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Original Article

Dentistry Section

Evaluation of the Shear Bond Strength of Composite Resin to Wet and Dry Enamel Using Dentin Bonding Agents Containing Various Solvents

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ABSTRACT

Introduction: Bonding of composite resin to dentin mandates a wet substrate whereas, enamel should be dry. This may not be easily achievable in intracoronal preparations where enamel and dentin are closely placed to each other. Therefore, Dentin Bonding Agents (DBA) are recommended for enamel and dentinal bonding, where enamel is also left moist. A research question was raised if the "enamel-only" preparations will also benefit from wet enamel bonding and contemporary DBA.

Aim: The aim of this study was to compare the shear bond strengths of composite resin, bonded to dry and wet enamel using fifth generation DBA (etch and rinse system) containing various solvents such as ethanol/water, acetone and ethanol.

Materials and Methods: The crowns of 120 maxillary premolars were split into buccal and lingual halves. They were randomly allocated into four groups of DBA: Group 1-water/ethanol based, Group 2-acetone based, Group 3-ethanol based, Group 4-universal bonding agent (control group). The buccal halves and lingual halves were bonded using the wet bonding and dry bonding technique respectively. After application of the DBAs and composite resin build up, shear bond strength testing was done.

Results: Group 1 (ethanol/water based ESPE 3M, Adper Single Bond) showed highest bond strength of (23.15 MPa) in dry enamel. Group 2 (acetone based Denstply, Prime and Bond NT, showed equal bond strength in wet and dry enamel condition (18.87 MPa and 18.02 MPa respectively).

Conclusion: Dry enamel bonding and ethanol/water based etch and rinse DBA can be recommended for "enamel-only" tooth preparations.

Keywords: Dry enamel bonding, Enamel-only, Wet bonding

INTRODUCTION

Predictable bond strength of 20-25 MPa can be achieved in bonding composite resin to acid-etched enamel surface [1]. Contamination of the surface lowers the bond strength by 20% to 100%; thus, moisture control is a mandatory part of enamel bonding [2]. Unlike enamel, dentin as a bonding substrate posed numerous challenges with regards to its mineral and water content [3]. Complete drying of the dentin resulted in poor bond strength with composite resin which led to the 'Wet bonding concept' where the dentin is intentionally left moist [4]. Generations of DBA evolved to achieve a predictable bonding to dentin [5]. The fifth generation DBAs are also known as etch and rinse systems, use the total etch and wet bonding concepts. Studies have shown that they provide better bonding than even the latest generation DBAs [6-9].

In intracoronal cavity preparations, where the enamel and dentin are closely placed to each other, it is clinically challenging to dry the enamel but keep only the dentin moist. Therefore, while using the contemporary DBAs, it is recommended that enamel is also kept moist. The clinical outcome of such intracoronal restorations might be majorly influenced by the bonding ability of larger surface of dentin than by the enamel's bonding. But some of the restorative procedures that involve only the enamel, such as pit and fissure sealant or diastema closure or direct veneering procedures, rely solely on enamel adhesion. Adopting the same wet bonding concept in these "enamel-only" preparations, eliminates the visualization of frosty white appearance after etching. This appearance has been the reliable indicator for proper etching of enamel. Consequently, there appears a lack of clinical validation of micromechanical retention that is generated from the enamel microporosities and also on thorough removal of the moisture from enamel crystallites.

Therefore, it becomes mandatory to assess and support this wet enamel bonding concept with robust scientific evidence. Current literature shows comparisons between the self-etch systems and etch and rinse systems on the moist enamel [10-13], and few studies have compared the influence of solvents in etch and rinse systems on moist enamel [14-17].

Thus, the aim of this study was to compare the shear bond strength of composite resin, bonded to enamel when dry and when moist, using fifth generation DBA (one bottle-two step etch and rinse system) containing various solvents such as ethanol/water, acetone and ethanol.

MATERIALS AND METHODS

Approval for this in vitro study was obtained from the Institutional Ethical Committee of Indira Gandhi Institute of Dental Sciences, Pondicherry, India. Patients in the age group of 18-25 years, who were undergoing maxillary premolar extraction for orthodontic reasons, were selected from the outpatient department of this institute. Informed consent was obtained from them. The extracted teeth were examined thoroughly. Teeth showing carious, non-carious, developmental defects and craze lines/fractures were excluded. A total of 120 premolars were selected, external debris was removed using ultrasonic scaler. The roots were removed and the crowns were split longitudinally in mesiodistal direction as buccal and lingual halves using diamond disc under running water mounted on a slow speed hand piece. The samples were randomly divided into four groups of 30 pairs each, using a simple lot method. An unfilled resin was used as the control.

The following fifth generation DBA (one bottle-two step etch and rinse system) were used: Group 1: (G1) used the water/ethanol based DBA. (Water- 5%, Ethanol- 25%) (3M ESPE-Adper Single Bond-USA)

Group 2: (G2) used the acetone based DBA. (Dentsply Prime and Bond NT-Australia)

Group 3: (G3) used the ethanol based DBA. (Ethanol- 25%) (Ivoclar-Tetric N bond-Liechtenstein)

The details of the DBA used in the study are shown in [Table/Fig-1].

The samples were embedded individually in acrylic resin blocks of (1x5 cm) exposing the buccal and lingual surface. The surfaces were flattened with silica carbide abrasives under running water to create flat surface of enamel. The smear layer that might be created on the surface of the enamel after abrasion were removed after acid etching as all the adhesives tested in this study were etch and rinse adhesive systems.

Adhesive tape with a circular window of 3 mm was cut using a punch holder. These cut adhesive tapes were pasted on the flat enamel surface exposing 3 mm diameter. Among the pairs, the buccal surface was used for wet bonding method and the lingual surface was used for dry bonding method. Acid etching was done with 37% phosphoric acid (N-etch-Ivoclar Vivadent) for 15 seconds. Surface was air dried thoroughly till a frosty white appearance was seen, for dry bonding. In wet bonding method, after the acid was rinsed off, the enamel was left moist and glistening. The surface was dried with gentle stream of oil-free compressed air for five seconds, from 10 cm distance, at 45° angle.

For all the four groups the DBA were applied using applicator tip as per the manufacturer's instruction. A transparent plastic tube of 3x2 mm dimension was placed perpendicularly over the enamel surface. The bonding agent was cured along with the transparent tube. Composite resin was condensed using a teflon coated plastic instrument into these tubes. A cover slip was placed on the top and the resin was polymerized in bulk with LED curing unit (L1330232, Gulin wood pecker, London, UK) with 800 mW/cm^2 intensity for 40 seconds. The composite was over cured as literature shows better microhardness which would help in shear bond strength testing without any adverse effects of over curing [18].

Trade Name	Brand Name	Lot No	Composition
ESPE Adper Single Bond	3M ESPE, USA	51202	Bis-GMA, HEMA, dimethacrylates, methacrylated poly- alkenoic acid, copolymer, initiators, water and ethanol
Prime and Bond NT	Dentsply Australia	052044	UDMA, PENTA, R5-62-1 resin, T-resin, D-resin, butylatedhydroxitoluene, EDMAB, cetylaminehydrofluoride, initia- tor, stabilizers, acetone and fumed silica nanofillers
Tetric N Bond	Ivoclar Liechtenstein	T08588	BIS-GMA, UDMA, dimethacrylates, HEAM, phosphonic acid acrylates, Sio_2, ethanol

[Table/Fig-1]: Dentin bonding materials used.

Groups	Dry/ Wet Enamel	Mean	SD	t	df	p
Group 1	Dry enamel	23.15	1.724	35.817	58	<0.001**
	Wet enamel	10.43	0.89747			
Group 2	Dry enamel	18.02	0.7501	4.29	58	0.8487
	Wet enamel	18.87	0.7774			
Group 3	Dry enamel	14.85	0.5335	2.3	58	0.7300
	Wet enamel	15.16	0.50009			

[Table/Fig-2]: Mean and SD of bond strength values in dry and wet enamel condition.
Student t-test** significant (p<0.001)

Testing of the shear bond strength was done at Bangalore Composite Technology Park, Bengaluru, Karnataka, India. Samples were subjected to shear bond strength test using universal testing machine (LR50K-LLOYD) at a cross head speed of 0.5 mm per minute until failure occurred. The point at which failure occurred was recorded in Newton and converted into MPa. The obtained data were tabulated.

STATISTICAL ANALYSIS

Statistical analyses were done using Graphpad InStat 3.ink. Student t-tests were done to study the effects of wet or dry enamel for each material. One-way analysis of variance (ANOVA) followed by post-hoc Tukey–Kramer multiple comparison test was used to study the effectiveness of dry and wet enamel condition for all groups.

RESULTS

Intra Group Analysis [Table/Fig-2]

Group 1, ethanol/water based DBA 3M ESPE-Adper Single Bond, showed mean shear bond strength of 23.15±1.72 MPa in dry enamel substrate followed by Group 2, acetone based DBA, Prime and Bond NT, and Group 3, ethanol based DBA, Tetric N Bond that showed 18.02±0.75 and 14.85±0.53 MPa respectively. In wet enamel bonding, acetone based DBA showed the highest mean shear bond strength of 18.87±0.78 MPa. This was followed by ethanol based DBA that showed bond strength values of 15.16±0.50 MPa.

The acetone based and the ethanol based DBA showed better bond strength values in wet enamel substrate than in dry. However, this difference was not statistically significant.

The water/ethanol based DBA showed better bond strength in dry enamel than in wet enamel. The difference between dry and wet is statistically significant (p<0.001).

Inter Group Analysis [Table/Fig-3,4]

A one-way analysis of variance (ANOVA) was used to determine if there was difference between materials, bonded to wet or dry enamel surface. As ANOVA proved significant, Tukey-Kramer multiple comparison tests were also done. A highly significant difference is observed in the mean values in dry enamel bonding among the four groups (p<0.001). Group 1 showed the maximum value in dry enamel compared to all the other DBAs. Tukey-Kramer multiple comparison test (p<0.001) showed a significant difference between Group 1 and all the other groups.

A highly significant difference is observed in the mean values in wet enamel condition among the four groups (p<0.001). Group

	Bond Strength From Dry Enamel		ANOVA	Bond Strength From Wet Enamel		ANOVA
	Mean	SD		Mean	SD	
Group 1	23.15	1.72	763.86**	10.43	0.90	1090.00**
Group 2	18.02	0.75		18.87	0.78	
Group 3	14.85	0.53		15.16	0.50	

[Table/Fig-3]: One-way ANOVA for dry enamel bonding and wet enamel bonding.
(**p<0.001)

Comparison	Dry Enamel Bond Strength		Wet Enamel Bond Strength		p
	Mean difference	q	Mean difference	q	
Group 1 vs Group 2	5.126	25.430	-8.433	57.054	***p<0.001
Group 1 vs Group 3	8.296	41.148	-4.728	31.987	***p<0.001
Group 2 vs Group 3	3.168	15.718	3.705	25.066	***p<0.001

[Table/Fig-4]: Tukey multiple comparison test in between groups for dry and wet enamel bonding.
***Significant p<0.001

2 showed the highest shear bond strength values than the other groups. Tukey-Kramer multiple comparison test (p<0.001) showed a significant difference between Group 2 and all the other groups, in this substrate.

DISCUSSION

The one-bottle, two step etch and rinse adhesives are DBAs that contain hydrophilic monomers, hydrophobic monomers and initiators dissolved in volatile organic solvents and water. The main action of the solvents is to carry the monomer into the dentinal substrate. By reducing the viscosity of the monomer, they enable better wetting and penetration into the dentin. They also displace the moisture content in the dentin as the monomer enters the demineralized dentin. The physical properties of the solvents such as the solubility parameter, vapour pressure and the hydrogen bonding capacity influence the behaviours of bonding to dentin. Three most commonly used solvents in DBAs are acetone, ethanol and water [19].

In this study ethanol, acetone and ethanol/water based DBA were used on dry or moist enamel. The results of the study show that the Group 1, ethanol/water based adhesives provided maximum bond strength of 23.15 MPa in dry etched enamel, amongst all the groups, This value is the closest to the optimal bond strength of resin to enamel (20-25 MPa). The 3M ESPE-Adper Single Bond used in this group has water as a co-solvent along with ethanol. This part of the result is similar to a study by Swift EJ et al., where one bottle adhesives with various solvents were tested on air dried etched enamel [20]. The authors had reported 27.8 MPA bond strength for single bond to dry etched enamel, which was higher than the acetone based or water based adhesives.

Both ethanol and water have good hydrogen bonding capacity. This capacity enables these solvents to penetrate and re-expand the collapsed demineralized dentin collagen and therefore, are recommended to be applied over dry demineralized dentin substrate [19]. This wettability can be the reason for single bond performing well over the dry enamel in the present study.

However, the above DBA had shown poor bond strength in wet enamel, in this study. This is in contradiction to many studies where it has been reported to generate similar bond strength in the wet and dry enamel [11,15-17]. This can be attributed to the drying method that has been adopted in the current study. In wet enamel bonding procedure, the enamel was air dried till it was left glistening and moist. A recent study in 2016 [21] compared the air-drying method and the blot drying method with total etch systems and has concluded that the blot drying method is more controlled than the air drying, that can leave the substrate optimally wet. Similar study was done in 2010 which reported that blot drying showed consistently better bond strength [22]. The variability associated with air drying used in the present study could have left the enamel substrate too moist, resulting in over-wetting and compromising the bond to enamel in a similar fashion as reported on dentin substrates [23]. In addition, the presence of 5% water in single bond could have added to this over-wetness. Lesser vapour pressure for ethanol and water, compared to the other organic solvents, could have collectively contributed to the ineffectiveness of this bonding agent to remove the residual moisture from the enamel substrate [19].

The results of this study show that the acetone-based DBA, exhibited highest shear bond strength of 18.9 MPa in wet enamel among all the other groups. But there was no significant difference in its bond to dry enamel. This is in accordance to previous studies where acetone based DBA was found not to be affected by moisture in enamel [11,14-17,24].

Acetone has the highest vapour pressure among all the solvents that effectively removes water from demineralized dentin matrix. It is less viscous than ethanol and can better penetrate through demineralized dentin and enamel. But it has low hydrogen bonding capacity, so might not be suitable on dry dentin, where it is expected to re-expand the collagen fibrils [19]. But in enamel bonding, the main issue seems to be surface wetting and removal of residual moisture from the enamel. Etched enamel binds water effectively and firmly due to its high surface energy, thus, removal of moisture requires a solvent with high vapour pressure [25]. As per the results of the present study and the previous studies, it is evident that acetone performs this effectively, irrespective of the amount of water present on the enamel surface.

It should be noted that among the ethanol/water based and the acetone based DBA used in this study, the highest bond strength was achieved with ESPE 3M Adper Single Bond on dry enamel, than Prime and Bond NT on moist enamel. The reason for reduced bond strength values of acetone based DBA can be attributed to the nanofiller content in Prime and Bond NT. Nanofiller are added with the intention of improving the strength of the hybrid layer and thus the bond strength. The fillers in Prime and Bond NT are in the range of 7 nm and has been stated that they tend to agglomerate to larger clusters, which can inhibit the proper penetration of the resin into dentin [26]. A similar behaviour in enamel substrate can be the reason for the results observed in the current study. In addition, the high vapour pressure of acetone can result in its evaporation during usage or storage, resulting in increased viscosity, which in turn could have affected the penetration of the resin [27].

The DBA used in the current study as Group 3 is Tetric N bond, which is ethanol based. Though this contains ethanol as solvent, it exhibited the least bond strength among the other two experimental groups. It has been stated that a solvent should have more hydrogen bonding capacity than the hydrogen bonding capacity of the demineralized collagen in dentin, in order to re-expand the collagen. Water is added as a co-solvent to improve this aspect [19]. However, Tetric-N-bond had no water as co-solvent. Whether this reason is applicable to etched enamel substrate also and can be attributed for the poor performance of Tetric-N-bond in this study is debatable.

LIMITATION

The research question in this study was raised with "enamel-only" preparation in focus. In these preparations, usually the enamel is intact and uncut. However, in the present study the enamel surface was abraded to create a flat surface to standardize the area for resin bonding. This could have influenced the results of the study. Studies that have tested bond strength on bur cut or abraded enamel surface have shown better bond strength than uncut enamel [10,28]. Yet, another limitation is that the water-based solvent was not tested in this study which could have added more evidence to effect of solvent on bond strength.

In addition, evidence is cripplingly depleted about ultrastructural analysis of interface morphology between wet/dry enamel and the DBA. Therefore, most of the reasoning for enamel's behaviour to DBA has been extrapolated from the abundant evidence available from dentinal bonding. Further studies are needed in this lacuna to observe if enamel substrate behaves a kin to dentin with the contemporary DBA.

CONCLUSION

DBAs are materials evolved to combat the challenges posed by the hydrophilic dentin to the hydrophobic resin. Using this material over enamel substrate, for the purpose of technical practicality might be acceptable for intracoronal cavity preparations. But abundant evidence is already available for reliable bonding of resin to acid-etched, dried, frosty white appearance enamel. Leaving the enamel moist for receiving these contemporary DBAs tend to leave an element of doubt, if the enamel is adequately moist or overly wet.

In this study, among the two-step, one bottle adhesives with various solvents, ESPE 3M Adper Single Bond with ethanol/water solvent shows better bond strength on the predictable and reliable etched and dried enamel surface. Thus, it can be recommended to use dry enamel bonding and ethanol/water based etch and rinse systems for "enamel-only" tooth preparations.

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