations and air thinned for approximately 5 seconds until the film no longer moved. The adhesive layer was light cured for 10 seconds according to the manufacturers' instructions. Composite restoration was built using 3M Filtek Z250 (3M™ Filtek™ Z250, trademark of 3M, U.S.A.) and cured for 20 seconds.

Tensile Bond Strength Testing
13 prepared samples in each group were selected for Tensile bond strength testing. A 'U' shaped bent 28" stainless steel wire was made to pass through the first cured layer of composite with the ends of the wire left free to grip on the Universal Testing Machine (LR50K Series, 50 kN Universal Testing System, Lloyd Instruments Ltd, United Kingdom). 2 increments of 2 mm each of light cure Z250 composite was measured with micro surveyor and cured over the placed stainless steel wire on the first layer.
The Tensile bond strength values of the specimens were obtained by testing the specimens in a Universal Testing Machine with a cross head speed of 1mm/minute. The values obtained in Newtons were converted to Megapascals by dividing the values obtained by the area of bonding. The values were tabulated and statistically analyzed.

Scanning Electron Microscopy
Two prepared samples in each group were selected for Scanning Electron Microscopy. The sectioned samples were fixed in Karnowsky's fixative which consists of 2.5% gluteraldehyde + 2.5% paraformaldehyde (Polysciences Asia-Pacific, Inc. 2F-1, 207 Tunhwa N. Rd. Taipei, Taiwan) for 24 hours. The cut surfaces were polished sequentially with 320, 600 and 1000 grit silicon carbide papers, immersed in 10% phosphoric acid for a period of 3-5 seconds and rinsed with distilled water for 15 seconds. Further, the specimens were subjected to treatment with 5% sodium hypochlorite solution for 5 minutes and rinsed thoroughly with distilled water. Following this, the specimens were dehydrated using ascending concentrations of ethanol by serial treatment with 30%, 40%, 50%, 70% 90% and 100% for 30 minutes each. The specimens were critically dried, gold sputtered and examined under the Scanning Electron Microscope to observe the resin-dentin interface morphology.

Results

Tensile Bond Strength
The mean tensile bond strength in dentine of primary and permanent molars subjected to Total Etch system (Primary, 25.50 MPa; Permanent, 30.38 MPa) **(Table 1)** was found to be significantly higher than that of the molars subjected to Self Etch system (Primary, 22.34 MPa; Permanent, 27.24 MPa)
A significant difference was recorded between dentine of Primary molars (Total etch, 25.50 MPa; Self Etch, 22.34 MPa) and dentine of Permanent molars

(Total Etch, 30.38 MPa; Self Etch, 27.34 Mpa) as results indicated higher bond strength values in permanent molars for samples subjected to both total etch and self etch systems.

Table 1. Table showing mean tensile bond strength values (in MPa) and student 't' test values to dentin among primary teeth and permanent teeth using total etch and self etch adhesive systems.

Group	Mean	tVal	P Val
Group I (Primary, Total Etch)	125.50		
		3.1895	p<0.01 significant
Group III (Primary, Self Etch)	22.34		
Group II (Permanent, Total Etch)	30.38		
		3.0903	p<0.01 significant
Group IV (Permanent, Self Etch)	27.34		
Group I (Primary, Total Etch)	25.50		
		5.1471	p<0.01 significant
Group II (Permanent, Total Etch)	30.38		
Group III (Primary, Self Etch)	22.34		
		4.8832	p<0.01 significant
Group IV (Permanent, Self Etch)	27.34		

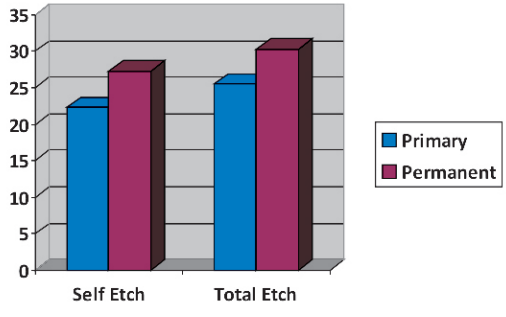

Figure 1. The average Tensile Bond Strength values (in MPa) among Primary and Permanent Teeth using Self Etch and Total etch adhesive systems.

Scanning Electron Microscopy

SEM examination was done to check the resin-dentine interface of total etch bonding system and self etch bonding system to dentine in primary and permanent molars for the morphology of resin tags and texture of hybrid layer. The resin tags appeared thick and funnel shaped in the specimens bonded with the total-etch system to both primary and permanent dentine **(Figure 1)**, whereas the resin tags appeared cylindrical in the self etch system **(Figure 2)**. The hybrid layer appeared to be rough in specimens subjected to total etch system **(Figure 1)** and smooth in those subjected to self etch system **(Figure 2)** irrespective of the primary or permanent specimen type.

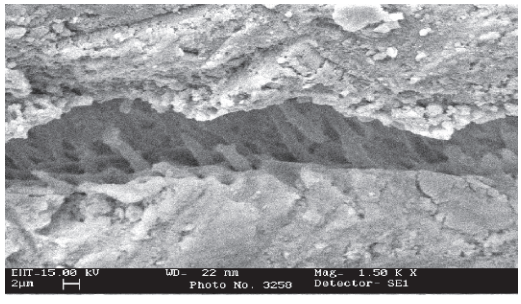

Figure 2. SEM photomicrograph showing the resin-dentin interface of a Molar treated with Total Etch adhesive system. Same type of interface was recorded both in primary and permanent dentin.

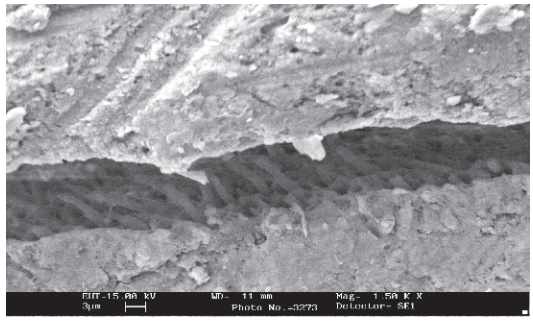

Figure 3. SEM photomicrograph showing the resin-dentin interface of a Molar treated with Self Etch adhesive system. Same type of interface was recorded both in pimary and permanent dentin.

Discussion

With the total etch technique, dentine and enamel were treated with an acid gel to demineralise the most superficial hydroxyapatite crystals. Following this chemical etching, a mixture of resin monomers (primer/adhesive) dissolved in an organic solvent is applied to infiltrate etched dentine. An intimate micromechanical entanglement of resin monomers with etched dentine make way for an excellent marginal fit and may even act as an elastic buffer that compensate for elastic shrinkage stresses during contraction of the composite, thus ultimately improving bond strengths.

Self etch adhesives contain ethanol, which is used to form co-solvents with water and resin for better impregnation. Studies by Ceballos et al (2003)[7], Perdigão et al (2006)[8] and Abdelaziz et al (2009)[9] reported less than optimum performance in dentine bonding by single step self etch adhesives because of a variety of problems associated with their higher hydrophilicity (causing water sorption), low viscosity (causing nano leakage), and monomer – solvent phase separation (causing hydraulic degradation) creating water droplets in the adhesive after polymerization[10]. This could have contributed to the poor performance of the self etch adhesive in both primary and permanent dentine tested in this study.

It has been said that permanent dentine is more highly mineralized, based on the fact that hardness is related to the degree of mineralization[11]. In another study, energy-dispersive-x-ray spectrometry (EDS) was used, and the results suggested that concentrations of calcium and phosphorus in both peritubular and intertubular dentine are more in permanent teeth as compared to primary teeth[12] owing to better acid conditioning in permanent teeth. Evaluation of the dentinal micromorphology also indicates potential differences between primary and permanent teeth[13].

Compared with permanent teeth, primary teeth also presented a lower concentration and a smaller diameter of dentinal tubules at a distance of 0.4 to 0.5 mm from the pulpal surface reflecting lower amount of remaining dentine thickness (RDT) and thus resulting in lower bond strength values in primary molars. This could be a reason for reported lower bond strengths in primary molars than permanent molars.

Our resin-dentine interface observations were similar to previous reports by Nakornchai et al (2005)[14] who reported the same appearance of funnel shaped tags using total etch and cylindrical tags using self etch system in case of primary dentine. Nakajima et al(5) observed numerous long well formed resin tags in the intact primary dentine with small funnel shaped lateral extensions of micro-tags extending from the main resin tags when subjected to total etch system. SEM observations by Yildirim et al in 2008 have shown similar interfacial morphology for total etch and self etch adhesive systems including the thickness of the hybrid layer and the length of the resin tags in primary dentin[15].

Conclusion

The total-etch systems provide better adhesion to primary and permanent tooth dentin. The self etch systems though convenient to use, do not match the bond strengths of conventional total etch systems. Also, it can be concluded that both adhesives show weaker bond strengths to primary tooth dentine.

Future studies emphasising on a qualitative analysis of the resin-dentin interface of both primary tooth dentine and permanent tooth dentine and its correlation with their bond strengths are recommended.

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