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Original Research

Comparative Evaluation of Shear Bond Strength of Three Dental Adhesives under Dry and Wet Bonding Conditions: An *In Vitro* Study

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Abstract:

Background: The old concept of total-etch/dry bonding has been superseded by total-etch/wet bonding, and later on by the self-etch systems. Manufacturers have introduced single bottle systems to be used in total-etch, self-etch, or selective-etch modes under both dry and moist conditions. The aim of this study was to evaluate shear bond strengths of three different dental adhesives under dry and wet bonding conditions, using etch-and-rinse and self-etch protocols.

Materials and Methods: A total of 60 extracted human mandibular molars were decoronated perpendicular to the long axis to expose flat coronal dentin surface embedded in acrylic resin. The teeth were randomly allocated into 4 experimental groups depending on bonding agent used: Group-I: Adper™ Single Bond 2 (Wet bonding); Group-II: Adper™ Easy Bond (Dry bonding); Group-III: Single Bond Universal™ (Dry bonding); and Group-IV: Single Bond Universal™ (wet bonding), with 15 teeth per group (n = 15). Composite cylinders of 3 mm height were built and subjected to shear bond strength testing using a Universal testing machine. Data was analyzed statistically by ANOVA followed by *post-hoc* Tukey test.

Results: Group-I (Adper™ Single Bond 2; total-etch/wet bonding) showed the highest shear bond strength followed in order by Group-IV (Single Bond Universal™; total-etch/wet bonding); Group-III (Single Bond Universal™; self-etch/dry bonding); and Group-II (Adper™ Easy Bond; self-etch/dry bonding). Group-I (Adper™ Single Bond 2) showed statistically significant difference ($P < 0.001$), with higher shear bond strength than Groups - II, III or IV.

Conclusion: A higher shear bond strengths may be attained with Adper™ Single Bond 2 (Group-I) used under wet bonding conditions.

Key Words: Dental adhesives, dry bonding, shear bond strength, wet bonding

Introduction

The advent of modern adhesive dentistry began with Buonocore in 1955, who reported that acids could be used to alter the surface of enamel to render it a more receptive to adhesion.[1] Ever since this landmark discovery, the main challenge for dental adhesives, to this day, is the need for an effective bond to dental substrates of different nature. Whereas bonding to enamel is reliable and durable,[2] it is a more difficult to achieve in the dentin due to its wet tubular ultrastructure and organic composition.[3] The fundamental bonding mechanism to enamel and dentin is essentially based on an exchange process in which minerals removed from the dental hard tissues are replaced by resin monomers that on polymerization become micromechanically interlocked in the created porosities.[3]

On the basis of the underlying adhesion strategy, i.e., the nature of interaction with enamel and dentin substrates, contemporary adhesive systems can be classified into etch-and-rinse and self-etch adhesives. While the etch-and-rinse approach requires a separate acid-etch step to promote dentin and enamel demineralization before monomer infiltration; in the self-etch approach, demineralization, and infiltration occur simultaneously.[4]

In the late 1970's, "dry bonding" was widely employed by clinicians who used the etch-and-rinse adhesives, to confirm that the etched enamel margins had a frosty or chalk-like color. The deleterious effect that resulted from drying the cavity was that there was the collapse of the demineralized dentin, thus hindering monomer infiltration.[5] Resin-enamel bond strengths has been measured to be as high as 20 MPa, but resin-dentin bond strengths were as low as 5 MPa when dry bonding is employed. These results collaborate with studies conducted by Pashley *et al.*, Titley *et al.* and Perdigão.[6] The low resin-dentin bond strength associated with "dry bonding" created dentin sensitivity, microleakage, secondary caries, and loss of bonded restorations. In 1991, Kanca found that water was an excellent rewetting agent and this later led to advent of the "wet bonding" concept. This technique increased the resin - dentin bond strength, allowing good sealing of dentin and less incidence of post-operative pain.[2] The increase in resin-dentin bond strengths were seen similarly when wet bonding was followed in studies done by Gwinett *et al*. and Perdigao *et al*.[7]

There is no simple answer to the question of what is "too wet" or "too dry." Thus, total-etch products are technique-sensitive. In the laboratory setting, one can obtain a relatively uniform surface "wetness" on flat dentin surfaces. Clinically, however, there is a tendency to over-dry the pulpal or axial wall of complex cavities and to pool water at axial-gingival line angles, leaving the dentin surface with a non-uniform degree of wetness and non-uniform resin infiltration. This, in turn, leads to microleakage and post-operative sensitivity.[8]

The development of self-etching primer adhesive systems has greatly simplified resin bonding procedures, as a separate etching step is no longer required. In addition; a rinsing step is not required. Bonding can be accomplished under relatively dry conditions, avoiding the variables associated with wet bonding.[8]

As new adhesive systems are being introduced into the market, with claims of improved composition and ability to bond to the tooth structure, scientists and researchers feel the obligation to substantiate these claims.[9] A new bonding agent, Single Bond Universal™ (3M ESPE) has been recently introduced into the market. The manufacturer claims that it is a single-bottle system for all surfaces, and can be used in total-etch, self-etch or selective-etch mode for both direct and indirect restorations. The manufacturer also claims that this new adhesive can be used under both dry and moist conditions. Further, it is claimed that when used in dry conditions, there is no compromise in bond strength.

Thus, the aim of this study was to evaluate the shear bond strength between Single Bond Universal™ using an etch-and-rinse and a self-etch protocol, compared with a self-etch bonding system (Adper™ Easy Bond) and a total-etch bonding system (Adper™ Single Bond 2), under dry and wet bonding conditions. The null hypothesis was there will be no difference in shear bond strength of the three dental adhesives to dentin, under dry and wet bonding conditions, using the etch-and-rinse and self-etch protocols.

Materials and Methods

About 60 extracted non-carious permanent human mandibular molars were selected. Teeth were cleaned with an ultrasonic scaler. Each tooth was decoronated using a diamond disc with water coolant applied perpendicular to the long axis of the teeth to expose a flat coronal dentin surface. The cut dentin surfaces were then abraded against 600-grit wet silicon carbide papers for 60 s to produce a uniform smear layer. The teeth were mounted on a plastic ring using acrylic resin up to 1 mm from cementoenamel junction, and then randomly allotted into the following 4 groups with 15 teeth each:

- Group I (n = 15): Total-etch system - 3M ESPE Adper™ Single Bond 2 (wet bonding);
- Group II (n = 15): Self-etch system - 3M ESPE Adper™ Easy Bond (dry bonding);
- Group III (n = 15): Self-etch system - 3 M ESPE Single Bond Universal™ (dry bonding);
- Group IV (n = 15): Total-etch system - 3M ESPE Single Bond Universal™ (wet bonding).

The trade name, chemical composition, and manufacturer of the adhesive materials are presented in Table 1.

The dentin bonding systems were applied following the manufacturer's instructions in all four groups. The following application of the adhesive, a Teflon mold was used to build up a cylinder of composite resin onto the dentinal surface of all samples, measuring 2 mm in diameter and 3 mm in height, using a two layer increment technique. Each layer was light-cured using an LED curing unit according to manufacturer's instructions. The specimens were then stored in distilled water at 37°C for 24 h. The samples were then subjected to 500 thermocycles with a dwell time of 5 s between 50°C and 55°C, according to the ISO TR 11450 standard (1994).

Finally, the specimens underwent a shearing bond strength test using a Hounsfield Universal testing machine. Each specimen was fixed on the jig in a manner in which the composite cylinder was parallel with the horizon, and the equipment blade was placed adjacent to the composite-tooth interface perpendicular to composite resin at a strain rate of 0.5 mm/min. The reading value taken from the gauge was measured in kilogram; the force was then converted into megapascal (MPa) by converting kilogram to Newton, then dividing by the composite resin surface area.

The data was analyzed statistically by one-way ANOVA for multiple comparisons followed by *post-hoc* Tukey test (Bonferroni method).

Results

The highest shear bond strength was recorded in Group-I (Adper™ Single Bond 2) used in wet bonding conditions; this was followed by decreasing shear bond strength values in the following order: Group-IV (Single Bond Universal™; wet bonding), Group-III (Single Bond Universal™; dry bonding) and Group-II (Adper™ Easy Bond; dry bonding) (Table 2). The difference in mean shear bond strength among the groups was found to be statistically significant ($P < 0.001$) (Table 3). Thus, the null hypothesis was rejected.

To find out among which pair of groups there exist a significant difference, multiple comparisons were done using the Bonferroni method (Table 4). The difference in mean shear bond strength was found to be statistically significant between

Table 1: Composition and procedural steps of the bonding systems as per the manufacturers' instructions.

Bonding agent tested	Composition	Self-etch strategy	Etch-and-rinse strategy
Adper™ Single Bond 2	1. Etchant: 35% phosphoric acid (Scotchbond Etchant) 2. Adhesive: Bis-GMA, HEMA, dimethacrylate, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly (itaconic) acids, 10% by weight of 5 nm-diameter spherical silica particles		1. Apply etchant for 15 s 2. Rinse for 10 s 3. Blot excess water 4. Apply 2-3 consecutive coats of adhesive for 15 s with gentle agitation 5. Gently air dry for 5 s 6. Light polymerize for 10 s
Adper™ Easy Bond	Methacrylated phosphoric esters Vitrebond™ Copolymer Nanofiller Ethanol Water Dimenthacrylate HEMA Initiators	1. Apply primer to tooth surface and leave in place for 20 s 2. Dry with air stream to evaporate the volatile ingredients 3. Apply bond to the tooth surface and then create a uniform film using a gentle air stream 0 4. Light polymerize for 10 s	
Single Bond Universal™	1. Etchant: 35% phosphoric acid (Scotchbond Etchant) 2. Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1. Apply the adhesive to the entire preparation with a microbrush and rub it in for 20 s. If necessary, rewet the disposable applicator during treatment 2. Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvent has evaporated completely 3. Light polymerize for 10 s	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry 2 s 4. Apply adhesive as for the self-etch mode

Table 2: Mean shear bond strength values recorded among the 4 groups.

Group	Mean	Standard deviation	SE of mean	95% CI for mean		Min	Max
				Lower bound	Upper bound		
Group I	16.06	0.54	0.14	15.77	16.35	14.88	16.88
Group II	13.94	0.39	0.10	13.73	14.14	13.32	14.65
Group III	13.86	0.52	0.13	13.58	14.13	13.14	14.71
Group IV	14.30	0.48	0.12	14.04	14.55	13.48	15.12

SE: Standard error, CI: Confidence interval

Table 3: ANOVA test for inter- and intra-group comparisons.

Source of variation	df	Sum of squares	Mean sum of squares	*F*	*P* value
Between groups	3	51.046	17.015	71.895	<0.001*
Within groups	60	14.200	0.237	-	-
Total	63	65.247	-	-	-

* p<0.001 indicates very strong evidence against the null hypothesis.

Groups I and II ($P < 0.001$), Groups I and III ($P < 0.001$), as well as between Groups I and IV ($P < 0.001$).

Discussion

An intimate bond between the adhesive and the substrate is crucial for adhesion. The overall bond strength of the resin composite to the dentin is the result of the intratubular penetration (resin tag formation) and penetration into partially demineralized dentin through hybrid layer formation. The biggest challenge for the hybrid layer and resin tag formation comes in the form of smear layer, which can be removed by acid etching the dentin: It not only removes the smear layer but also opens up the dentinal tubules, increases dentin permeability and decalcifies the intertubular and peritubular dentin.[10] If the dentin is air-dried thoroughly, as is advocated for the enamel, it leads to the collapse of the collagen fibril network, leading to an impairment in the formation of hybrid layer and ultimately a compromised bond strength.[9]

The technique sensitivity of etch-and-rinse adhesives is mostly related to the etching step itself and to the role of water in the bonding protocol. The concept of "dry bonding" advocated with the introduction of the etch-and-rinse adhesive systems in the 1970's was fraught with detrimental effects. Air-drying leads to collapse of the demineralized dentin with subsequent loss of the interfibrillar spaces between the collagen fibrils that served as inward diffusion channels for monomer infiltration.[5] This leads to compromised resin-dentin bond strengths of around 5 MPa when dry bonding is employed,[6] potentially culminating in dentin sensitivity, microleakage, secondary caries, and loss of bonded restorations. In 1991, Kanca found that water was an excellent rewetting agent, and this later led to advent of the "wet bonding" concept. This technique increased the resin-dentin bond strength, enabled good sealing of dentin and much less post-operative pain.[2,7]

A certain amount of water is crucial to prevent the collagen network from collapsing while an excessively wet surface may contribute negatively toward effective bonding to dentin.[5] Water-based primers have shown an ability to rehydrate or re-wet the air-dried and collapsed collagen network, enabling

Table 4: Bonferroni method for multiple comparisons.

(I) Group	(J) group	Mean difference (I-J)	SE of mean	*P* value	95% CI	
					Lower bound	Upper bound
Group I	Group II	2.120	0.172	<0.001*	1.651	2.589
	Group III	2.200	0.172	<0.001*	1.731	2.669
	Group IV	1.760	0.172	<0.001*	1.291	2.229
Group II	Group III	0.080	0.172	1.000	−0.389	0.549
	Group IV	−0.360	0.172	0.243	−0.829	0.109
Group III	Group IV	−0.440	0.172	0.078	−0.909	0.029

SE: Standard error, CI: Confidence interval, *p<0.001 indicates very strong evidence against the null hypothesis.

the hydrophilic primer monomers to interdiffuse. The remaining solvent should be air-thinned (evaporated) from the dentin surface by air-drying. Non-evaporated solvent may create voids in the adhesive interface, thus jeopardizing the polymerization of resin monomers.[5,9]

Of the various methods have been adopted by authors to evaluate the bond between the resin and enamel surface, such as fracture toughness, microleakage at the interface, tensile bond strength and shear bond strength,[11,12] the shear bond strength at the resin-dentin interface was assessed in this study, as shear stress is considered to be a more representative of the clinical situation in comparison to the tensile stress.[2,7]

Because the bond strength is also largely influenced by the composite used, the same resin composite (Filtek Z350, a nanocomposite) was used with all the adhesives so that this variable was excluded. The incremental placement technique has been advocated to reduce contraction stresses generated during polymerization of composite resin.[13,14]

The present study was done to evaluate the shear bond strength of the newly introduced multimode adhesive system: Single Bond Universal™, which can be used either as a total-etch, self-etch or selective-etch adhesive system; and compare it with the representative of two-step total-etch systems: Adper™ Easy Bond; and the self-etch system: Adper™ Single Bond 2.

In the present study, Group-I (Adper™Single Bond 2) showed the highest bond strength of 31.89 MPa. This can be due to the likelihood that: (1) Single bond 2 combines the functions of the primer and adhesive components of the conventional three-step adhesive system and has ethanol and water as solvents in its composition. The presence of ethanol in the adhesive increases the diffusion into the dentin, thereby enhancing the adhesion. The moisture of dentin tubules pulls the alcohol into and in between the tubules taking the resin with it. The ethanol and moisture then vaporize from the substrate leaving behind the resin.[15] (2) Single Bond 2 contains water, which may facilitate resin penetration into the collagen framework, regardless of the moisture condition of dentin. To achieve a good bond, it is important for the adhesive system to penetrate thoroughly into the demineralized dentin substrate.[16]

The exposed collagen may provide reactive groups that can chemically interact with bonding primers. The solvent used in Adper™ Single Bond 2 (total-etch/wet bonding) is ethanol, 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 dentin and the superficially demineralized dentin. These results also collaborate with the various results, in which wet bonding with adhesives has shown increased bond strengths than dry bonding.[12,17,18]

In light of the significance of collagen network in the bonding process, a recent study by Mohanbabu *et al.* (2015) concluded that the removal of dentin collagen fibrils by NaOCl application ("deproteinization") improves the marginal seal between resin and dentin when using acetone-based adhesive systems. The NaOCl treatment increases the surface roughness of dentin and its wettability by exposing a labyrinth of lateral secondary tubules, i.e., nanometric porosities of intertubular dentin. However, for the ethanol - water-based adhesive systems, this treatment did not improve the marginal seal between the adhesive and dentin.[19]

In the present study, Groups II and III (self-etch adhesives) and Group IV (total-etch adhesive; wet bonding) exhibit lower bond strength than Group I (total-etch adhesive; wet bonding). Adper™ Easy Bond (pH = 2.3) and Single Bond Universal™(when used in self-etch mode has a pH = 2.7) can be classified as mild self-etch adhesives. This mild self-etching action does not completely deprive the collagen fibrils of their hydroxyapatite content, which is in contrast to the total-etch systems. The residual hydroxyapatite may serve as a receptor for additional intermolecular interactions with specific carboxyl or phosphate groups of the functional monomers. Therefore, what ensues is a "chemo-mechanical" bonding mechanism.[12,20] On the downside, since the self-etching systems incorporate a significant amount of water as a solvent, these dental adhesives become 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.[18]

Conclusion

Within the limitations of the present study, the following conclusion can be drawn:

The total-etch adhesive system, Adper™ Single Bond 2 (Group I - Wet bonding), provided the highest shear bond strength values, followed by Single Bond Universal™ (Group IV - wet bonding). The latter performed better when compared to the same system under dry bonding conditions in the self-etch mode (Group III), as well as the self-etch system Adper™ Easy Bond (Group III - dry bonding).

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