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Bond Strength of Newer Dentin Bonding Agents in Different Clinical Situations

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Clinical Relevance

The bond strengths of adhesive systems are affected by the amount of moisture on the bonding surface of the tooth.

SUMMARY

This study compared the tensile bond strengths of different adhesive systems to different dentinal substrate conditions. The adhesive systems used were Adper Single Bond 2 total etch, XP Bond total etch, and two all-in-one adhesives, Adper Easy One and Xeno V. Ninety-six intact human premolars extracted on periodontal or orthodontic grounds were collected and stored in 0.9% physiologic saline for less than four weeks. Teeth were carefully cleaned to remove the soft tissue remnants. The occlusal surfaces were ground until the level of the dentinoenamel junction, exposing superficial dentin using a low-speed diamond disc. A standard smear layer was produced by wet sanding the dentin surface with 600-grit silicon carbide sandpaper for 40 seconds. The 96 specimens were embedded in autopolymerizing acrylic resin and divided into four groups of 24 each based on the dentin bonding agents used. Each group consisted of 24 specimens, which were further divided into two subgroups of 12 specimens each, moist dentin and dentin air-dried for 10 seconds. The regions of interest for adhesion were demarcated in each sample using adhesive tape, with a 4-mm–diameter hole isolating the peripheral region, and each adhesive system was employed. Four adhesive systems, two total etch and two self-etching adhesive, were applied to different dentinal substrates as per the manufacturers' instructions. Following the adhesive application, the specimens were restored with composite material using a metallic mold measuring 5 mm in diameter and 5 mm in height to keep the material in cylindrical form and were light cured per manufacturers' instructions. After being stored for 24 hours in distilled water at room temperature, the specimens were thermocycled for 550 cycles at temperatures ranging from 5°C to 60°C with a dwell

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time of 15 seconds in each bath and a transfer time of five seconds. The tensile bond strengths were determined using a Universal loading machine (Lloyd Universal Testing Machine) at a cross-head speed of 1 mm/min. On moist dentin, total-etch adhesives showed higher bond strength than did the all-in-one adhesives. Under the dry dentin condition, XP Bond exhibited significantly higher bond strength than did the Adper Single Bond 2 and two all-in-one adhesives, Adper Easy One and Xeno V.

INTRODUCTION

The achievement of high-strength, durable bonds between tooth structure and restorative materials has been a long-term goal of the dental profession.[1] Dental adhesive systems are used to promote adhesion between composite resins and dental structure and should present a similar performance on both enamel and dentin.[2] Resin bonding to enamel is considered a durable and predictable clinical procedure.[3-5] The micromechanical nature of the interaction of dental adhesives with enamel is a result of the infiltration of resin monomers into the microporosities left by acid dissolution of enamel and the subsequent enveloping of the exposed hydroxyapatite crystals with polymerized monomers within pores in the enamel surface.[6] Successful attempts to bond to dentin in a similar fashion have been extensively studied.[5] The dentin substrate differs from enamel, as it presents more organic contents, an increased presence of fluid inside the dentinal tubules, a smear layer, and an inherent surface wetness.[2,7]

Adhesive systems can be classified based on how they react with the smear layer. Three mechanisms of adhesion are currently in use with modern adhesives: etch-and-rinse adhesives, which remove the smear layer and superficial hydroxyapatite through etching with a separate acid gel; self-etch adhesives, which make the smear layer permeable without removing it completely; and glass ionomer adhesives, which are self-adhesive to tooth tissues.

The etch-and-rinse adhesion strategy, or total-etch technique, involves at least two steps—three steps in its most conventional form—with application of the conditioner or acid etchant, followed by the application of a primer or adhesion-promoting agent and then application of the adhesive resin.[8] For the etch-and-rinse adhesive systems, typically two steps are required: selective dissolution of hydroxyapatite crystals and exposure of collagen network through etching, followed by *in situ* resin polymerization.[9]

The bonding mechanisms of self-etching primers are based upon the simultaneous etching and priming of the smear layer–covered dentin using an acidic primer, followed by the application of the adhesive resin.[5]

Recently, many clinicians have shifted to one-step self-etch adhesive systems, also referred to as all-in-one adhesives, in which the manufacturers have attempted to incorporate all of the primary components of adhesive systems (etchant, primer, and bonding resin) into a single solution. All-in-one adhesives are user friendly, in that fewer steps are required for the bonding protocol.[7]

The adhesion of glass ionomer adhesives has been determined to be a twofold process. Micromechanical interlocking is achieved by shallow hybridization of the microporous, hydroxyapatite-coated collagen fibril network. In this respect, glass ionomers can be considered as adhering to tooth tissue through the "mild self-etch approach" produced by the heavy-molecular-weight, polycarboxyl-based polymer. As the second component of the self-adhesion mechanism, true primary chemical bonding occurs through the formation of ionic bonds between the carboxyl groups of the polyalkenoic acid and calcium of the remaining hydroxyapatite around the exposed surface collagen.[8]

The total-etch and self-etch adhesive systems are contemporary and are both dividing the preference of clinicians, mainly when technical simplification vs effectiveness of adhesion to different dentinal substrates is considered.[2] Dental adhesives display different bonding strengths on different substrates and in different conditions on the same substrate.[10] The durability of the adhesive-dentin interface is related to its quality, based upon the ability of adhesive monomers to occupy the spaces created by the removal of apatite mineral by acid etching and on their ability to envelop the exposed collagen fibrils. After the dentin surface is conditioned, it is recommended that the surface should be maintained in a moist state prior to bonding, which is commonly referred to as wet bonding. The degree of wetness that is ideal for resin dentin bonding varies widely among the different adhesive systems and depends on the incorporated solvents.[11]

The bond strength of total-etch adhesives to moist dentin was found to be higher than to dry dentin in previous studies.[2,11-16] However, the moisture condition of substrates interfered with the performance of one-step self-etching adhesives, with the best moisture condition being material dependent.[17] In a study by Hashimoto and others,[18,19] the bond strength of all-in-one adhesives to dry dentin was significantly greater than to wet dentin. Further, those results showed that bond strength and nanoleakage formation depended on the bonding substrate (wet vs dry dentin) before bonding. Those authors concluded that one-bottle self-etching adhesives might adsorb the water from dentinal tubules during bonding, leading to nanoleakage formation and, thus, a decline in bond strength.[18,19,20] Mohan and Kandaswamy[15] compared different bonding systems and techniques to assess which was the most successful. These authors concluded that maximum infiltration was seen when the dentin was kept moist after acid etching. They determined that the best way to keep the acid-etched dentin moist was by blotting the excessive moisture using an absorbent paper.[15] Manso and others[11] concluded that surface moisture affected the bond strength, thereby influencing the stability of bonds over time.

With advancements in adhesive systems, manufacturers claim that newer dentin adhesive systems are less sensitive to moisture while bonding effectively to dry dentin substrates. Hence, scientific studies are required to support the manufacturers' claims. The purpose of this study was to evaluate the bond strengths of newer dentin bonding agents to an altered dentin surface.

MATERIALS AND METHODS

Ninety six intact human premolars, extracted for periodontal or orthodontic reasons, were collected and stored in 0.9% physiologic saline for less than four weeks. The teeth were disinfected and handled per the recommendations and guidelines laid down by the Occupational Safety and Health Administration and the Centers for Disease Control and Prevention.[21,22] Teeth were carefully cleaned with curettes to remove the soft tissue remnants. The current study was carried out by one person. The occlusal surfaces were ground to the level of the dentinoenamel junction, thus exposing superficial dentin, using a low-speed diamond disc. A standard smear layer was produced by wet sanding the dentin surface with 600-grit silicon carbide abrasive papers (Navyug Sales Corporation, Delhi, India) for 40 seconds. The 96 specimens were embedded in autopolymerizing acrylic resin and divided into four groups of 24 specimens each, based on the dentin bonding agents used. Each group consisted of 24 specimens, which were further divided into two subgroups of 12 specimens each, moist dentin and dentin air-dried for 10 seconds. The four commercially available adhesive systems tested comprised two total-etch adhesives: Adper Single Bond 2 (3M ESPE, St Paul, MN, USA) and XP Bond (Dentsply Detrey, Konstanz, Germany) and two all-in-one adhesives: Adper Easy One (3M ESPE, 3M Gulf Ltd, Dubai, UAE) and Xeno V (Dentsply Detrey).

The regions of interest for adhesion were demarcated in each sample using adhesive tape, with a 4-mm–diameter hole isolating the peripheral region. The four adhesive systems were applied to different dentinal substrates per the manufacturers' instructions (Table 1). Following application of the adhesive, the specimens were restored with a composite material (Filtek Z 350, A2 shade, 3M ESPE, 3M India Ltd, Bangalore, India) in increments using a metallic mold, which was 5 mm in diameter and 5 mm in height, to keep the material in a cylindrical form. The cylindrical form was restored in three increments with light curing of each increment using a quartz tungsten halogen light (QLF 75 curing light, Model 502, Dentsply Caulk, Milford, CT, USA), per the manufacturer's instructions. The light output of the curing unit was monitored using a Light Optimeter (Optilux radiometer, Kerr Sybron dental specialties, Middleton, WI, USA).

After storage for 24 hours in distilled water at room temperature, the specimens were thermocycled for 550 cycles at temperatures ranging from 5°C to 60°C, with a dwell time of 15 seconds in each bath and a transfer time of five seconds. The tensile bond strengths were determined using a Universal Testing Machine (Lloyd Instruments, LR 50K, West Sussex, PO22 9ST, UK) at a cross-head speed of 1 mm/min. The tensile bond strengths (in MPa) were calculated by dividing the maximum force that induced failure by the bonded area. The bond strength data obtained in this present study were subjected to statistical analysis using one-way analysis of variance (ANOVA) and Tukey Honestly Significantly Different (HSD) tests to determine the effects of dentinal moisture on the bond strength of different dentin bonding agents.

RESULTS

The present study assessed and compared the tensile bond strengths of two total-etch and two self-etch adhesive systems under different dentinal substrate conditions: moist and air-dried for 10 seconds. The mean bond strengths and standard deviations (in MPa) are shown in Table 2.

Table 1: *Composition and Application Procedures of the Adhesives Systems*

Adhesive	Manufacturer	Composition	Procedure
Adper Single Bond 2	(3M ESPE, St Paul, MN, USA)	Etchant: 35% phosphoric acid Adhesive: Bis GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, a methacrylate functional copolymer of polyacrylic and polyitaconic acids, silica filler	1. Apply acid for 15 s and rinse a. Subgroup 1: blot dry b. Subgroup 2: air-dry 10 s 2. Apply two coats with mild agitation and dry gently 3. Light cure for 20 s
XP Bond	(Dentsply Detrey, Konstanz, Germany)	Etchant: 36% phosphoric acid Adhesive: carboxylic acid modified dimethacrylate (TCB resin), PENTA, UDMA, TEGDMA, HEMA, butylated benzenediol (stabilizer), ethyl-4-dimethylaminobenzoate, camphorquinone, functionalized amorphous silica, t-butanol, water	1. Apply acid for 15 s and rinse a. Subgroup 1: blot dry b. Subgroup 2: air-dry 10 s 2. Apply two coats, leave undisturbed for 15 s, and dry gently 3. Light cure for 20 s
Adper Easy One	(3M ESPE, 3M Gulf Ltd, Dubai, UAE)	Adhesive: 2 HEMA, Bis GMA, methacrylated phosphoric esters, 1,6 hexanidol dimethacrylate, methacrylate functionalized polyalkenoic acid, camphorquinone, ethanol, water, silica filler, and stabilizer	1a. Subgroup 1: blot dry 1b. Subgroup 2: air-dry 10 s 2. Apply adhesive, agitate for 20 s, and dry gently for 5 s 3. Light cure for 10 s
Xeno V	(Dentsply Detrey)	Adhesive: bifunctional acrylates, acrylic acid, acid phosphoric functionized ester, acid acrylated water, tertiary butanol, phosphine oxide initiator, stabilizer	1a. Subgroup 1: blot dry 1b. Subgroup 2: air-dry 10 s 2. Apply adhesive, agitate for 20 s, and dry gently for 5 s 3. Light cure for 20 s

Abbreviations: 2 HEMA, hydroxyethylmethacrylate; BIS-GMA, (1 methylethliede)bis[4,1-phenyleneoxy (2-hydroxyl-3,1propanediyl)bismethacrylate; TCB, Butan-1,2,3,4-tetracarboxylic acid, di-2-hydroxyethylmethacrylate ester; PENTA, dipentaerytritolpentacrylate-phosphoricacid-monomer; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

When the tensile bond strengths of the four groups under dry and moist dentinal substrate conditions were analyzed using one-way ANOVA, a very high statistical significance was found ($p<0.05$), revealing that bond strengths were influenced by different dentinal substrate conditions. An intercomparison between adhesive systems under moist and dry dentinal substrate conditions was done using the Tukey HSD test.

Under the dry dentinal substrate conditions, XP Bond showed significantly higher bond strengths than the other dentin adhesives tested. There was a statistically significant difference between the two all-in-one adhesives tested (Table 3).

Under the moist dentinal substrate conditions, the total-etch adhesive systems exhibited significantly higher bond strengths than the self-etch all-in-one adhesives, with XP Bond showing the highest bond strength. Adper Easy One showed greater bond strength than Xeno V among the all-in-one adhesives tested (Table 4).

DISCUSSION

Adhesive dentistry is a rapidly evolving discipline. New product development is occurring at an unprecedented rate.[23] Adhesion to dentin depends not only on the adhesive systems but also on the dentin substrate.[24] Clinical trials are the ultimate test for dental restorations, but they cannot differentiate the true reason for failure because of the simultaneous impact of diverse stresses on restorations within the aggressive oral cavity. Lab testing can evaluate the effect of a single variable while keeping all other variables constant.[8]

Etch-and-rinse adhesives require a separate step of etching, which is usually performed with 30–40% phosphoric acid.[25,26] In etch-and-rinse adhesive systems, the quality of the created bond is greatly influenced by the duration of the etching process and

Table 2: *Minimum, Maximum, and Mean Bond Strength (Standard Deviation) of the Four Groups in Dry and Moist Dentin Substrate Conditions*

Groups	N	Minimum, MPa	Maximum, MPa	Mean, MPa
Dry dentin[a]				
Adper Single Bond 2	12	12.64	18.68	15.7 (1.93)
XP Bond	12	13.65	20.23	17.95 (1.92)
Adper Easy One	12	13.24	15.63	14.32 (0.98)
Xeno V	12	4.8	10.21	8.5 (1.54)
Moist dentin[b]				
Adper Single Bond 2	12	20.01	25.01	23.26 (1.65)
XP Bond	12	21.96	26.78	24.42 (1.72)
Adper Easy One	12	15.2	19.85	18.09 (1.59)
Xeno V	12	12.63	16.52	14.68 (1.28)

[a] *$F=71.71$ for dry dentin, $p<0.05$.*
[b] *$F=100.25$ for moist dentin, $p<0.05$.*

by the amount of dentin surface moisture following rinsing of the etching acid and prior to resin infiltration. In the present study, the total-etch adhesives, XP Bond and Adper Single Bond 2, showed greater bond strengths to moist dentin when compared to the other self-etch adhesives, Adper Easy One and Xeno V. This finding is in agreement with studies conducted by Susin and others[2]; Perdigao[7]; Van Meerbeek and others[8]; Hegde and Bhandary[16]; Ceballos and others[27]: and Cardoso and others.[28] Kanca,[12] Manso and others,[11] and Reis and others[29] showed that during wet bonding, water maintained the collagen fibrils in an expanded condition, and the adhesive systems were able to infiltrate the substrate and produce high bond strengths. Reis and others[29] confirmed that total-etch adhesive systems required a moist dentin surface before bonding.

Table 3: *Comparison of Tensile Bond Strength Between Groups Under Dry Dentinal Substrate Conditions—Tukey Honestly Significantly Different (HSD) Post Hoc Multiple Comparisons Test*[a]

Dry Dentin	XP Bond	Adper Easy One	Xeno V
Adper Single bond 2	0.008	0.187	0.001
XP Bond	–	0.001	0.001
Adper Easy One	–	–	0.001

[a] *The mean difference is significant at the 0.05 level.*

Table 4: *Comparison of Tensile Bond Strength Between Groups Under Moist Dentinal Substrate Conditions—Tukey Honestly Significantly Different (HSD) Post Hoc Multiple Comparisons Test*[a]

Moist Dentin	XP Bond	Adper Easy One	Xeno V
Adper Single Bond 2	0.280	0.001	0.001
XP Bond	–	0.001	0.001
Adper Easy One	–	–	0.001

[a] *The mean difference is significant at the 0.05 level.*

The depth of demineralization promoted by phosphoric acid determines the thickness of the hybrid layer, as the application of phosphoric acid before the primer/adhesive acts by removing the smear layer, demineralizing the dentin structure, and consequently exposing the collagen fibrils to allow formation of the hybrid layer.[7] The exposed collagen may provide reactive groups that can chemically interact with bonding primers. The solvents used are t-butanol in XP Bond and ethanol in Adper Single Bond 2, as a result of their high vapor pressure, which competes with and replaces moisture, promoting infiltration of monomers through the nanospaces of the exposed collagen network. This collagen network serves as a framework for the creation of a resin-demineralized dentin hybrid layer, resulting in strong micromechanical interlocking between the resin and the superficially demineralized dentin.[30] Hence, under moist conditions, total-etch adhesive systems showed better bond strengths than self-etch adhesive systems.

Self-etching systems incorporate a significant amount of water as a solvent in order to promote the ionization of the acidic monomers, making these dental adhesives permeable membranes that are highly susceptible to the degrading effects of water. After solvent evaporation, the adhesive layer can be very thin, and its mechanical properties may be low.[31] Self-etching adhesive systems rely on acidic monomers to simultaneously demineralize and infiltrate both the enamel and the dentin. This acidity must be neutralized by the mineral content of the tooth structure to allow complete polymerization of the adhesive film. With total-etch adhesives, the smear layer and the dissolved mineral are removed during the rinsing step. As there is residual acidity and an inability to totally remove the smear layer, questions regarding the long-term hydrolytic stability of self-etching adhesive systems have been raised.[23]

The bond strength of all of the adhesives was decreased under dry conditions. When dentin surfaces were dried with an airstream, much of the water located in the matrix was removed, causing the matrix to collapse, approximating collagen fibrils. As collagen peptides touch, they form new interpeptide H bonds that stabilize and stiffen the matrix in a shrunken state. Air-dried, shrunken dentin has few interfibrillar spaces for resin penetration, thereby compromising the bonding of resins to dentin.[32] This is in corroboration with the findings of other studies,[3,12-15] in which there was a significant decrease in bond strength with dry substrate conditions.

Under the dry dentin substrate conditions, XP Bond showed the greatest bond strength. XP Bond is a new, one-bottle etch-and-rinse adhesive, composed of a premixed solution of monomers dissolved in T butanol. Based on an improved ability to diffuse through partially collapsed demineralized dentin, XP Bond is less technique sensitive to moisture.[33] Other components that contribute to the higher bond strength and better adhesion of XP Bond may be dipentaerytritolpentacrylate–phosphoric acid-monomer (PENTA), which chemically bonds to the residual calcium in the tooth, and butane-1,2,3,4-tetracarboxylic acid, di-2-hydroxyethylmethacrylate ester (TCB resin). PENTA is a weak acid, an adhesion promoter that facilitates penetration of resin monomers into dentin for micromechanical bonding.[3]

The solvent content of adhesives influences their bond strength to dry dentin. Adhesive systems that include water as one of the solvents in the mixture have been shown[11] to be able to promote re-expansion of the collapsed fibrils. The bond strength to dry dentin was comparable for water-/ethanol-based adhesive systems, as with Adper Single Bond 2 and Adper Easy One. Reis and others[29] suggested that water-based adhesive systems were capable of producing high bond strengths when applied to extensively dried dentin. Ethanol and water are polar solvents and have the potential of expanding a collagen matrix that has collapsed because of drying. The use of adhesives that contain solvents with the potential for hydrogen bonding, as with ethanol and water, would be useful where the dentin surface has been allowed to dehydrate excessively.[1]

Self-etch adhesives differ in their aggressiveness. They can be classified into three categories, according to acidity, as mild, moderate, and aggressive. Xeno V, with a $pH<2$, is a mild self-etch adhesive system and demineralizes dentin to a depth of 1 μm. This superficial demineralization occurs only partially, allowing residual hydroxyapatite to remain attached to the collagen. Nevertheless, sufficient porosity is created to obtain micromechanical interlocking through hybridization. The thickness of the hybrid layer is, however, much smaller than that produced by the strong self-etch or etch-and-rinse approach.[8]

In the fundamental principles of adhesion, the primary mechanism contributing to the formation of adhesion is micromechanical bonding. It is suggested that advances in adhesion, for which reliability and durability of the bonds are the ultimate goal, should be based on improved chemistry of the interface and the ability to have adhesives react with tooth substrates. This reaction can only occur when the barrier layers are removed and the substrate itself is exposed. This is difficult to accomplish with no-rinse adhesive systems because calcium salts, amorphous calcium phosphate, and dissolved proteins remain as part of the monomer mixture at bonding sites. Bonding systems applied to surfaces from which these barriers are not removed likely inhibit the ability to form bonds directly with the substrates.[1]

CONCLUSIONS

The following conclusions can be drawn from the current study:

1) Both total-etch and self-etch adhesives exhibit greater bond strength to moist dentin than to dry dentin, indicating that a certain amount of moisture is essential for obtaining optimal bonding.
2) Drying the dentin substrate comparatively reduced the bond strength of all of the adhesives.
3) XP Bond showed the highest bond strength for both the moist and dry dentin conditions.

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