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Effect Of Solvent Type On Microtensile Bond Strength Of Total-Etch One-Bottle Adhesive System To Moist Or Dry Dentin

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Abstract: *The purpose of this study is to evaluate the effect of various solvents namely ethanol, acetone and ethanol/water on the microtensile bond strength of one bottle, total etch adhesive systems applied to dry or moist dentin.*

Material and Methods: *Forty eight non carious human upper first molars with roots removed and randomly divided into three groups of sixteen specimens each based on the bonding agents with different solvents were used in this study..Each group was further divided into subgroup A and B, with eight specimens each based on the moist or dry dentin surface treatment. Microtensile bond strength was checked using LLOYD - universal testing machine.*

Results: *Moist bonding technique produced higher values in all the three groups. Ethanol/water based adhesives performed better when compared to acetone and ethanol based adhesives .Ethanol and acetone based adhesives performed almost in a similar manner in dry & moist conditions.*

Conclusion: *Moist bonding produced superior values for all the three types of adhesives and Ethanol/water based adhesives produced the maximum bond strength values in both moist and dry bonding conditions.*

Keywords: *Adhesives, dry bonding, microtensile bond strength, moist bonding, vapour pressure*

I. Introduction

Adhesive restorations have been increasingly used in restorative dentistry primarily because of their potential to allow conservative preparation designs and for esthetics. Adhesion of resin composite to the tooth being restored is required to provide retention and ensure marginal integrity and durability of the restoration. Bonding of resin composite to enamel has been quite a straight forward and reliable task since the introduction of phosphoric acid etch technique by Buonocore in 1955, On the other hand, bonding to dentin has proved to be more difficult and less predictable, mainly because of its morphologically heterogeneous and physiologically dynamic structure.

The fundamental principle of adhesion to tooth substrate is based upon an exchange process by which inorganic tooth material is exchanged for synthetic resin (Van Meerbeek 2001). This exchange process involves two phases. The first phase involves removal of inorganic components to create micro porosities at the exposed enamel & dentin surfaces. The second phase is the `hybridization phase' to form a hybrid layer which involves infiltration and subsequent `in situ' polymerization of resin within the created surface micro porosities. This results in micromechanical interlocking which is primarily based on mechanisms of diffusion. The hydrophilic monomers and the organic solvents which help in diffusion are added to the primer and adhesives. The 3 major solvents employed in primers are acetone, alcohol & water. These solvents determine which clinical method - either `moist bonding' as advocated by Kanca & Gwinnett in 1992 or `dry bonding' to be employed to achieve adequate hybridization and to provide predictable bond strength values.

The rationale behind bond strength testing is that the higher the actual bonding capacity of an adhesive the better it will withstand stresses and longer the restoration will survive in vivo. The bond strength values are determined by subjecting the composites bonded to tooth substrate to tensile or shear stress. This study attempts to evaluate the effect of various solvents namely, ethanol, acetone and ethanol/water on the microtensile bond strength of one bottle, total etch adhesive systems applied to dry or moist dentin.

II. Materials & Methods

Forty eight non carious human upper first molars freshly extracted for periodontal reasons and stored in saline were utilized for this study. After cleaning all the teeth, the roots were removed. The occlusal surface was

ground under running water to expose a flat dentin surface parallel to the occlusal surface. The flat dentin bonding surface was prepared by polishing with a series of wet silicon carbide paper (220, 360 and 600 grit). The 48 teeth were randomly divided into 3 groups of 16 specimens each. The groups were divided based on the bonding agents with different solvents used in this study.

Group I is Gluma Comfort Bond (Kulzer) which is ethanol based adhesive, Group II is Prime & Bond NT (Dentsply) which is acetone based adhesive, Group III is Single bond (3M ESPE) which is Ethanol & Water based adhesive. The dentin surfaces of all the teeth were etched with 37 % phosphoric acid gel for 15 seconds and rinsed with water for 20 seconds. After this procedure, each group was further divided into subgroup A and B, of 8 specimens each based on the moist or dry dentin surface treatment.

In Sub Group A, the etched and rinsed dentin was partially dried by blotting the excess water with a small piece of absorbent paper, leaving a visibly moist surface (Wet bonding technique). In Subgroup B, the etched and rinsed dentin was dried for 10 seconds with an air syringe at a distance of approximately 2 cm from the surface (dry bonding technique). After completing the dentin surface treatment procedures for the moist & dry subgroups, the bonding agents were applied as per manufacturer's instructions. The composition of the bonding agents selected for this study is given in the Table 1.Hilux light curing unit was used in this study. The intensity of the light was checked after curing each specimen.

In group 1, the bonding agent was applied in two consecutive coats over the prepared dentin surface with a saturated disposable brush, it was lightly dried for two seconds to evaporate the solvent and the bonding agent was light cured for 20 seconds. In group 2, generous amount of the adhesive was applied thoroughly to wet the prepared dentin surface using an applicator. The specimen was left undisturbed for 20 seconds. It was air dried for 5 seconds, so that a uniform glossy appearance is seen. The bonding agent was light cured for 10 seconds. In group 3, two consecutive coats of bonding agents was applied thoroughly to the specimen with a fully saturated brush. It was air dried for 3 to 5 seconds. The bonding agent was light cured for 10 seconds.

All the bonded surfaces of the 48 teeth were restored with hybrid resin composite (Solare, GC) of 2 mm increments to a height of 4 to 5 mm. Each layer was light cured for 20 seconds. All the specimens were stored in water at room temperature for 24 hrs before being sectioned. The restored teeth were attached to a cutting machine (Isomet - model 650, South Bay technology, Sun Clemente, CA, USA) where a diamond disc running at a slow speed with water coolant was used to section the specimens into buccolingual parallel slabs of 0.7mm thick Fig1. New sections of 0.7 mm thickness were cut in each slice perpendicular to the first section and the slabs were converted into sticks. A total of 10 sticks specimens were prepared for each subgroup. All the prepared sticks were stored in water for 24 hrs at room temperature before evaluating the microtensile bond strength (MTBS) measurements.

Each specimen was then glued to metal heads of long screws with cyanoacrylate adhesive and stressed in tension in a LLOYD - universal testing machine, operating at a speed of 0.5mm / minute until failure . The MTBS values were calculated as the maximum load at failure divided by the bonded cross sectional area and expressed in MPa. The results were then statistically evaluated using a One way ANOVA (Independent Variable: solvent type & dentin moisture; Outcome variable: MTBS) and Post hoc (Scheffe test) method.

2.1 Microtensile Bond Strength Test

Traditional bond strength testing methods tend to use large surface areas in the order of 7-12 mm^2 and fractures in these larger surface area specimens frequently occur cohesively in dentin. This form of failure does not provide reliable information with regard to adhesive strength of the bond.

Sano et al (1994) introduced a **micro tensile** test method that used a bonded surface area of approximately 1mm^2 obtained from the experimental tooth through a series of cross and longitudinal sections. These sections can be in the form of hourglass shape or dumb bell shape or in the form of beam shaped sticks [1] .One half of the substrates consists of dental tissue and the other half contains the restorative material. The two substrate are held together by an adhesive system at an interface, which has a very small cross sectional area of 0.5 to 1.5mm^2 depending on the technique [2].

Creating the hourglass shape at the bonding interface with burs is more prone to cause premature failures. Alternatively, the non trimming - technique (Shono et al) used in this study, leaves the micro tensile specimens with a beam shape, which is less traumatic and can be used to measure bond strengths as low as 5 MPa. The graphic representation of preparation of molar and procedural sequence is given in Fig 2 and 3 respectively.

III. Result

Moist bonding technique produced higher values in all the 3 groups than dry bonding. Ethanol/water based adhesives performed better with higher bond strength values, when compared to acetone and ethanol based adhesives. Ethanol and acetone based adhesives performed almost in a similar manner in dry & moist conditions.

IV. Statastical Analysis

Statistical analysis was done to compare the values within the group and among the groups using one-way analysis of variance and post hoc (Scheffe test) method shown in Table 2 and Table 3.

Comparing within the groups, Group III produced statistically significant higher bond strength values than groups I and II. Group I produced better values when compared to Group II, but no statistically significant difference was noted between those groups.

Comparing within the subgroups, In Subgroup A, bond strength values were maximum for Group III, followed by Group I & II. In Subgroup B, Group III performed better than groups I and II. No statistical difference was noted in values obtained for Groups I & II. Graphical representation is given in Fig 4.

V. Discussion

Dentin bonding can be described as a " hydro dynamic micromechanical bonding [3]. This approach involves penetration of resins into partially demineralised dentin and intratubular dentin and their polymerization in situ. The overall bond strength of resin composite to dentin can be regarded as a summation of the individual bond strengths provided by intratubular penetration (resin tag formation) and penetration into partially demineralised intertubular dentin (hybrid layer formation). For the complete infiltration of the demineralised hydrophilic dentin by the hydrophobic adhesive resin, a primer plays the key role favouring diffusion. A primer is basically a bi functional monomer dissolved in a solvent such as alcohol, acetone or water. A bifunctional monomer is one that has a hydrophilic end and a hydrophobic end. (Causton 1982).

A balance is struck between wet and dry dentin, as a result, moisture is preserved on the surface and inside the collagen web & in the dentinal tubules. This state of dentin is achieved by gentle air drying or blotting the surface and leaving it visibly moist[4].This technique of `moist bonding' needs a system that could compete with moisture and replace the same, while taking the monomer along with it and then evaporate. This is possible by using high vapour pressure solvents like acetone and ethanol. Also dried dentin matrix can be rewetted or rehydrated by using water based adhesives.

In this study, 3 different bonding agents based on 3 different solvents have been evaluated for their effect on the MTBS to moist and dry dentin. The results of this study indicate that Group III (ethanol/water) adhesives produced significantly higher bond strength values than groups I & II. In all the groups, at the subgroup level, moist bonding produced better results than dry bonding. The results of this study can be interpreted based on 3 important properties namely molecular size of the solvent, solubility parameter for hydrogen bonding and vapour pressure of the solvent. The performance of various solvents/monomers has been rated based on their ability to solvate a dried, shrunken, demineralised dentin matrix. Presumably the rate of solvation would depend upon the molecular size of the solvents and their solubility parameter for hydrogen bonding [5].

According to Pashley, only solvents with **Hansen's solubility parameter for hydrogen bonding (δh)** higher than 19.0 $(J/cm^3)^{½}$ are capable of breaking the interpeptide H-bonds between the collagen fibrils and re-expanding the matrix. In other words, for solvents like ethanol and acetone, to break the interpeptide H- bonds, they must have a δh that is greater than the attractive H-bonding forces of the peptides for each other. The presence of water in Group III adhesives probably resulted in a mixture with a δh higher than 19 that allowed the partial expansion of the collapsed matrix, resulting in better resin infiltration and hence improved bond strength [6] .The same phenomenon did not occur with acetone based adhesives because of its lower δh due to presence of acetone and absence of water [7,8]. Thus the bigger molecular size and low δh of acetone can be attributed to the poor performance of Group II when compared to Group I and III.

Also, the presence of moisture, which is considered to be a prerequisite to keep the interfibrillar nanospaces open for resin permeation makes the acetone based adhesives more technique sensitive, favorable solvent option[9].In case of Group I adhesives the factors namely solubility parameter, molecular size and vapour pressure are all in favour to produce better bond strength values when compared to Group II. In case of Group III apart from the presence of water as a constituent, its superior performance over Group I can also be attributed to its polyalkenoic acid content [10]. The polyalkenoic acid base complexes (Polyalkenoic acid + Ca^{++} of the hydroxyapatite) may have an intrinsic stress relaxation capacity and resistance to degradation in a humid environment. Furthermore, the inherent content of water as a constituent reduces the impact of the moisture condition of dentin on Group III adhesives [11].

The optimal water concentration that is required for the re-expansion of the collapsed collagen network might be somewhere between 9% and 50% [11]. The water concentration of 3% to 8% of group III adhesives may not be sufficient for complete penetration of the adhesive into the dried demineralized dentin. This can be accounted as a possible explanation for the bond strengths of Group III B (dry) being less than Group III A (moist) [13].

From the above results and discussion, we infer that moist bonding produced better results for all 3 types of adhesives when compared to dry bonding. Also ethanol/water based adhesives performed better than acetone and ethanol based adhesives. So it can be concluded that simultaneous inclusion of a high vapour pressure solvent and

water in an adhesive system (as in Group III) may be fundamental for adequate infiltration and a less technique sensitive procedure to produce high bond strengths.

VI. Summary

Superficial occlusal dentin of 48 noncarious freshly extracted human upper 1st molars was used for this study. The 48 teeth were randomly divided into 3 groups depending upon the bonding agents with 3 different solvents.

Group I - Ethanol based
Group II - Acetone based
Group III - Ethanol/water based

The dentin surface of all the teeth were etched with 37% phosphoric acid gel for 15 seconds and rinsed. Each group was then further subdivided into two subgroups - A & B.

Subgroup A - moist bonding
Subgroup B - dry bonding

Moist bonding technique was adopted by using absorbent paper to remove excess water and to leave the surface visibly moist. Dry bonding was done by drying the etched surface with air syringe at a distance of 2 cms for 10 secs. Bonding agent was applied to all the 16 teeth of each group as per manufacturer's instructions and the surfaces were then restored with 4-5mm thick hybrid resin composite.

The specimens were sliced using a slow speed diamond saw into sticks of 0.7 mm x 0.7 mm dimensions. The prepared sticks were then subjected to tensile testing in an universal testing machine. Microtensile bond strength was calculated for each group, results tabulated and values were statistically analyzed and discussed.

VII. Conclusion

From the study, it was concluded that

1. Moist bonding produced superior values for all the 3 types of adhesives when compared to dry bonding revealing that water plays a crucial role in bonding.
2. Ethanol/water based adhesives produced the maximum bond strength values in both moist and dry bonding conditions.
3. Ethanol/water based adhesives performed better than plain ethanol based adhesives revealing the importance of water in bonding.
4. Acetone and ethanol based adhesives performed almost similarly in moist & dry conditions, with higher values in moist bonding state.

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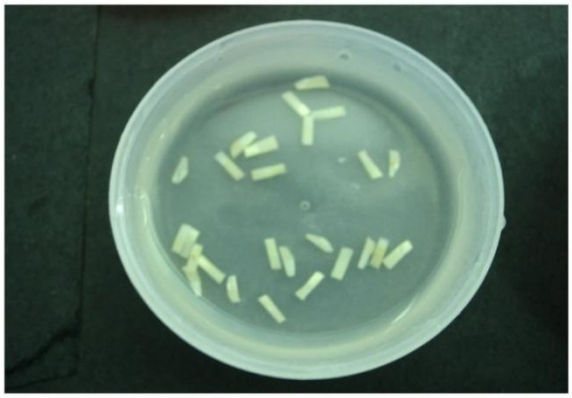
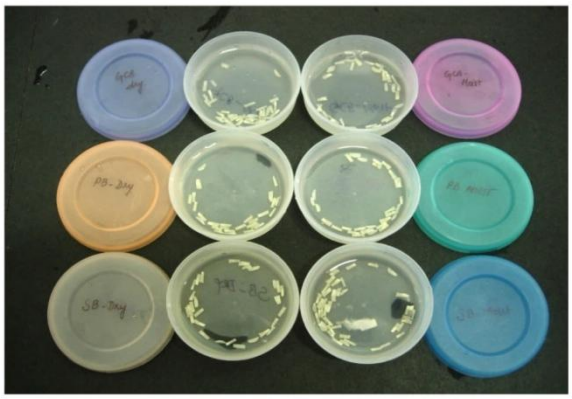


Prepared specimen of 0.7 X 0.7 mm dimension



Prepared specimen of all the three groups

Fig 3 Specimen sectioned buccolingually 0.7 thickness in dimension

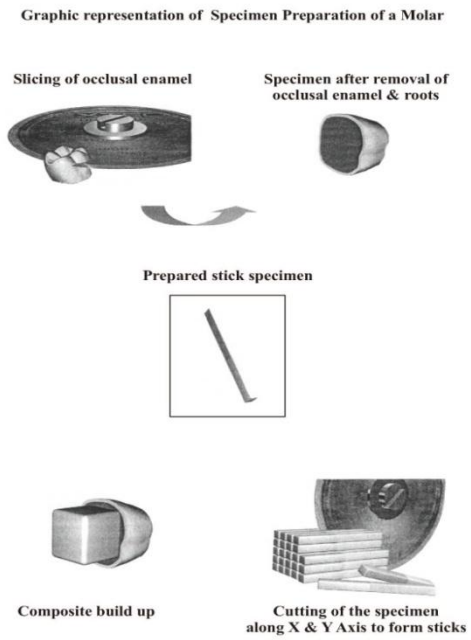


Fig 5 Graphic representation of preparation of molar.

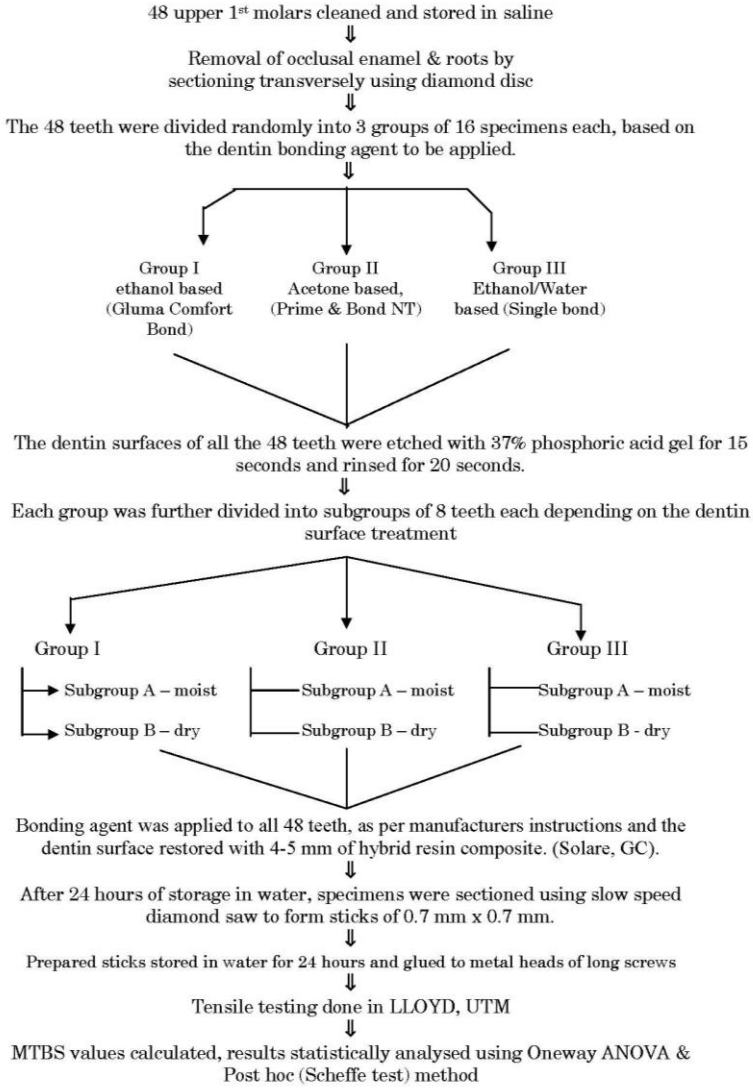


Fig 6 Procedural sequence

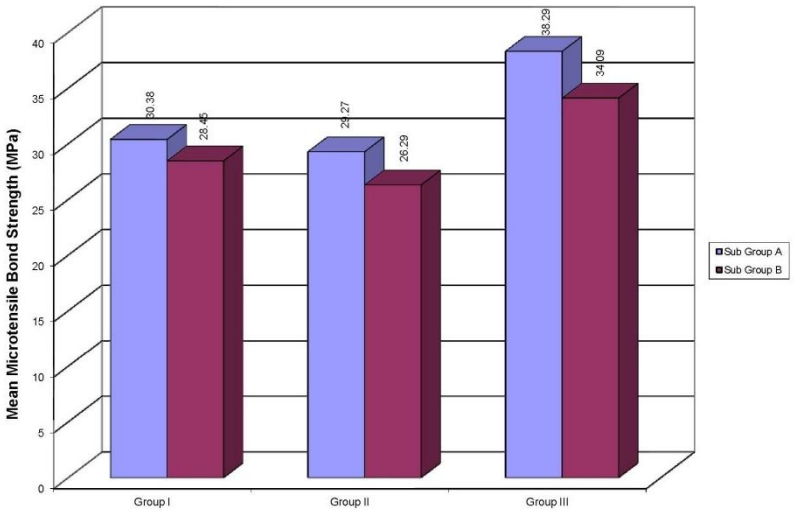


Fig 7 Microtensile bond strength in groups and subgroups.

Table 1

Group	Bonding agent	Composition	Solvent	Manufacturer
I	Gluma Comfort Bond	UDMA, HEMA, 4-META, Polyacrylic acid and dicarboxylic acids	Ethanol	Heraeus Kulzer
II	Prime & Bond NT	Di & trimethacrylate resins, PENTA, nano-filler-amorphous silica, photoinitiators, stabilizers, cetylamine hydrofluoride	Acetone	Dentsply
III	Single Bond	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer	Ethanol & Water	3M (ESPE)

Table 5 Mean and standard deviation , ***** -denotes significance at 5% level, ****-** denotes significance at 1% level, different alphabets between groups denote significance at 5% level .

	GROUP	SUBGROUP-A		SUBGROUP-B		P Value between subgroups
		MEAN	STANDARD DEVIATION	*MEAN*	STANDARD DEVIATION	
1.	Group – I	30.38[a]	1.82	28.45[a]	1.56	0.020[*]
2.	Group – II	29.27[a]	2.19	26.29[a]	2.39	0.009[**]
3.	Group – III	38.29[b]	2.63 2.63	34.09[b]	3.27	0.005[**]
	P value between groups	< 0.001[**]		< 0.001[**]		

Table 6 Comparison of microtensile bond strength values

	Group IA	Group I B	Group II A	Group II B	Group III A	Group IV B
No. of samples	10	10	10	10	10	10
Minimum value (MPa)	28.03	26.00	25.00	22.32	34.00	30.26
Maximum Value (MPa)	32.65	31.05	32.55	29.58	42.30	38.25
Mean (MPa)	30.38	28.25	29.27	26.29	38.29	34.09
Standard deviation	1.82	1.56	2.19	2.39	2.63	3.27