

Micro-shear Bond Strength of a Nanofiller Bonding agent with and without Thermocycling in a Newly Invented Device

Z. Jaber Ansari ¹, H. Khalili ², MA. Keshavarz Tork ³, M. Amiri Siavashani ⁴, 

¹Associate Professor, Department of Restorative Dentistry, School of Dentistry, Shahid Beheshti University of Medical Sciences, Tehran, Iran

²Assistant Professor, Department of Computer Engineeri, School of Engineeri, Shahid Beheshti University. Tehran, Iran

³Dentist

⁴Resident, Department of Restorative Dentistry, School of Dentistry, Shahid Beheshti University of Medical Sciences, Tehran, Iran

Abstract

Background and Aim: Estimation of the survival of dental materials is especially important when manufacturing new materials. Thermocycling can greatly help in this respect. The purpose of this study was to evaluate the microshear bond strength of a nanofiller bonding agent with and without thermocycling in a newly invented device.

Materials and Methods: In this experimental study, human third molars were sectioned into 1.5 mm slices after extraction and disinfection. Clearfil Tri-S nanofiller bond was applied to the dentin part of sections according to the manufacturer's instructions. Clearfil AP-X composite resin was placed over the dentin using tubes with 0.75 mm internal diameter and one mm height and light cured. Specimens were randomly divided into three groups and subjected to thermocycling in a newly invented device for zero, 3000 and 5000 cycles between 5±2°C and 55±2°C. Micro-shear bond strength was measured by microtensile tester with a cross-head speed of 0.5 mm/min. Obtained data were analyzed using one-way ANOVA. Tukey's test was used for multiple comparisons with a 95% confidence interval.

Results: The mean micro-shear bond strength after zero, 3000 and 5000 thermal cycles was found to be 19.27±4.56, 17.00±6.52 and 11.58±4.64 MPa, respectively. The reduction in bond strength between zero and 3000 thermal cycles was not statistically significant (p=0.3) but this reduction between zero and 5000, as well as 3000 and 5000 cycles was statistically significant (p<0.002 and p<0.03, respectively).

Conclusion: Increase in number of thermal cycles for more than 3000 reduces the micro-shear bond strength of Tri-S bonding agent.

Key Words: Micro-shear bond strength, Bonding, Thermal cycles, Nanofiller

 Corresponding author:
M. Amiri Siavashani, Resident,
Department of Restorative Dentistry, School of Dentistry, Shahid Beheshti University of Medical Sciences, Tehran, Iran *Manusha_amr@yahoo.com*

Received: 28 Oct 2012
Accepted: 29 June 2013

Journal of Islamic Dental Association of IRAN (JIDAI) Autumn 2013 ;25, (4)

Introduction

The science of dental materials progresses by the invention of new restorative materials and improvement of the properties of those currently available. Improper application or weak formulation of dental materials can cause patient discomfort, side effects, complications or increased cost of health and dental care services. Stability, survival and longevity are important factors when it

comes to the quality of materials. Survival of a restoration necessarily depends on its optimal marginal seal. Reducing the marginal microleakage decreases the risk of secondary caries, marginal ditching, post-operative hypersensitivity and pulp involvement. Thus, evaluation of microleakage is an important criterion for the assessment of newly produced materials and can help in improving their quality [1]. Restorative materials in the oral cavity

are constantly subjected to extreme thermal changes due to the consumption of food and beverages with varying temperatures. To assess marginal microleakage, the effect of these thermal changes on the bond strength of composite resin to enamel and dentin should be evaluated in conditions simulating oral cavity [2]. Experimental models like thermocycling, application of mechanical forces, pH cycles and aging in distilled water, NaOCl and solutions simulating food products can provide us with valuable information regarding basic mechanisms affecting the tooth-resin interface [3]. Several studies have evaluated the quality of restorations such as their bond strength under certain conditions simulating thermal changes. Also, due to the invention of new restorative materials such as nanofiller and nanocomposites, their assessment in conditions simulating thermal changes of the oral cavity seems necessary [4].

On the other hand, with the development of a new generation of dentin bonding agents and introduction of one-step self-etch systems as well as bonding agents with very high nanofiller content [5] such as Clearfil Tri-S bond, concerns about the destructive effects of humidity and thermal changes of oral cavity on these materials have raised.

Mousavinasab et al. evaluated the effect of water storage and thermocycling on microshear bond strength of total etch and self-etch systems and reported that the microshear bond strength and survival of Clearfil SE bond were within an acceptable range [6].

Nagayassu et al, in 2011 assessed the microshear bond strength of 5 adhesive systems to enamel: Single Bond 2; Clearfil SE Bond; Adhe SE; Adper Prompt L-Pop; and Xeno III. They stored the samples in distilled water at 37°C for 24h and then subjected them to thermocycling. However, they failed to find a significant difference between them in terms of their microshear bond strength to enamel [7].

Resistance of dental materials to destruction plays an important role in their clinical service; thus, evaluation of the effects of experimental methods such as thermocycling on microshear bond strength is necessary to foresee the clinical behavior of restorative materials and bonding agents overtime in the oral cavity.

Considering all the above, the present study was conducted to assess the microshear bond strength of a nanofiller bonding to dentin with and without thermocycling in a newly invented device.

Materials and Methods

This experimental study was conducted on human sound third molars. After surgical extraction, the teeth were stored in saline solution for 2 months. Twenty-four hours before the conduction of experiment, the teeth were observed visually and examined by an explorer and sound teeth with no restoration, caries, fracture or crack were selected for the study. After cleaning with a rotary brush and pumice paste, the teeth were stored in 0.05% thymol solution for disinfection. Each tooth was longitudinally sectioned parallel to its long axis into 1.5 mm slices using Hamco thin sectioning machine (Hamco Machines Inc., Rochester, NY, USA). The sectioned surfaces were polished with 600 grit silicon carbide abrasive paper under running water for 30s. Clearfil Tri-S one-step self-etch nanofiller bonding agent was applied to the prepared surfaces according to the manufacturer's instructions. The bonding was first applied to the surface using an applicator. After 20s, the surface was dried using high-pressure air spray for more than 5s to spread a thin layer of bonding over the surface. Next, several Tygon tubes (Tygon, Norton Plastic, Cleveland, OH, USA) with an internal diameter of 0.75 mm and length of 1mm were placed over dentin surface and light cured for 10s. The tubes were filled with A2 shade AP-X composite resin (Kuraray, Japan) and light-cured for 40s. Specimens were then immersed into saline solution. After an hour, the Tygon tubes were extracted and the specimens were again immersed in saline solution for 24h. The light-curing device used in this study was Aryalux light curing unit (Apadana Tak) with an intensity of more than 650 m W/cm². To measure bond strength, specimens were first evaluated under a light stereomicroscope (Olympus model SZX-ILLB2000, Japan) at 30X magnification to detect bonding defects. Defective specimens (voids or gaps at the bonding agent-composite resin interface) were excluded from the study and replaced with sound specimens. Samples were then randomly divided into 3 groups of 11 each and

subjected to thermocycling in a recently invented device for zero, 3000 and 5000 cycles at 5 ± 2 and $55\pm 2^\circ\text{C}$. Each cycle contained three phases of hot water bath for 15s, cold water bath for 15s and a dwelling time of 15s. Our invented device had several advantages over the available ones including the followings:

1. Digital control of the device
2. A key pad and display screen making the device more user friendly
3. Software program with easy to use menu and voice commands on the screen and the ability to change the program at any time
4. Displaying the temperature of the bath and work status of the device on the screen
5. Automatic control of the water level of baths
6. Precise control of the temperature of water baths with maximum error of one degree
7. Automatic function of mechanical arm between the two baths hot and cold
8. The ability to test materials with up to 250 cm³ volume and 1.2 kg weight
9. The ability to perform 30,000 cycles continuously without the presence of user
10. Automatic draining of water baths if required
11. Alarm system to go off in case of any error
12. RAM memory to save information about thermal cycles
13. Continuation of function in case of power interruption
14. Having an emergency valve for overflow of water in baths

Microshear bond strength of specimens was measured using a microtensile tester (Bisco Wire and Loop with a tensile force of 200N at a crosshead speed of 0.5 mm/min. The amount of load at failure was recorded in Newton and the microshear bond strength was calculated by dividing the applied force at the time of failure (N) to the cross section of specimens (mm²) and recorded in MPa.

After measuring the bond strength, the mode of failure was evaluated under stereomicroscope in the Pathology Department and fell into one of the following categories of adhesive failure (between the dentin and composite resin), cohesive failure (within the composite) or mixed (a combination of adhesive and cohesive failures).

Data were statistically analyzed using one-way ANOVA and Tukey's test.

Results

The mean microshear bond strength of Clearfil Tri-S bond was 19.27 ± 4.56 MPa in specimens without thermocycling. This rate was 17 ± 6.52 MPa in specimens that underwent 3000 thermal cycles and 11.58 ± 4.64 in specimens subjected to 5000 cycles.

Comparison of the microshear bond strength of this bonding at three groups of zero, 3000 and 5000 thermal cycles using ANOVA revealed significant differences in microshear bond strength among the three groups ($P<0.006$).

Considering the significance of the results of ANOVA, Tukey's test was used for pairwise comparison of groups. ANOVA failed to find a significant difference between groups with zero and 3000 cycles ($P=0.3$) while the difference between 3000 and 5000 and also zero and 5000 cycles was statistically significant ($P<0.03$ and $P<0.002$, respectively). Evaluation of mode of failure with stereomicroscope revealed that the majority of specimens fractured at the bonding agent-tooth interface (adhesive type).

Discussion

In the recent decade, most studies have focused on the use of one-step self-etch bonding agents due to their simple application. In these systems, the etchant, primer and bonding agent are applied to the tooth surface all at the same time followed by the application of composite resin [8]. In other words, researchers manufactured the one-step self-etch adhesives by using molecular technology. Using this technology, they incorporated hydrophobic and hydrophilic components of two-step bonding agents into one compound in a stable state. The one-step agents are able to decalcify the surface and well penetrate into the tooth structure. Thus, no void will form on the surface of adhesive enamel. Clearfil Tri-S bond is a one-step self-etch agent with a very high nanofiller content. Its composition includes: MDP, Bis-GMA, HEMA silanized colloidal silica, dimethacrylate, 10-camphorquinone, alcohol and water. The liquid components (monomer, water, ethanol) and the nanofillers are fluid at first, but after air spraying, the solvent evaporates, the adhesive hardens and provides a stable bond. Hydrophilic monomers present in single-step adhesives are usually not able to polymerize sufficiently and thus cause a

weak bond; but due to high filler content this bonding agent is able to cause a strong durable bond [5].

In our study, the mean microshear bond strength was 19.27, 17.00 and 11.58 MPa in the three groups of zero, 3000 and 5000 thermal cycles, respectively. Results showed that bond strength after 3000 thermal cycles was not significantly different from the bond strength in specimens that did not undergo thermocycling ($P=0.3$). But, a significant difference in bond strength was noted between specimens subjected to 5000 thermal cycles and the control specimens ($P<0.002$). The difference in bond strength between 3000 and 5000 thermal cycles was statistically significant as well ($P<0.03$). Thermocycling causes artificial aging of the bonding system. Two mechanisms of hydrolysis and bond dissolution occur at the interface because of hot water, repeated expansion and shrinkage and stress accumulation due to thermal changes [9,10]. Studies by Nikaido and Frankenburger showed that thermocycling along with mechanical loads reduce the bond strength of composite resin restorations [11, 12].

Controversy exists regarding the number of thermal cycles and their effects on bond strength. According to LinoCarracho, 200 thermal cycles caused a significant reduction in bond strength of two bonding systems [13]. Masahi reported that 3000 and 10,000 thermal cycles had no effect on bond strength of his understudy materials and only after 30,000 thermal cycles a significant reduction occurred in two bonding agents [14].

Gale stated that 10,000 thermal cycles correspond to one year of clinical service of a restoration [9]. Despite all the above, ISO.TR.114.SO announced that 500 thermal cycles at 5-55°C is a suitable artificial aging test [8].

In our study, the microshear bond strength of Clearfil Tri-S bond was 19.27 MPa without thermocycling; which is similar to the result of Wang who evaluated the bond strength of Clearfil Tri-S bond to enamel and dentin [15]. This value is higher than the rate reported by Masahi who reported the microshear bond strength of single bond and Prime & Bond systems to be 15 and 12MPa, respectively [14]. However, our rate is much lower than the rate reported by Shimada [16].

Jaberi Ansari [8] and Kasraei [17] in their studies evaluated the microshear bond strength of Clearfil Tri-S bond to dentin after 24h without thermocycling and reported it to be 35.99 and 29.99 MPa, respectively. These rates are significantly different from our obtained rate although the same materials were used. The difference in this respect may be attributed to the lack of thermocycling in their study or difference in methodology of studies. For example, in our study, specimens were immersed into saline solution immediately after composite bonding which might have interfered with the final bond. Experience, expertise and skills of the operator may play a role as well.

Lower bond strength of one-step materials may be explained by the fact that in these systems water and bond solvents like alcohol and acetone are present altogether. After applying the bonding agent and before curing, water and solvents are expected to evaporate by the use of air spray. If, for any reason, this does not occur, polymerization is compromised resulting in a reduction in bond strength [18]. On the other hand, microshear bond strength testing is very accurate because of the small testing area. The smaller the surface, the lower the risk of errors. However, working with small surfaces requires higher precision [19] and technical errors will have a greater effect on the outcome. Furthermore, duration and media of storage of teeth before the experiment play a role in bond strength. When stored in saline solution, the hardness of enamel and dentin decreases due to the loss of their surface calcium content. The longer the storage period, the greater the loss of hardness [15, 20]. Storage in distilled water will have a less softening effect on tooth structure.

Takaya in 2008 reported the microshear bond strength of Clearfil Tri-S bond to dentin to be 10.7 MPa after 24h without thermocycling. This value was 12.2 MPa after 7 days without thermocycling and 7.2 MPa after 20,000 thermal cycles [21]. These results are different from our findings. This difference may be due to the fact that in Takaya's study, dentin specimens were obtained from bovine teeth; which has a different composition than human dentin. Also, before testing the teeth were stored in a dry environment and number of thermal cycles in their study was much more than in our study.

Burger et al, in 2007 evaluated the effect of increasing the number of thermal cycles (100, 500, 1000, 2000 and 4000) on microshear bond strength of bonding to dentin and found no statistically significant difference in this respect between groups. Similarly, we found no significant difference in bond strength between the two groups of zero and 3000 thermal cycles ($P=0.3$); however, the difference between 3000 and 5000 cycles was statistically significant ($P<0.03$). There is a possibility that increasing the number of thermal cycles to more than 4000 could have yielded a different result in Burger's study [22].

In a study by Mousavinasab et al, in 2009 microshear bond strength of Clearfil SE Bond was 33.2 MPa after one day of water storage and 31.5 MPa after thermocycling (3000) and 3 months of water storage. These rates are greater than our obtained rate. This finding may be due to the use of two-step CSE self-etch bonding system [6].

Evaluation of mode of failure by stereomicroscope revealed that 71.9% of failures were of adhesive type indicating that the weak point of the system is at the bonding-tooth interface. Future studies should focus on developing bonding agents with a more reliable bond to tooth structure.

Conclusion

Increasing the number of thermal cycles had a negative impact on Clearfil Tri-S bond to tooth structure and the most common mode of failure was adhesive type.

Acknowledgement

This study was part of the doctoral thesis of Mohamamd Ali KeshavarzTork; the thesis supervisor was Dr. Zahra Jaberi Ansari and the consulting professor was engineer Hamid Khalili. The authors would like to thank all the instructors who cooperated in the conduction of this study.

References

- 1- Prabhakar AR, Madan M, Raju OS. The marginal seal of a flowable composite, an injectable resin modified glass ionomer and a compomer in primary molars- An in vitro study. *J Indian Soc Pedod Preve Dent*. 2003 Jun; 21(2):45-8.
- 2- Huang MS, Ding SJ: The effect of thermocycling and dentin pretreatment on the durability of the

bond between composite resin and dentin. *J Oral Rehabil*. 2004 May; 31(5):492-9.

- 3- Amaral FL, Colucci V, Palma-Dibb RG, Corona SA. Assessment of in vitro methods used to promote adhesive interface degradation: A critical review. *J Esthetic Rest Dent*. 2007 Nov, 19(6):340-353.

- 4- Lee H, lim BS, Rhee SH, Yang Hc, Powers JM. Changes of optical properties of dental nano-filled resin composites after curing and thermocycling. *J Biomed Mater Res. B Apple Biomater*. 2004 Oct; 71(1):16-21.

- 5- Poptani B, Gohil KS, Shukla M. Microtensile dentin bond strength of fifth with five seven-generation Dentin bonding agents after thermocycling: An in vitro study. *Contemp Clin Dent*. 2012 Sept, 3(Suppl2):S167-S171.

- 6- Mousavinasab SM, Farhadi A, Shabani M. Effect of storage time, thermocycling and resin coating on durability of dentin bonding systems. *Dent Res J*. 2009 Spring; 6(1):29-37.

- 7- Nagayassu MP, Shintome LK, Arana-Chavez V E, Fava M. Micro-shear bond strength of different adhesives to human dental enamel. *J Clin Ped Dent*. 2011 Spring, 35(3):301-304.

- 8- Jaberi Ansari Z, Sadr A, Moezizade M, Amnian R, Ghasemi A, Tagami J, Shimada Y, Jaberi Ansari Sh, Moayedi S. Effect of one-year storage in water on bond strength of self etching adhesives to enamel and dentin. *Dent Mat J*. 2008 Mar; 27(2):266-72.

- 9- Gale MS, Darwell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent*. 1999 Feb; 27(2):89-99.

- 10- Lelou PG, Dhoore W, Bouter D, Derange M, Hardo N Yamaguchi S. Metaanalytical review of factors involved in dentin adherence. *J Dent Res*. 2002 Jul; 80(7):1605-14.

- 11- Frankenberger R, Pashley DH, Reich SM, Lohbauer U, Pechelt A, Tay F. Characterization of resin-dentin interfaces by compressive cyclic loading. *Bio Mat*. 2005 May; 26(14):2043-52.

- 12- Frankenberger R, Strobel WO, Karmer N, Lohbauer U, Winterscheidt B, Winterscheidt J, Pechelt A. Evaluation of the fatigue behavior of the resin-dentin bond with the use of different methods: *J Biomed Mater Res*. 2003 Nov; 67(2): 712-21.

- 13- LinoCarracho A. The effect of storage and thermocycling on the shear bond strength of three dental adhesives. Quintessence 1991 Sept; 2(9): 745-52.
- 14- Masahi M. Influence of thermocycling on dentin bond strength of two step bonding systems. AM J Dent. 1998 Jun; 11:118-122.
- 15- Wang H, Shirnada Y, Tagami J. Shear bond stability of current adhesive system to enamel. Oper Dent. 2004 Mar-Apr; 29(2):168-71.
- 16-Shimada Y, Senawongse P. Effective bond strength of current adhesive systems on deciduous and permanent dentin. Oper Dent. 2004 Mar-Apr; 29(2):196-202.
- 17- Kasraei Sh, Khamverdi Z. Effect of Mechanical load cycling on Microtensile bond strength of Self-Etch Systems to Dentin. Journal of Dentistry of Tehran University of Medical Sciences 2008 Autumn; 5(4):173-178.
- 18- Sadr A, ShimadaY, Tagami J. Bond strengths of two adhesive and effects of application methods. J Dent Res. 2005 Mar; 84(10):150-6.
- 19- Shirnada Y, Yamaguchi S, Tagami J. Micro-shear bond strength of dual-cured resin cement to glass ceramics. Dent Mater. 2002 Jul;18(5):380-8.
- 20- Roberson TM, Heyman H, Swift EJ. Art and science of operative dentistry. 4th ed. St. Louis: The C. V Mosby Co; 2002, 237-61.
- 21-Takaya I, Naako O, Ak.ko O, Hiroyasu K. Bond Strength to bovine dentin of a composite core build-up material combined with four different bonding agents. J Oral Sci. 2008;50(3):329-333.
- 22-Burger KM, Cooley RL, Garcia-Godoy F. Effect of thermocycling times on dentin bond strength bond. J Esthet Rest Dent. 2007 July 1; 4(6):197-198.