Original Article

Effect of Mechanical Load Cycling on Microtensile Bond Strength of Self-Etch Systems to Dentin

Sh. Kasraei [1, 2], Z. Khamverdi [1]

[1]Associate Professor, Department of Operative Dentistry, School of Dentistry, Hamadan University of Medical Sciences, Hamadan, Iran
[2]Associate Professor, Dental Research Center, Hamadan University of Medical Sciences, Hamadan, Iran

Abstract:

Objective: Optimal dentin bonding is not always obtained in clinical practice due to functional forces. These forces may provide stresses throughout the tooth and restorative system, which in turn may affect the adhesive bond. This experimental study evaluated the effect of load cycling on bond strength of self-etch systems.
Materials and Methods: One third of the coronal portions of 48 intact human extracted upper premolars were removed. The teeth were equally divided into six groups. In groups Unloaded-Clear tri-S Bond (ULCB) and Loaded-Clear tri-S Bond (LCB), prepared dentin surfaces of each tooth were treated with Clearfil tri-S Bond (Kurary medical inc, Okayama, Japan), in groups Unloaded-I Bond (ULIB) and Loaded-I Bond (LIB) with I bond (Heraeus Kulzer, GmbH,&Co KG, Germany), and in groups Unloaded-Xeno III (ULX) and Loaded-Xeno III (LX) with Xeno III dentin adhesives (Dentsply Detrey GmbH, Konstanz, Germany). Then, the teeth were restored with Filtek Supreme resin composite. After thermal cycling of the samples, Groups LCB, LIB and LX were submitted to mechanical loading (100,000 cycles, 60 N). Microtensile bond strength (MTBS) test was performed for all of groups. Two-way ANOVA and Tukey HSD tests served for statistical analysis.
Results: Results showed that groups ULCB and ULIB had significantly more bond strength than group ULX ($P<0.05$). For all the tree adhesive systems, the MTBS values in teeth subjected to load cycling was significantly lower than unloaded teeth ($P<0.001$).
Conclusion: Load cycling seems to decrease MTBSs of one-step adhesive systems to dentin.

Key Words: Dentin-Bonding Agents; Fatigue; Adhesives

Corresponding author:
Z. Khamverdi, Department of Operative Dentistry, School of Dentistry, Hamadan University of Medical Sciences, Hamadan, Iran.
zkhamverdi@yahoo.ca

Received: 31 December 2007
Accepted:16 April 2008

Journal of Dentistry, Tehran University of Medical Sciences, Tehran, Iran (2008; Vol: 5, No.4)

INTRODUCTION

Micro-mechanical retention within the demineralized dentin surface through the formation is currently known as the mechanism for bonding adhesive resins to dentin [1,2]. Based on clinical application steps, modern dentin bondings agents can be classified into three-step total-etch, two-, and single-step systems. Two types of two-step systems exist: the total etching adhesive systems requiring a separate etching step; and the self-etching primer systems requiring an additional bonding step [3,4]. Development and marketing of new generations of bonding agents are growing rapidly. In order to simplify clinical procedure, the most recent commercially available systems have attempted to reduce the number of system components through combining the bonding procedures into a single step application. These adhesives are one-step self-etch, or the so called "All in one" [3,5]. It has been reported that their ability to simultaneously demineralize and penetrate into enamel significantly reduces the working time, which is of great advantage in pediatric dentistry [6]. Moreover, some *in vitro* studies have reported high bond strengths for these materials [3,5,7,8].

Since such factors as normal daily functioning, thermal stresses, malocclusion, and habitual bruxism stresses through out the tooth and restorative system may affect and destroy the adhesive bond, optimal dentin bonding may not always be obtained in clinical practice [9,10].

Applying cyclic mechanical stress on restoration could simulate actual conditions. This simulation can provide valuable information on dentin bonding durability especially for newly introduced agents. However, a few studies exist on this subject in the literature [11-14].

The purpose of this *in vitro* study was to evaluate the effect of mechanical load cycling on microtensile bond strength (MTBS) of three self-etch dentin adhesives.

MATERIALS AND METHODS

The study was done on 48 freshly extracted sound human upper premolars. The specimens had been stored in 0.5% Chloramines-T (Prolabo, Paris, France) solution at 4ºC for less than four months. Twenty-four hours before starting of the experiment, they were immersed in distilled water. The teeth were cleaned and polished by using slurry pumice with a brush and low-speed handpiece. Coronal portion of the teeth was removed using trimmer machine (Dentaurum, Pforzheim, Germany) with water coolant. The cut dentin surface was abraded against 400 grits and then 600 grits wet silicon carbide papers (991 A, Softlex, Germany) to obtain a standard and uniform smear layer.

The teeth were randomly divided into six groups of 8 each and prepared as fallows:

In groups Unloaded-Clear tri-S Bond (ULCB) and Loaded-Clear tri-S Bond (LCB), the prepared surface of each tooth was treated with Clearfil tri-S Bond dentin adhesive, in groups Unloaded-I Bond (ULIB) and Loaded-I Bond (LIB) with I bond dentin adhesive, and in groups Unloaded-Xeno III (ULX) and Loaded-Xeno III (LX) with Xeno III dentin adhesive according to manufacturers' instructions (Table 1). Then, the treated surfaces restored with a light cured composite resin (Single Bond®, 3M ESPE, USA), to a height of approximately 5mm. Each increment was separately cured for 20 seconds using a Halogen light curing unit (Coltolux II, Coltene, Altatatten, Germany) with output of 500 mW/cm^2.

Specimens were stored in distilled water at 37ºC for a week. In all groups thermo cycling was separately simulated with a thermo cycle apparatus for 2500 cycles. The teeth were

Table 1. The adhesive systems applied on 48 extracted premolars to compare microtensile bond strength with and without load cycling

Adhesive system	Manufacturer	Application Protocol
ClearfilTri-S Bond	Kurary medical inc, Okayama, Japan	Dispense one drop of liquid into mixing well Apply to dentin for 20 seconds Relatively strong stream of air to dry and light irradiation for 10 seconds
I Bond	Heraeus Kulzer, GmbH,&Co KG, Germany	Apply in two consecutive layer and rub for 30 seconds, Gentle air dry until adhesive moves no more Thoroughly air dry for 5 seconds and Light cure for 20 seconds
Xeno III Bond	Dentsply Detrey GmbH, Konstanz, Germany	Dispense one drop of liquid A and liquid B into mixing well and Mix for 5 seconds Apply generously onto preparation surfaces Leave for at least 20 seconds, Gentle stream of air for at least 2 seconds until there is no flow and light irradiation for 10 seconds

placed in separate mesh bags, and alternated between 5ºC (SD=1) and 55ºC (SD=1) with dwell time of one minute in each bath and 15-second transition time between the baths.

Groups LCB, LIB and LX were selected to be load cycled. The mechanical load-cycling test was performed as fallows:

Root surfaces were wrapped in two layers of 0.1mm foil (Adapta System; Bego, Bremen, Germany) prior to being embedded in clear acrylic resin (GC Pattern Resin, GC America, USA). This procedure allowed embedding the root in the resin in a way that the crown margin was two millimeters above the level of the acrylic resin in order to simulate the position of the alveolar bone in natural teeth. Roots were mounted in the acrylic resin blocks with PVC rings 2 cm in diameter. When the first signs of polymerization appeared, the teeth and the foil spacers were removed from the acrylic resin blocks and the root surfaces, respectively. An injectionable type vinyl polysiloxane impression material (Rapid, Coltene AG, Altstätten, Switzerland) was delivered with a dispenser gun (3M, USA) through the mixing tip into the acrylic resin alveolus. The teeth were reinserted in the blocks and then impression material was allowed to polymerize. The excess silicone material was removed with a scalpel blade to provide a flat surface 2 mm below the facial CEJ of each tooth. The thin layer of silicone material simulated the periodontal ligament.

A stainless metal plate (10 mm diameter, 1 mm thick) was abraded with alumina and luted to resin composite with panavia F2.0 cement (Kuraray Co. Ltd, Osaka, Japan) to avoid stress formation in the center of resin composite restoration during load cycling procedures. The pulp chamber was penetrated buccally or palatally with a tube (sealed with DBA, Optibond, FL, USA), which was connected to a simulated pulpal circulation of saline water under a pressure of 14 cm H_2O. All specimens were submitted to 100000 cycles with 60N loading forces. The axial force was exerted at a two-hertz frequency following a one-half sinus wave curve. Composite restored surfaces were contacted by antagonist artificial surfaces made of stainless steel with a hardness similar to natural enamel (Vickers hardness: enamel=320-325; steel=315). Loading was performed with Universal testing machine (MTS 858; MTS systems Corp, Edent Prairie, Minn, USA).

In order to perform the MTBS test, the coronal part of the teeth were sectioned parallel to the long axis with a hard tissue cutting machine (Ham Co. machines, Inc., Rochester, NY, USA) to form 1 mm wide slices. Each slice was trimmed and shaped along the adhesive interface with a super-fine C-16 diamond bur (GC Ltd, Tokyo, Japan) resulting in a 1 mm^2 cross-section. Digital calipers were used to measure the bonded interface dimensions. Two specimens were prepared from each tooth and each specimen was tested with a micro tensile testing machine (Bisco Inc, IL, USA) at a cross-head speed of 0.5 mm/min. Sixteen specimens were measured in each tested group. The data were analyzed using one-way ANOVA and Tukey HSD tests.

Table 2. Microtensile bond strength means and standard deviations (SD) of three adhesive systems applied on 48 extracted premolars (two specimen from each tooth) with and without load cycling

Group	N	**Mean** (MPa)	**SD** (MPa)
Unloaded-Clear tri-S Bond (ULCB)	16	29.99	7.98
Loaded-Clear tri-S Bond (LCB)	16	19.54	5.25
Unloaded-I Bond (ULIB)	16	18.42	3.23
Loaded-I Bond (LIB)	16	11.73	2.88
Unloaded-Xeno III (ULX)	16	22.93	3.72
Loaded-Xeno III (LX)	16	16.57	3.22

RESULTS

Table 2 summarizes the means of micro tensile bond strength and the standard deviations for the tested groups. ULCB showed the highest bond strength, while Group LIB had the lowest values. Results of two-way ANOVA test indicated statistically significant differences of MTBS means for the main factors (load cycling and dentin bonding type) (P=0.001) but no significant difference existed for their interactive effect (P=0.15) (Table 3).

Tukey HSD tests as post-hoc test showed that MTBS of load cycled groups were significantly lower than unloaded groups (P=0.001). No significant difference existed between mean MTBS values of groups ULCB and ULX (P=0.06), but the mean MTBS of Group ULIB was significantly lower than these two groups (P<0.05).

DISCUSSION

The results of this study showed that loaded specimens have the lower micro tensile bond strengths than unloaded specimens. A reliable and durable bonding between resin materials and dentin is important in the field of adhesive dentistry. An ideal study method to evaluate quality of new bonding systems is long term clinical trial [4,15]. The performance of such studies, however, is difficult because of operator variability, substrate differences, recall failure, and taking time and resources [16]. The present study was designed to simulate clinical situations. Thus, the samples were subjected to load and thermo cycling.

In self-etch adhesive dentin bonding systems presence of acidic monomers results in formation of a thin hybridoid layer on dentinal surface. This layer provides micro mechanical retention for restorations. It is thought that this layer is the weakest link to achieve long-term durable bonding. A possible explanation for the obtained result can be concentration of main stress in hybridoid layer interface probably leading to plastic deformation of the adhesive interface when mechanical load cycling was applied. Fatigue could be a facilitating factor for failure in hybridoid layer. Our results confirmed previous studies that indicated fatigue could decrease resin-dentin bond strength [17-20].

It has been reported that demineralized dentin became weaker after cyclic loading [21]. Nikaido et al [11] evaluated the effect of thermocycling and mechanical loading on bond strength of a self-etching primer system to dentin and concluded that surface preparation, C-factor, cavity depth and dentin substrate influence bond strength values after thermal and fatigue loadings. A study on a total etch adhesive system showed that mechanical cycling alone did not affect bond strength but when thermal and mechanical load cycling were performed bond strength decreased significantly [12]. Some studies indicated that no significant difference existed between shear bond strength of unloaded and loaded groups when cyclic compressive loading were applied [22-24].

Compared to other adhesive bond strength tests, MTBS test has several advantages including the improvement of stress distribution during testing, the prevention of cohesive failures in dentin, the ability to measure regional differences in resin-dentin bond strength, and the ability to measure the higher bond strength of newly developed materials [25].

Table 3. Results of Two-way ANOVA test to compare microtensile bond strength means among three adhesive systems applied on 48 extracted premolars (two specimen from each tooth) with and without cyclic loading.

Source	df	F	P value
Dentin bonding agent	1	34.2	0.001 (S)
Load Cycling	1	64.7	0.001 (S)
Dentin bonding vs. Load cycling	2	1.93	0.15 (NS)

S=significant, NS=not significant, df=degrees of freedom, F= F-statistics

It should be emphasized that randomized controlled clinical trials are the most reliable way to assess the long-term behavior of the adhesive systems. Therefore, further researches in this field are recommended.

CONCLUSION

Under the limitations of the present study, MTBS of Clearfil tri-S Bond and Xeno III dentin adhesives was significantly more than I Bond dentin adhesive. Moreover, load cycling influenced adversely bond strength of these dentin adhesives.

ACKNOWLEDGMENTS

The authors wish to thank Dr. Mohammad Hossinpour for his help in performance of this research.

REFERENCES

1-Nakabayashi N, Ashizawa M, Nakamura M. Identification of a resin-dentin hybrid layer in vital human dentin created in vivo: durable bonding to vital dentin. Quintessence Int 1992 Feb;23(2):135-41.

2-Yeşilyurt C, Bulucu B. Bond strength of total-etch and self-etch dentin adhesive systems on peripheral and central dentinal tissue: a microtensile bond strength test. J Contemp Dent Pract 2006 May 1;7(2):26-36.

3-Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent 2003 May-Jun;28(3):215-35.

4-Koliniotou-Koumpia E, Dionysopoulos P, Koumpia E. In vivo evaluation of microleakage from composites with new dentine adhesives. J Oral Rehabil 2004 Oct;31(10):1014-22.

5-Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, et al. Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials 2007 Sep;28(26):3757-85.

6-Celiberti P, Lussi A. Use of a self-etching adhesive on previously etched intact enamel and its effect on sealant microleakage and tag formation. J Dent 2005 Feb;33(2):163-71.

7-Perdigão J. New developments in dental adhesion. Dent Clin North Am 2007 Apr;51(2):333-57

8-Bradna P, Vrbova R, Dudek M, Roubickova A, Housova D. Comparison of bonding performance of self-etching and etch-and-rinse adhesives on human dentin using reliability analysis. J Adhes Dent 2008 Dec;10(6):423-9.

9-Davidson CL, Abdalla AI. Effect of occlusal load cycling on the marginal integrity of adhesive Class V restorations. Am J Dent 1994 Apr;7(2):111-4.

10-Kubo S, Yokota H, Sata Y, Hayashi Y. The effect of flexural load cycling on the microleakage of cervical resin composites. Oper Dent 2001 Sep-Oct;26(5):451-9.

11-Nikaido T, Kunzelmann KH, Chen H, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R, Tagami J. Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin. Dent Mater 2002 May;18(3):269-75.

12-Bedran-de-Castro AK, Pereira PN, Pimenta LA, Thompson JY. Effect of thermal and mechanical load cycling on microtensile bond strength of a total-etch adhesive system. Oper Dent 2004 Mar-Apr;29(2):150-6.

13-Abdalla AI, El Zohairy AA, Aboushelib MM, Feilzer AJ. Influence of thermal and mechanical load cycling on the microtensile bond strength of self-etching adhesives. Am J Dent 2007 Aug;20(4):250-4.

14-Monticelli F, Osorio R, Pisani-Proença J, Toledano M. Resistance to degradation of resin-dentin bonds using a one-step HEMA-free adhesive. J Dent 2007 Feb;35(2):181-6.

15-Cho BH, Dickens SH. Effects of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength. Dent Mater 2004 Feb;20(2):107-15.

16-Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Tagami J, et al. Long-term durability of dentin bonds made with a self-etching

primer, in vivo. J Dent Res 1999 Apr;78(4):906-11.
17-Yoshikawa T, Sano H, Burrow MF, Tagami J, Pashley DH. Effects of dentin depth and cavity configuration on bond strength. J Dent Res 1999 Apr;78(4):898-905.
18-Lodovici E, Reis A, Geraldeli S, Ferracane JL, Ballester RY, Rodrigues Filho LE. Does adhesive thickness affect resin-dentin bond strength after thermal/load cycling? Oper Dent 2009 Jan-Feb; 34(1):58-64.
19-Mitsui FH, Peris AR, Cavalcanti AN, Marchi GM, Pimenta LA. Influence of thermal and mechanical load cycling on microtensile bond strengths of total and self-etching adhesive systems. Oper Dent 2006 Mar-Apr;31(2):240-7.
20-Bedran-De-Castro AK, Pereira PN, Pimenta LA. Long-term bond strength of restorations subjected to thermo-mechanical stresses over time. Am J Dent 2004 Oct;17(5):337-41.
21-Tonami K, Takahashi H. Effects of aging on tensile fatigue strength of bovine dentin. Dent Mater J 1997 Dec;16(2):156-69.
22-dos Santos PH, Sinhoreti MA, Consani S, Sobrinho LC, Adabo GL, Vaz LG. Effect of cyclic compressive loading on the bond strength of an adhesive system to dentin after collagen removal. J Adhes Dent 2005 Summer;7(2):127-31.
23-Nikaido T, Kunzelmann KH, Ogata M, Harada N, Yamaguchi S, Cox CF, et al. The in vitro dentin bond strengths of two adhesive systems in class I cavities of human molars. J Adhes Dent 2002 Spring;4(1):31-9.
24-Bedran-de-Castro AK, Cardoso PE, Ambrosano GM, Pimenta LA. Thermal and mechanical load cycling on microleakage and shear bond strength to dentin. Oper Dent 2004 Jan-Feb;29(1):42-8.
25-Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. Dent Mater 1994 Jul;10(4):236-40.