

An In-vitro Comparative Evaluation of Shear Bond Strength of Different Self-Etch Dentin Bonding Agents

Ruhani Cheema¹, Ekta Choudhary²

ABSTRACT

Introduction: Adhesive procedures have evolved rapidly and have become routine in the daily practice of dentistry. Aim of the study was to examine the in vitro shear bond strengths to dentin of two one-step self-etch bonding systems and a two step self-etch bonding system.

Materials and method: The current study examined in vitro shear bond strengths to dentin of two one-step self-etch bonding systems and two step self-etch bonding system. 90 extracted non-carious permanent human premolars were used. The teeth were stored in 1% chloramine T solution at 4°C. The occlusal surfaces were trimmed with tungsten carbide bur and finished with 600-grit silicone-carbide discs. Three adhesive systems used: Clearfil SE bond (Kuraray) (two-step self-etch), Single Bond Universal (3M ESPE) (one-step self-etch) and Xeno V+ (Dentsply) (one-step self-etch). Filtek Z350 composite were bonded to the teeth. The specimens were stored at 37°C for 24 hours and were tested on the universal testing machine with a crosshead speed of 1 mm/min. Shear force required to debond the specimen was recorded.

Results: ANOVA test was used to compare statistical difference of shear bond strength between the groups (*P* value less than 0.05 was considered to be statistically significant). Multigroup comparison was done using Tukeys HSD test. There was a significant difference in the shear bond strengths among bonding systems. Clearfil SE reported a shear bond strength value of 36.6 MPa followed by single bond universal 27.29 MPa and Xeno V+ 25.1 MPa.

Conclusion: Clearfil SE delivered the highest shear bond strength value followed by single bond universal and Xeno V+. Two step self-etch recorded higher shear bond strength values to one-step.

Keywords: Dental bonding, Single step, Bond strength

INTRODUCTION

Adhesive dentistry has revolutionized dentistry. The major determinant of successful esthetic dentistry remains the effective adhesion between the substitute and the restorative material.

Adhesive systems and bonding techniques have been constantly evolving since the introduction of Sevriton Cavity Seal, in the late 1940, by Oskar Hagger.¹ In 1955, Buonocore reported the use of 85% phosphoric acid to improve retention of an acrylic resin on enamel.² Bonding to enamel revolutionized the practice of restorative dentistry and has proven to be durable.

Dentin is a dynamic substrate and its morphology and physiology directly affect the ability of adhesive systems to produce durable bonds to its prepared surfaces. Dentin is a heterogeneous structure as it is a living tissue with fluid

filled channels that run from the pulp to the dentino-enamel junction (DEJ). While enamel is 96% hydroxyapatite (mineral) by weight while dentin is approximately 75% inorganic material (mineral), 20% organic material (mainly Type I collagen) and 5% water. It is subjected to continuous physiologic and pathologic changes affecting its microstructure, composition and permeability.^{3,4} The number of tubules is 45,000/mm² at pulpal side; 35,000/mm² 1 mm from the pulp; 23,000/mm² 2mm from the pulp and 19,000/mm² subjacent to amelodentinal junction.⁵ Therefore, bonding to dentin represents a challenging substrate for bonding.

Currently available resin-based adhesives may be divided into two major categories, self-etch and etch-and-rinse, based on the number of clinical steps required for each and their respective interactions with the tooth surface.⁶

Largely because of this continuing problem with total-etch adhesives, much of the current product development and clinician interest is focused on self-etching systems. The original self-etch systems included two steps—an acidic, self-etching primer followed by a separate bonding resin. Some of the newer systems are considered all-in-one, and contain etch, prime and bond functions in a single solution. The former group of materials can be described as self-etch primer systems, and the latter can be called self-etch adhesives.⁷ Self-etch adhesive incorporates classic steps of etching, priming and bonding into one solution.

MATERIALS AND METHOD

Type of Study: It is an in-vitro study with a sample size of 90 permanent human maxillary premolar.

Materials used in the study (Fig. 1)

1. 1% Chloramine T
2. Cold cure acrylic resin
3. Xeno V+
4. Single bond universal
5. Clearfil SE Bond 2
6. Z 350 XT Composite resin system

¹Final Year Post Graduate Student, ²Professor, Head of the Department of Conservative Dentistry and Endodontics, School of Dental Sciences, Sharda University, Greater Noida, U.P., India

Corresponding author: Dr. Ruhani Cheema, Sharda University, Greater Noida, U.P., India

How to cite this article: Ruhani Cheema, Ekta Choudhary. An in-vitro comparative evaluation of shear bond strength of different self-etch dentin bonding agents. International Journal of Contemporary Medical Research 2016;3(2):473-478.

the unit's integrated light meter. After polymerization, all the specimens were transferred to distilled water, and stored at 37°C for 24 hours in incubator (Remi Instruments Ltd, Gurgaon, India).

(d) Shear bond strength testing

After storage, specimens were mounted on the universal testing machine (Banbros, Ghaziabad, India) force applied by the machine on each specimen was at a crosshead speed of 1 mm/min using a notch-edge blade parallel to the adhesive-dentin interface. The bonded composite cylinder was positioned horizontally, so that the shearing blade is perpendicular at composite-dentin interface. Each specimen was loaded until failure (Fig. 4). Debonding stress in megapascal was then

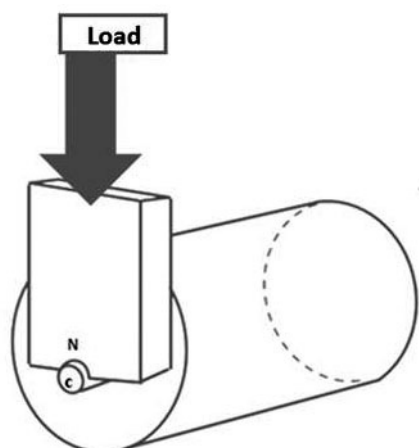


Figure-4: Schematic diagram of a shear test using a notched-edge

calculated by the ratio of maximum load in Newton to the surface area of prepared resin cylinder.

STATISTICAL ANALYSIS

In analysing the results of the variables under various methods considered in this research work, the statistical analysis like arithmetic mean, standard deviation and one way analysis of variance (abbreviated ANOVA) were used appropriately.

$$1. \text{ Shear bond strength (MPa)} = \frac{\text{Force (N)}}{\text{Cross sectional area (mm}^2\text{)}}$$

$$2. \text{ Arithmetic mean } x = \frac{1 \sum X}{n}$$

Where,

$\sum X$ is sum of all data values

n is number of data items per sample

$$3. \text{ Standard Error} = \frac{SD}{\sqrt{n}}$$

$$4. \text{ Post hoc =HSD} = q \sqrt{MSE / n^*}$$

where q = the relevant critical value of the studentized range statistic

n^* is the number of scores used in calculating the group means of interest.

Statistical analysis was performed using SPSS 17.0 (Chicago III) for analysis of data and Microsoft Word and Excel have been used to generate graphs, tables, etc. ANOVA test was used to compare statistical difference of shear bond strength between the groups (p value less than 0.05 was considered to be statistically significant). Multigroup comparison was done using Tukeys HSD test.

Adhesive	Composition	Manufacturer	Lot	Manufacturer's Application Protocol
Clearfil SE Bond	Primer: MDP, HEMA, hydrophilic aliphatic dimethacrylate, CQ, ethyl alcohol, water Bond: MDP, HEMA, Hydrophobic dimethacrylate, CQ, silanated colloidal silica pH=2.7	Kuraray Noritake Dental Inc., Japan	Primer (AD0112) Bond (AE0180)	Primer was applied to the entire tooth with a brush, left in place for 20 s and finally volatile ingredients were evaporated for 10 s. Bond was applied with a brush, dispersed with a very gentle stream of air and polymerised for 10s
Single Bond Universal	MDP phosphate monomer, dimethacrylates resins, HEMA, vitrebond copolymer, filler, ethanol, water, initiators, silane, pH ~ 2.7	3M ESPE, St. Paul, MN, USA	532119	Adhesive was applied to dentin with a brush and gently agitated for 20 s, then direct a gentle stream of air for 5 s until the adhesive no longer moves and polymerized for 10 s
Xeno V ⁺	Bifunctional acrylamides, ethyl 2-(5-dihydrogen phosphoryl-5,2-dioxapentyl) acrylate, acrylamide 2-methylpropanol-2 sulfonic acid, t-butanol, water pH ~ 1.3	Dentsply Detrey Konstanz, Germany	1311000781	Adhesive was applied to dentin and rub ifor 20 seconds, dried with a gentle stream of air for 5 seconds, and polymerized for 10 seconds.
Filtek™ Z350 XT Universal restorative	Bis-gma, UDMA and Bis-EMA. Additional contents: stabilizers, catalysts and pigments.	3M ESPE Dental Products, St. Paul, MN, USA	N 633113	Place Filtek Z 350 –shade A2. Light-cured each increment of 1.5mm for 40 s

Table-1: Materials used in the study with their compositions, manufacturers and application procedures

RESULTS

The results can be summarized as follows (Table 2, Graph 1 and 2):

Group 1 demonstrated the highest shear bond strength value of 36.6 MPa while group 3 demonstrated least shear bond strength 25.1 MPa. Intergroup comparison done between the group 1 and group 2 showed statistically significant result ($P < 0.05$). Intergroup comparison done between group 2 and 3 showed no statistically significant results ($P > 0.05$). Intergroup comparison done between the group 1 and group 3 showed statistically significant result ($P < 0.05$).

DISCUSSION

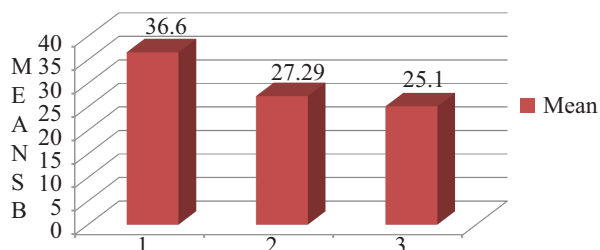
An accepted principle in restorative dentistry states that the transition between the restorative material and the dental hard tissue must be continuous to increase the survival probability of the restoration. Poor marginal adaptation may produce postoperative hypersensitivity, marginal discoloration, and ultimately secondary caries and pulpal inflammation.⁸ The long-term clinical success of adhesive restoration is primarily dependent on the bonding quality of adhesive systems to dentin, and the key parameter for evaluating the bond quality of different dentin-adhesives systems is bond strength. An ideal bond strength test should be accurate, clinically reliable, and less technique sensitive. It should involve the use of relatively unsophisticated and inexpensive test protocols.⁹ Oilo et al¹⁰ classified them into qualitative screening tests and quantitative tests. Qualitative tests study bond failures, and quantitative tests predict the load capacity and lifetime of the bond. Bond strength can be assessed by laboratory methods and clinical performance. It can be measured statically using a macro- or micro-test set-up, basically depending upon the size of the bond area.⁹

International Standards Organization (ISO) Technical Specification No. 29022 provides guidance on substrate selection, storage, and handling. It also presents some specific test methods for bond strength measurements.¹¹

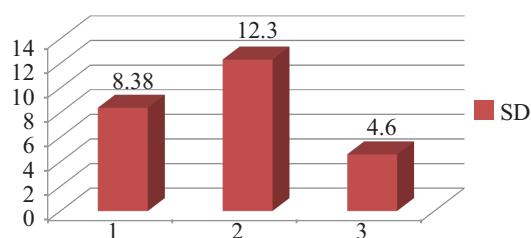
Testing with a bonded cross-sectional area of 3mm² or less is referred to as micro shear bond strength. Recently, the micro-shear bond strength (μ -SBS) test has been advocated as a modified method for evaluating the bonding ability of dentin-adhesive systems. A significant advantage over micro-tensile strength methods is that μ -SBS specimen is pre-stressed prior to testing only by removal.¹² Compared to the macro-shear bond strength test, the μ -SBS test is more advantageous; it has fewer internal defects as well as more homogeneous stress distributions at the interface due to the use of smaller specimen.

Recently, Shimaoka et al.¹³ proposed that the adhesive area should be delimited and constrained to the dentin substrate so as to equate the area between the adhesive and the resin and to eliminate differences in test results caused by traditional adhesive application technology.

Scherrer et al¹⁴ carried out a meta-study on publications dating from 1998 to 2009 investigating the bond strength of resin composite to dentin using four protocols: shear, tensile,



Graph-1: Mean shear bond strength values of Group 1 (Clearfil SE), Group 2 (Single Bond Universal) and Group 3 (Xeno V+).



Graph-2: Standard deviation of Group 1 (Clearfil SE) Group 2 (Single Bond Universal) and Group 3 (Xeno V+).

Groups	Mean Difference	Std.error	P Value
Group 1 and Group 2	9.31	2.32	0.01
Group 1 and Group 3	11.5	2.32	0.01
Group 2 and 3	2.19	2.32	0.34

Table-2: Intergroup comparison of shear bond strength using post-hoc test, Where $P < 0.05$ statistically significant and $P > 0.05$ is non significant

microshear and microtensile bond strength. Six adhesives were selected covering three-step systems (OptiBond FL, Kerr-Sybron; Scotch Bond MP Plus, 3M ESPE), two-step systems (Prime and Bond NT, Dentsply; Single Bond, 3M ESPE; Clearfil SE Bond, Kuraray) and a one-step adhesive (Adper Prompt L Pop, 3M ESPE). The pooled results of 147 references showed high scattering in the bond strength data regardless of adhesive and test method.

Rüttermann et al¹⁵ evaluated shear bond strength was different on human and bovine teeth. Since bovine enamel and dentin develop more rapidly during tooth formation, bovine enamel has larger crystal grains and more lattice defects than human enamel. This influence bond strength because different grain sizes and defective lattice structures will be differently attacked by chemicals. This might explain the different performance of self-etch and etch and rinse adhesives.

High shear bond strengths to occlusal dentin were observed than buccal dentin. Thus, substrate location has to be specified while studying bond strength as done in the current study where the occlusal dentin was used for shear bond test.¹⁶

Freshly extracted teeth are the most suitable substrate for *in vitro* evaluation of adhesive systems. Titley et al.¹⁷ have reported that when teeth are stored by freezing to maintain their freshness, shear bond strength of resin to dentin is the highest. Distilled water, saline, 0.05% saturated solution of thymol, 0.5% chloramine-T, 2% glutaraldehyde, and 10% formalin solutions were studied as storage media for bond strength tests. According to the ISO technical specification

22902, bond strengths should be measured immediately post-extraction or within six months and chloramine T being ideal solution.¹¹

Self-etching adhesives gave higher bond strengths when dentin surfaces were prepared with tungsten carbide burs and adhesives performed significantly better when a smaller grit size was used to prepare the dentin surface.¹⁸ A meta-study carried out by Munck et al¹⁹ revealed that the most-used preparation methods were by either a carbide or diamond dental bur or by silicon-carbide (SiC) paper. The diamond bur produced a thick smear layer and uneven dentin surface, while the tungsten carbide bur produced a thin, evenly distributed smear layer with a smoother dentin surface.

Hara et al²⁰ used a knife-edge steel rod to test specimens with 3-mm diameter bonding area and found statistically higher bond strengths for those loaded at 1.0 and 5.0mm/min compared to 0.5 and 0.75mm/min. Poitevin et al. recommended a crosshead speed of 1 mm/min for more uniform stress-time pattern.²¹

Feilzer et al²² showed that polymerization stress in composite fillings is related to restoration configuration. The configuration factor C was defined as the ratio of the restoration's bonded to unbounded surfaces. In most bonding studies, the resin composite is bonded to the tooth at the bottom of the mould only, not to the sides. This results in a C-factor less than one and imparts little stress on the bond to the tooth as the resin polymerizes. If the resin composite can be bonded to the walls of the mould, as occurs in a tooth, this might provide a more clinically relevant test of bond strength.²³

Clearfil SE was chosen specifically for this study as the comparison to the two universal agents based on the fact that it utilizes the same self-etching primer, MDP, and has also been shown in multiple studies to have consistently stronger bond strengths than other two-step self-etch bonding agents and similar to those of the three-step etch-and rinse with superior clinical longevity. As Inoue S et al²⁴ state, "long-term durability of adhesive-dentin bonds depends on the chemical bonding potential of the functional monomer," of which 10-MDP seems to be the most important due to its ability to strongly interact with hydroxyapatite and form a hydrolytically stable calcium salt.

In this in vitro study the two-step self-etch adhesives showed a superior in vitro performance in comparison to one-step self-etch adhesive. These results are in accordance with the other studies.

Serious limitation of all-in-one adhesives are as follows: incomplete polymerization and continued demineralization of the adjacent dentin structure in the tubules. For all-in-one adhesives to be acidic, the formulation have become more hydrophilic, thereby allowing deeper penetration. As these adhesives penetrate the wet dentinal tubules deeply, the water content increases. Studies have shown that this water acts as a major interfering factor in polymerization which leads to unpolymerized acidic and aggressive monomers to continue etching the dentin, thereby leading to a detrimental impact on the bond.²⁵

Takahashi et al²⁶ the two-step self-etch adhesive systems

have been reported to yield higher bond strengths compared to one-step self-etch adhesive systems may be due to the proportions of their chemical constituents. Both contain functional monomers, crosslinking monomers, solvent, inhibitors and activators, but in different proportions. The one-step self-etch adhesive systems generally have less crosslinking monomers. These provide most of the mechanical strength, therefore, there is a potential for lower bond strength.

An estimated shear bond strength of 17-21 MPa has been proposed as the critical value needed to withstand these stress of polymerization contraction of composite material.²⁷ Clinical experiences confirm that this bond strength is sufficient for successful retention of resin restoration.

Bond strength to dentin depends on the material and the test method used. Additional clinical studies are needed to further evaluate the efficacy of all systems.

CONCLUSION

Two-step Clearfil SE gave highest bond strength compared to all other groups. It can be concluded that the two-step self-etch adhesive bond recorded higher bond strength than the single-step self-etch adhesives.

REFERENCES

1. Anusavice KJ, Phillips RW, Shen C, Rawls HR. Phillips' science of dental materials. 12th ed. St. Louis, Mo. Elsevier/Saunders; 2013.
2. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 1955;34:849-53.
3. Perdigao J. New developments in dental adhesion. *Dent Clin N Am* 2007; 51:333-57.
4. Carvalho RM, Tjäderhane L, Manso AP, Carrilho MR, Carvalho RA. Dentin as a bonding substrate. *Endod Topics* 2012; 21: 62-88.
5. Asmussen E, Uno S. Adhesion of restorative resin to dentin: chemical and physiochemical aspects. *Oper Dent Suppl* 1992; 5: 68-74.
6. Swift EJ. Dentin/enamel adhesives: review of the literature. *Pediatr Dent* 2002; 24: 456-61.
7. Van Meerbeek B, De Munck J, Yoshida Y, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 2003;28:215-35.
8. Heintze SD. Systematic Reviews: I. The Correlation Between Laboratory Tests on Marginal Quality and Bond Strength. II. The Correlation Between Marginal Quality and Clinical Outcome. *J Adhes Dent* 2007; 9: 77-106.
9. Sirisha K, Rambabu T, Shankar YR, Ravikumar P. Validity of bond strength tests: A critical review: Part I. *J Conserv Dent* 2014;17: 305-11.
10. Oilo G. Adhesion of dental materials to dentine: Debonding tests. In: Thylstrup A, Leach SA, Qvist V, ed. *Dentine and dentine reactions in the oral cavity*. Oxford: IRL Press Ltd.; 1987. p. 219-24.
11. ISO. Dental materials – testing of adhesion to tooth structure using notched etch. Technical specification no. 22902; 2013.
12. Sirisha K, Rambabu T, Shankar YR, Ravikumar P. Va-

- lidity of bond strength tests: A critical review: Part II. *J Conserv Dent* 2014;17: 420-6.
13. Shimaoka AM. The importance of adhesive area delimitation in a microshear bond strength experimental design. *J Adhes Dent* 2011;13: 307-14.
 14. Scherrer SS, Cesar PF, Swain MV. Adhesion in dentistry – analysing bond strength test methods, variables and outcomes. Direct comparison of the bond strength results of different test methods: a critical literature review. *Academy of Dental Materials Annual Meeting – Portland Oregon, USA; 2009; 29-31.*
 15. Rüttermann S, Braun A, Janda R. Shear Bond Strength and Fracture Analysis of Human vs. Bovine Teeth. *Plos one* 2001; 8: 1-6.
 16. Carvalho RM, Tjäderhane L, Manso AP, Carrilho MR, Carvalho RA. Dentin as a bonding substrate. *Endod Topics* 2012; 21: 62-88.
 17. Titley KC, Chernecky R, Rossouw PE, Kulkarni GV. The Effect of Various Storage Methods and Media on Shear-Bond Strengths of Dental Composite Resin to Bovine Dentine. *Arch of Oral Bio* 1998; 43: 305-11.
 18. Perdigao J. Dentin bonding — Variables related to the clinical situation and the substrate treatment. *Dent Mater* 2010; 26: 24-37.
 19. Munck JD, Mine A, Poitevin A, Ende AV, Cardoso MV, Landuyt KL, Pneumans M, Van Meerbeek B. Meta – analytical Review of Parameters Involved in Dentin Bonding. *J Dent Res* 2012; 9: 351-7.
 20. Hara AT, Pimenta LA, Rodrigues Jr AL. Influence of cross-head speed on resin–dentin shear bond strength. *Dent Mater* 2001;17:165-9.
 21. Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, et al. Critical analysis of the influence of different parameters on the microtensile bond strength of adhesives to dentin. *J Adhes Dent* 2008;10:7-16
 22. Feilzer AJ, De Gee AJ, Davidson CL. Setting stress in composites resin in relation to configuration of the restoration. *J Dent Res* 1987; 66: 1636-9.
 23. Bouillaguet S, Ciucchi B, Jacoby T, Wataha JC & Pashley D. Bonding characteristics to dentin walls of Class II cavities, in vitro. *Dent Mater* 2001; 17: 316-21.
 24. Inoue S, Koshiro K, Yoshida Y, De Munck, Nagakane K, Suzuki K, Sano H, Van Meerbeek. Hydrolytic stability of self-etch adhesives bonded to dentin. *J Dent Res* 2005; 84: 1160-4.
 25. Perdigao J, Gomes G, Gondo R, Fundingsland JW. In vitro bonding performance of all-in-one adhesives. Part I—microtensile bond strengths. *J Adhes Dent* 2006;8: 367-73.
 26. Takahashi A, Sato Y, Uno S, Pereira PN, Sano. Effects of mechanical properties of adhesive resins on bond-strength to dentin. *Dent Mater* 2002;18:263-8.
 27. Hegde MN, Bhandary S. An evaluation and comparison of shear bond strength of composite resin to dentin, using newer dentin bonding agents. *J Cons Dent* 2008; 11:71-5.

Source of Support: Nil; **Conflict of Interest:** None

Submitted: 29-12-2015; **Published online:** 12-01-2016