Received: 25th March 2014 Accepted: 10th June 2014 Conflict of Interest: None

Source of Support: Nil

Original Research

Comparative Evaluation of Microshear Bond Strength of 5^{th} , 6^{th} and 7^{th} Generation Bonding Agents to Coronal Dentin Versus Dentin at Floor of Pulp Chamber: An *In vitro* Study

Velagala Lakshmi Deepa¹, Bhargavi Damaraju², Bollu Indira Priyadharsini³, Vummidisetti V Subbarao⁴, K Rama Krishna Raju⁵

Contributors:

¹Reader, Department of Conservative Dentistry and Endodontics, KLR'S Lenora Institute of Dental Sciences, Rajahmundry, Andhra Pradesh, India; ²Senior Lecturer, Department of Conservative Dentistry and Endodontics, KLR'S Lenora Institute of Dental Sciences, Rajahmundry, Andhra Pradesh, India; ³Senior Lecturer, Department of Conservative Dentistry and Endodontics, St. Joseph Dental College, Eluru, Andhra Pradesh, India; ⁴Reader, Department of Conservative Dentistry and Endodontics, Lenora Institute of Dental Sciences, Rajamundry, Andhra Pardesh, India; ⁵Professor and Head, Department of Conservative Dentistry and Endodontics, KLR'S Lenora Institute of Dental Sciences, Rajahmundry, Andhra Pradesh, India.

Correspondence:

Dr. Deepa VL. Department of Conservative Dentistry and Endodontics, KLR'S Lenora Institute of Dental Sciences, Rajahmundry, Andhra Pradesh, India. Email: drdeeparamesh@yahoo.com

How to cite the article:

Deepa VL, Damaraju B, Priyadharsini BI, Subbarao VV, Raju KR. Comparative evaluation of microshear bond strength of 5th, 6th and 7th generation bonding agents to coronal dentin versus dentin at floor of pulp chamber: An *in vitro* study. J Int Oral Health 2014;6(5):72-6.

Abstract:

Background: Lack of seal and adhesion between the final restoration and tooth structure adversely affects the results of root canal treatment. Lots of adhesive bonding agents are marketed to overcome this deficiency and achieve successful restoration. So the study compares and evaluates the micro shear bond strength of coronal dentin and pulp chamber dentin using three different generation dentin bonding systems and to know clinical efficiency for clinical use.

Materials and Methods: Different generation dentin bonding systems used were: (1) One bottle total etch system (XP Bond-5th generation), (2) Two-step self-etch system (Clearfil SE Bond-6th generation) and (3) All-in-one system (G Bond-7th generation). Thirty human mandibular molars were collected out of which sixty samples were prepared by sectioning each tooth into coronal dentin and pulpal floor dentin. They were divided into two major groups. Group I: 30 Coronal dentin samples. Group II: 30 Pulpal floor dentin samples. Both the groups were further subdivided depending on the bonding agent used. Subgroup Ia: XP Bond, Subgroup Ib: Clearfil SE Bond, Subgroup Ic: G Bond, Subgroup IIa: XP Bond, Subgroup IIb: Clearfil SE Bond, Subgroup IIc: G Bond. Resin composite was bonded to these samples and tested for micro-shear bond strength. The mean bond strengths and standard deviations were calculated and analyzed using one-way ANOVA test and Student's t-test (unpaired) and honestly significant difference post-hoc tests.

Results: Coronal dentin showed higher values of micro shear bond strength than the pulpal floor dentin. All-in-one system (G Bond) showed least bond strength values to both the regions coronal dentin and pulpal floor dentin.

Conclusion: Factors affecting the shear bond strength are dependent on material (adhesive system), substrate depth and adhesive/depth interaction. Hence composition and substrate treatment should be considered for good adhesive. Chemical composition of adhesive system determines clinical successes.

Key Words: All-in-one system, coronal dentin, microshear bond strength, pulpal floor dentin, self-etch, total etch

Introduction

The overall prognosis of the tooth after obturation depends on the quality of coronal restoration. Obturation will not provide a thorough seal if tooth is not appropriately restored. Lack of seal and adhesion between the final restoration and tooth structure adversely affects the results of root canal treatment. Amalgam as restorative has several beneficial properties such as high strength, high modulus of elasticity and operator friendliness, one of its main drawbacks is a lack of seal and adhesion to tooth structure. Amalgam also requires placement of retentive features that demands excessive removal of tooth structure that further weakens the already weakened non vital tooth. The use of dental amalgam is declining worldwide because of legislative, safety and environmental issues.

We are in the era of adhesive dentistry. Adhesive restorations bond directly to the tooth structure and reinforce weakened tooth structure.¹ Restoration of endodontically treated teeth with resin-based composite has increased due to development of better, more reliable bonding systems. Composite core buildup provides the high bond strength to tooth structure and increased resistance to fracture.² Composite core material should have a good bond strength to the pulpal floor dentin so that it enhances retention and maximizes the seal.²

Opportunity for restoration of non-vital teeth with resin-based composite has increased due to the development of better and more reliable dentin bonding systems. Various bonding agents were being introduced into the market. Most recent developments have focused on simplification of multistep bonding processes using different approaches i.e., total etch, two-step self-etch and all-in-one system. The laboratory parameter most commonly used to measure the bonding

effectiveness with dentin adhesives is micro shear bond strength. Hence, the objective of this study was to compare and evaluate the microshear bond strength of coronal and pulpal floor dentin using three-generation dentin bonding systems.

Materials and Methods

Materials used were as follows: (1) Composite resin: Clearfil APX (Kuraray) (2) Bonding agents: XP Bond (Dentsply) -5th generation, Clearfil SE Bond (Kuraray) - 6th generation, G Bond (GC) - 7th generation, (3) Acid etchant: 37% Phosphoric acid (d-tech), and (4) Storage media- saline (Figure 1a). Thirty human mandibular molars extracted for periodontal reasons were collected for the study (Figure 1b) and the teeth were cleaned with ultrasonic scalers and stored in saline. The occlusal enamel was removed with high-speed diamond disc to expose a flat mid coronal dentin. 2 mm thick slabs of coronal dentin and pulpal floor dentin samples were prepared by sectioning at midpoint between floor of the pulp chamber and root furcation. These prepared dentinal slabs were finished with wet silicon carbide sand paper under a stream of water to create an uniform smear layer. Samples were divided into two major groups depending upon the dentin location are Group I: 30 Samples of coronal dentin and Group II: 30 Samples of dentin at floor of the pulp chamber. Each group was further subdivided into three subgroups (Figure 2a-f) of 10 samples each depending upon the bonding agent used (Subgroup a - XP Bond, Subgroup b - Clearfil SE Bond, Subgroup c - G Bond).

Subgroup Ia, IIa was bonded with two-step total etch technique (XP Bond), Subgroup Ib, IIb was bonded with two-step self-etch technique (Clearfil SE Bond), Subgroup Ic, IIc was bonded with one-step self-etch technique (G Bond) according to manufacturer's instructions. After applying the adhesive, polyethylene tube (1 mm diameter, 1 mm height) was placed and the adhesive was light-cured for 10 s according to manufacturer's instructions, thereby fixing the tube to dentin



Figure 1: (a and b) armamentarium, material and study samples.

the surface. Resin composite was placed in the tube, and light cured. The intensity of curing light was measured by a portable radiometer, prior to each bonding procedure to confirm the values >600 Mw/cm². After the completion of composite resin buildup, polyethylene tubes were removed with a sharp knife. All specimens were stored at 37°C in water.

Measurement of microshear bond strength

The specimens were attached to the universal testing machine (Figure 3). A thin wire (0.010 inches in diameter) was looped around resin composite cylinder and gently held flush against the dentin at resin dentin interface and loaded at a rate of 1 mm/min until bond failure occurred.

The resin dentin interface for the test, the wire loop and the center of load cell were aligned as straight as possible to ensure correct application of the shear force.

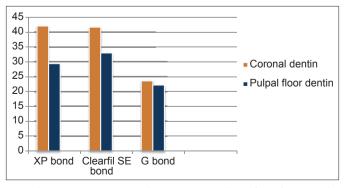
The load at failure was recorded in Newton's/mm square and then converted to MPa. The data were submitted to statistical analysis using honestly significant difference *post-hoc* tests for multiple group comparisons. P = 0.05 or less was considered for statistical significance.

Results

In the present study, coronal dentin showed high micro shear bond strengths compared to pulpal floor dentin (Graph 1). No statistically significant differences were observed between the mean bond strengths between XP Bond and Clearfil SE in each region (P > 0.05). Between XP Bond and G Bond, the mean bond strength of XP Bond was significantly higher than that of G Bond in both the regions (P < 0.05). Between Clearfil SE and G Bond, the mean bond strength of Clearfil SE was significantly higher than that of G Bond in both the regions (P < 0.05). All-in-one system (G Bond) showed least bond strength values to both the regions (Tables 1 and 2).

Discussion

The overall prognosis of the tooth after obturation depends on the quality of coronal restoration. Obturation alone will not provide a thorough seal if tooth is not appropriately restored.



Graph 1: Comparison of mean microshear bond strengths between coronal dentin and pulpal floor dentin.

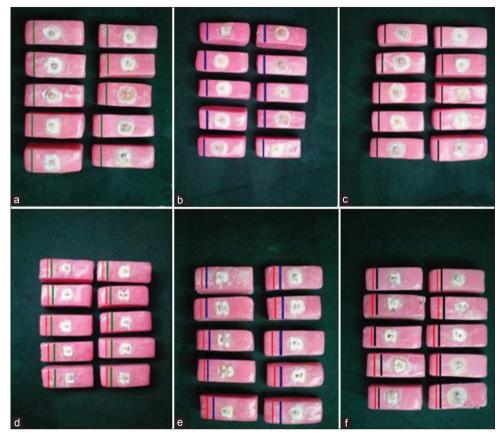


Figure 2: Two groups of specimens (coronal and pulpal floor dentin) bonded with (a and d) Xp bond, (b and e) Clearfil Se bond (c and f) G-bond.



Figure 3: Shearing of composite material using universal strength testing machine.

Dental Amalgam has been considered to be the most commonly used final restorative material to fill the access opening after endodontic treatment with a long history of clinical success. Though Amalgam has several beneficial properties like high strength, high modulus of elasticity and operator friendliness, one of its main drawbacks is a lack of seal and adhesion to tooth structure. The use of dental amalgam is declining worldwide because of legislative, safety and environmental issues. In the era of Adhesive restorations, we need to have sound knowledge of different adhesive bonds available in the market for proper

| Table 1: Tukey HSD <i>post-hoc</i> test - multiple comparisons between coronal dentin. | | | | | |
|--|-----------------------|--------|-------------|--|--|
| Groups | Mean difference (I-J) | SE | Significant | | |
| Ia | | | _ | | |
| Ib | 0.3800 | 2.3860 | 0.986 | | |
| Ic | 18.4700* | 2.3860 | 0.000 | | |
| Ib | | | | | |
| Ia | -0.3800 | 2.3860 | 0.986 | | |
| Ic | 18.0900* | 2.3860 | 0.000 | | |
| Ic | | | | | |
| Ia | -18.4700* | 2.3860 | 0.000 | | |
| Ib | -18.0900* | 2.3860 | 0.000 | | |
| The mean difference is significant at the 0.05 level. SE: Standard error, HSD: Honestly significant difference | | | | | |

| Table 2: Tukey HSD <i>post-hoc</i> test - multiple comparisons between pulpal floor dentin. | | | | |
|---|--|--------|-------------|--|
| Groups | Mean difference (I-J) | SE | Significant | |
| IIa | | | _ | |
| IIb | -3.6000 | 2.4729 | 0.328 | |
| IIc | 7.2000* | 2.4729 | 0.019 | |
| IIb | | | | |
| IIa | 3.6000 | 2.4729 | 0.328 | |
| IIc | 10.8000* | 2.4729 | 0.000 | |
| IIc | | | | |
| IIa | -7.2000* | 2.4729 | 0.019 | |
| IIb | -10.8000* | 2.4729 | 0.000 | |
| - | fference is significant at the 0.05 level. SE: Standard error, HSD: Honestly | | | |

significant difference

and successful use. As these adhesive bonds directly to the tooth structure and reinforce weakened tooth structure.¹

A reliable and durable bond to dentin has been more difficult to achieve. Dentin is complex biological structure whose structure and properties change with location, age and disease.

Variation in dentin depth and permeability can significantly influence the bond strength of direct resin-based composite restorations. It is also probable that the bonds made to floor of pulp chamber versus coronal dentin may differ.³ Bonding to coronal dentin usually involves sheared surfaces of coronal dentin, smear layer and smear plugs within the dentinal tubules. On the contrary pulpal floor, dentin is a complex biological structure, which includes primary dentin, regular and irregular secondary dentin and this dentin is not usually contacted by cutting instruments.¹ Hence, the thickness of smear layer produced here is less compared to coronal dentin and the tubule diameter is much smaller and tubule density is high making it more challenging bonding substrate.⁴

Group I (coronal dentin group) showed significantly higher values of micro shear bond strength when compared to Group II (pulpal floor dentin group) it may be due to morphological and structural variations in dentin, presence of more inorganic material in coronal dentin and density of dentinal tubules, which is around $45,000/\text{mm}^2$ and even diameter of dentinal tubules was $2.5\,\mu\text{m}$, which is much larger when compared to pulpal floor dentin. The results were also in accordance with previous studies. 2,3,5

Group Ia (coronal dentin with XP Bond) showed higher values of micro-shear bond strengths when compared to Group Ib (coronal dentin with Clearfil SE Bond), but were not statistically significant it may be attributed because of demineralization and resin infiltration into collagen occur simultaneously to the same depth of demineralized dentin. It is presumed that no gap or void exists. Clearfil SE Bond showed almost similar values as XP Bond in accordance with other studies.⁵⁻⁸ This may be because of components of adhesive resin⁷ – fillers, functional monomer, 10-MDP and also it's pH that will influences bond strength. Presence of highly hydrophilic 10-MDP monomer in its composition, which is believed to improve wetting of the moist tooth surface more over it has two hydroxyl groups that may chelate to calcium of dentin.⁵⁻⁸ Fillers present in Clearfil SE bond were necessary to increase bond strength and improve mechanical properties of bonding agents.9,10

Group II (pulpal floor dentin) showed lower values of micro shear bond strength compared to Group I in accordance with previous studies^{2,3,5} it may be because of ultra-structure of dentin at floor of the pulp chamber seems similar to reparative or secondary dentin containing fewer irregular narrower

tubules. These tubular irregularities may have occurred because of mineral deposits, organic components of odontoblastic process or peritubular deposits. These changes could impact penetration of monomers into dentinal tubules resulting in poorer bonding to this region. Pulpal floor dentin seems to be rich in organic components and low in mineral. Presence of greater organic content, resulted in reduced penetration of monomer. Predentin on floor of the pulp chamber is thought to affect the bond strength. The reduced surface area of intertubular dentin available for bonding may also contributed to lower bond strength.

Group IIb (pulpal floor dentin with Clearfil SE Bond) showed higher bond strengths when compared to Group IIa and Group IIc (pulpal floor dentin with XP Bond and G Bond) is in accordance with previous studies. ^{2,3,5} This may due to devoid of smear layer as pulpal floor dentin not contacted by any cutting instruments. Acid conditioning of primer in Clearfil SE bond appeared sufficient to demineralize the dentin and envelope collagen fibers and hydroxyapatite crystals. Camphoroquinone contained in the primer is likely to enhance adhesion to dentin because it generates free radicals that increase surface energy and wetting ability thereby increasing bond strength and Presence of highly hydrophilic 10- MDP monomer in its composition, which is believed to improve wetting of the moist tooth surface; moreover, it has two hydroxyl groups that may chelate to calcium of dentin. Also, fillers present in Clearfil SE bond were necessary to increase bond strength and improve mechanical properties of bonding agents.^{9,10} Use of 37% phosphoric acid with XP Bond quickly removes all inorganic matter in peritubular dentin causing deeper penetration of acid in to dentin resulting in over-etching and subsequent collapse of collagen network thus leading to porous zone within hybrid layer.11

Group IIa (pulpal floor dentin with XP Bond) showed lower bond strengths when compared to Group IIb, which was statistically insignificant. The results were in accordance with previous studies conducted: Toba *et al.*³ and Akagawa *et al.*⁵ because pulpal floor dentin is rich in organic components and less in mineral. Use of 37% phosphoric acid will result in overetching and collapsing of collagen fibers leading to decreased bond strengths.

All-in-one system (G Bond) showed least bond strength values to both the regions (coronal dentin and pulpal floor dentin). The results were in accordance with previous studies conducted by Sidhu *et al.* and Yazici *et al.*⁴ All-in-one system has greater technique sensitivity compared with other bonding agents. Problem with water based all-in-one system mainly arises from the hydrolytic instability of methacrylate monomers used. One-step self-etching adhesives are more hydrophilic than two-step self-etching adhesives, and they attract more water. As it is difficult to evaporate water from these adhesives,

water will rapidly diffuse back from the bonded dentin into adhesive resin and subsequently, lower mechanical strength results. Although in the present study, G Bond showed lower bond strengths, a recent study by Burrow *et al.*⁷ using G Bond showed good results. It is probable that the differences between the two studies may be due to the different methodologies employed.

Simplification of self-etching priming systems has not led to an improvement in bond strength. Though there is a tendency toward adhesives with simplified application procedures simplification does not guarantee improved or equal bonding effectiveness. The application of new components with improved hydrolytic stability may help to solve the problems¹² of all-in-one systems. Further investigations should be carried out to determine whether additional etching^{13,14} or application of additional more hydrophobic resin layer.¹⁴

Conclusion

Based on our study, we conclude that adhesive and dentin depth are the factors affecting the bond strength. The dental adhesive systems also have significant influence on shear strength. Additional etching or application of additional more hydrophobic resin layer prior to application of self-etching solutions will provide clinical benefits to retention rates should be further investigated to give clinical orientation. However, further studies are needed to investigate the bond strengths of these adhesive systems under clinically acceptable conditions.

References

- 1. Belli S, Zhang Y, Pereira PN, Ozer F, Pashley DH. Regional bond strengths of adhesive resins to pulp chamber dentin. J Endod 2001;27(8):527-32.
- 2. Kijsamanmith K, Timpawat S, Harnirattisai C, Messer HH. Micro-tensile bond strengths of bonding agents to pulpal floor dentine. Int Endod J 2002;35(10):833-9.
- Toba S, Veerapravati W, Shimada Y, Nikaido T, Tagami J. Micro-shear bond strengths of adhesive resins to coronal

- dentin versus the floor of the pulp chamber. Am J Dent 2003;16:51A-6.
- 4. Yazici AR, Celik C, Ozgünaltay G, Dayangaç B. Bond strength of different adhesive systems to dental hard tissues. Oper Dent 2007;32(2):166-72.
- Akagawa H, Nikaido T, Takada T, Burrow MF, Tagami J. Shear bond strengths to coronal and pulp chamber floor dentin. Am J Dent 2002;15(6):383-8.
- 6. Toledano M, Osorio R, Ceballos L, Fuentes MV, Fernandes CA, Tay FR, *et al.* Microtensile bond strength of several adhesive systems to different dentin depths. Am J Dent 2003;16(5):292-8.
- 7. Burrow MF, Kitasako Y, Thomas CD, Tagami J. Comparison of enamel and dentin microshear bond strengths of a two-step self-etching priming system with five all-in-one systems. Oper Dent 2008;33(4):456-60.
- 8. Bouillaguet S, Gysi P, Wataha JC, Ciucchi B, Cattani M, Godin C, *et al.* Bond strength of composite to dentin using conventional, one-step, and self-etching adhesive systems. J Dent 2001;29(1):55-61.
- 9. Takahashi A, Sato Y, Uno S, Pereira PN, Sano H. Effects of mechanical properties of adhesive resins on bond strength to dentin. Dent Mater 2002;18(3):263-8.
- 10. Sensi LG, Lopes GC, Monteiro S Jr, Baratieri LN, Vieira LC. Dentin bond strength of self-etching primers/adhesives. Oper Dent 2005;30(1):63-8.
- 11. Tanumiharja M, Burrow MF, Tyas MJ. Microtensile bond strengths of seven dentin adhesive systems. Dent Mater 2000;16(3):180-7.
- 12. Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel-dentin adhesives: A systematic review. Dent Mater 2005;21:895-910.
- 13. Proença JP, Polido M, Osorio E, Erhardt MC, Aguilera FS, García-Godoy F, *et al.* Dentin regional bond strength of self-etch and total-etch adhesive systems. Dent Mater 2007;23(12):1542-8.
- 14. Lührs AK, Guhr S, Schilke R, Borchers L, Geurtsen W, Günay H. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. Oper Dent 2008;33(2):155-62.