Micro-tensile bond strength of one-step adhesives to dentin

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Citation

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Abstract

Purpose: To evaluate the micro-tensile bond strength of 2 one-step adhesive systems and a 1 two-step etch-and-rinse to dentin before and after thermal cycling.
Materials and Methods: Occlusal surfaces of 30 extracted human molars were prepared using diamond tips to expose flat dentin surfaces. Two one-step, G-Bond
(GB) and Adper Prompt L-Pop (APL), and 1 two-step, Excite
(Ex), adhesive systems were applied to bond composite to the prepared dentin surfaces. The prepared specimens were sectioned for micro-tensile bond strength testing (μTBS). The de-bonding patterns of the fractured specimens were also analyzed.
Results: The one-way ANOVA test indicated presence of differences between the bond strength of different adhesives to dentin before and after thermal cycling (ANOVA, $p < 0.001$). There was no statistical difference between the 2 one-step adhesives (Tukey's comparison, $P > 0.05$) before or after thermal cycling. This difference was not statistically significant (Tukey's comparison, $P > 0.05$) when the bond strength of unthermal-cycled G-bond was compared with the control (Excite) or the thermal-cycled G-bond was compared with the thermal-cycled (Excite). The admix (adhesive/cohesive) de-bonding was the dominant pattern of specimens' failure.
Conclusion: The one-step adhesive APL does not perform worse regarding μTBS, (16.3 ± 2.7) than the newer one GB (18.4 ± 4.9). The μTBS of GB is also comparable to that of the control, Ex two-step adhesive (20.9 ± 2.4). Thermal cycling has no statistically significant deteriorating effect on the performed bond of different adhesives under investigation, APL-Th (13.1 ± 1.2), GB-Th (14.7 ± 2.4), Ex-Th (18.4 ± 1.7).

INTRODUCTION

Restoring teeth with minimal sacrifice of sound tooth structure currently forms the basis of restorative dentistry. Essential in achieving this goal is the adhesive that provides strong and durable bond to the remaining sound tooth tissues especially when shrinking materials such as resin composites are planned to be used. [1,2]

The early successful adhesive systems have typically accomplished resin-dentin bonding in three steps respectively are the etching, priming, and application of bonding resin. [3] Using these systems, the quality of the created bond is greatly influenced by the duration of the etching process, and by the amount of dentin surface humidity following rinsing of the etching acid and prior to resin infiltration. [4] Therefore, most of the recent researches and developments in dentin adhesion are directed to simplify the bonding procedures and to eliminate all possible technical sensitivities by reducing the number of bonding steps. These developments have been started when the primer and bonding resins were combined together in one bottle. The self-etching primers were then released with the ability to etch and prime the dentin in one step. [4,5]

The self-etching approach seemed promising as it reduces the chair-side time, and eliminates the critical and difficult standardization of the bonding steps. [6] The one-step self-etch adhesives were subsequently introduced simplifying the conditioning, priming, and bonding procedures just in a single step. However, the early types of these adhesive systems seemed to achieve lower bond strength values in comparison to the two-step systems. [7,8] Newer types of the one-step self-etching adhesive systems have recently been introduced to the market claiming to have higher bond strength via formation of an unusual very thin interaction layer. [9,10] A conflict was raised regarding the success of these

new one-step self-etching (all-in-one) adhesives as it depends on the specific composition of each product. Therefore, it was suggested that these new adhesives are in need for more screening before recommending them for clinical use. [11]

The micro tensile test is increasingly used to evaluate the strength of such tooth-adhesive bond. [12,13,14,15] This test provides a purely tensile load on a very small cross-section of the bonding interface regardless the specimens' design. [16] Over such a limited surface, stress distribution is expected to be uniform, thus enabling the test measurements to truly express the interfacial bond strength. [13,14,15] In addition, the micro tensile method has allowed the mapping of bond strength in different regions or at different depths of dental tissues. [17,18]

To stand on the efficiency of the new all-in-one adhesives bonding to dentin, this in vitro study aimed to evaluate the micro-tensile bond strength (μTBS) of 2 one-step self-etching adhesive systems to dentin before and after thermal cycling. The bond strength of a 1 two-step adhesive system was also considered to act as control. A microscopic analysis of the fractured surfaces was also carried out to detect the exact mode(s) of bond failure.

MATERIALS AND METHODS

Thirty caries-free freshly-extracted human molars were selected for this study. The collected teeth were cleaned (Pro-sonic 300 MTH, Sultan Chemists Inc, Englewood, NJ) and stored in de-ionized water that contained antibacterial agent (0.2% sodium azide) for a maximum of one month before trimming their occlusal anatomy (LabMaster, Ray Foster Dental Equipment, Huntington Beach, CA) to prepare flat dentin surfaces. The flattened surfaces were then finished using long cylindrical diamond tips (F31273, EDENTA GmbH, Lustenau) mounted to high speed handpiece. Immediately after finishing and before performing the bonding procedure, the flat dentin surfaces were subjected to thorough cleaning using air-water spray. The prepared teeth were dried with air then equally divided into 3 main groups (n=10 for each adhesive system). The description and manufacturers of the materials used are shown in Table 1.

Figure 1

Table 1: Materials used in this study

Material	Description	Composition	Manufacturer
I. Adhesive systems:			
G-Bond (GB)	1-step, Self-etch adhesive	Phosphoric acid ester monomer, UDMA, 4-MET, TEGMA, acetone, water, initiators	GC America, St. Alsip, IL, USA
Adper Prompt L-Pop (APL)	1-step, Self-etch adhesive	Compartment 1 : Methacrylated phosphoric acid esters, photoinitiator, stabilizers Compartment 2 : Water, HEMA, polyalkenoic acid, stabilizers	3M/ESPE Dental Products, St. Paul, MN, USA
Excite (Ex)	2- step, etch and rinse adhesive	HEMA, DMA, phosphoric acid acrylate , highly dispersed silicone dioxide, initiators and stabilizers in alcohol solution	Ivoclar Vivadent, Schaan, Liechtenstein
II. Restorative material:			
Tetric Ceram	Light-curing, fine-particle hybrid resin composite	BisGMA, UDMA, TEGDMA, ytterbium trifluoride, barium glass , pigments and initiators	Ivoclar Vivadent

For the control group; one 2-step single component adhesive system Excite was used to retain the Tetric Ceram composite to dentin. The flat surfaces of the prepared teeth were etched using 37% phosphoric acid (Vivadent, Schaan, Liechtenstein) for 15 s, washed under copious air-water spray and gently dried using cotton pellets. Two successive coats of the single component resin adhesive were then applied to the moist dentin surfaces using a brush for 10 s and cured by the aid of Hawe-Neos halogen light-curing device (Gentilino, Switzerland) providing intensity of 600 mW/ cm^2 for another 20 s after air drying of the excess material. Two one-step, self-etch (G-Bond); and (Adper Prompt L-Pop) adhesive systems were used in the other 2 groups. The self-etch bonding resins were applied to the prepared dentin surfaces and left for 10s before air thinning. Curing of both self-etching adhesives was performed using the same light-curing device for 10s.

The composite material was then incrementally built up to be 2 mm above the flat occlusal surfaces. Each increment was light-cured for 40 s and a rubber mold 8 mm in diameter and 2 mm high, situated over the flat occlusal plane of each prepared tooth, aided in both building and contouring the composite restoration. The restored teeth were incubated in water at 37 1 ° C for 24 h and half of them (5 teeth from each group) were also subjected to thermo-cycling (Th) (Willytec Thermocycler 10714, Munich, Germany) at 5 176; C, 37 ° C and 55 ° C for 5000 cycles with 30-s dwell time resulting in 6 groups. [19,20]

MICRO-TENSILE BOND STRENGTH TESTING

Roots of the restored teeth were implanted in plastic rings, 1.5 cm in diameter, by means of self-cured acrylic resin (Duracrol, Sofa-Dental, Prauge, Czech Republic). These rings kept the teeth properly oriented at the time of sectioning. The composite build-ups and the underlying dentin were sectioned in both buccolingual and mesiodistal directions. The sectioning process was carried out the same way as that of El-Kholaney et al., [21] using diamond disks (Edenta Gmbh, Lustenau, Austria) in straight handpiece fixed to a specially-designed bench-mounted orienting apparatus, under air-water spray cooling. The cutting process resulted in sticks of nearly 1 mm^2 cross-sectional area. The sticks were then separated from the dentin base and carefully observed to select 10 sticks from each group of samples. The selected sticks were again incubated for 24 h before conducting the bond strength testing. The exact dimensions of each stick were measured using a digital caliper (Model CD-S6 CP, Mitutoyo Corp., Japan) before they were affixed with Zapit-brand cyanoacrylate adhesive (Dental Ventures of America, Ventura, USA) to the specially designed jigs of the universal testing machine (Type 500, Lloyd instrument, England). Sticks were stressed to failure under tension at a crosshead speed of 2mm/min. The micro-tensile bond strength for each sample was calculated in MPa by dividing the maximum force at fracture in Newton by the sample's cross-sectional area in mm^2 .

ASSESSING THE MODE OF SPECIMENS' FRACTURE

The 2 parts of the tested sticks were evaluated for the mode of bond failure using a stereoscope microscope (Olympus Zoom Stereomicroscope, Sz 40-45 TR, Japan) at 30 original magnification. The evaluation process took place by every author included in this study. Their results were compared and the detected differences were discussed before nominating one author to reexamine the sticks for the second time considering his results as final. The detected modes of failure were classified as adhesive when the fracture site was entirely within the adhesive/ dentine or adhesive/ composite interface; cohesive, when the fracture occurred exclusively within the resin composite or dentin; or mixed when the fracture site continued from the adhesive into either the resin composite or dentin. Some fractured samples (n=5) were randomly selected for further scanning electron microscopic observation (SEMx1000) at 30 KV (JEOL, JSM, 5600LV, Tokyo, Japan).

STATISTICAL ANALYSIS

Bond strength data of the thermal-cycled and un-thermal-cycled samples of all groups were subjected to statistical analysis using SPSS statistical package version 10 one-way ANOVA (P= 0.05) to detect any differences existing between the adhesive systems under both conditions. The Tukey's comparisons (P= 0.05) were then used to show the significance of those differences detected between all groups.

RESULTS

Means and standard deviations of the recorded micro-tensile bond strengths are shown in table 2. The one-way ANOVA (Table 2) indicated presence of significant differences ($p < 0.001$) between the bond strength values of different adhesives to dentin before and after thermal cycling. No statistical difference was detected between the 2 one-step adhesives (Tukey's comparisons, $P > 0.05$) under the same testing conditions (Table 3), APL (16.3 ± 2.7 MPa), APL-Th (13.1 ± 1.2 MPa), GB (18.4 ± 4.9) and GB-Th (14.7 ± 2.4). This difference was not statistically significant (Tukey's comparison, $P > 0.05$) when the bond strength of unthermal-cycled G-bond was compared with the control Excite (20.9 ± 2.4) or the thermal-cycled G-bond was compared with the thermal-cycled Excite (18.4 ± 1.7). Both adhesive and admix (adhesive/cohesive) types of bond failure were evident as revealed by the stereomicroscope, (Figure 1). However, SEM images (Figures 2a-c) indicated that the majority of bond failures belong to the admix pattern.

Figure 2

Table 2: Microtensile bond strength in MPa of different adhesives

	Groups					
	Ex	APL	GB	Ex-Th	APL-Th	GB-Th
Bond strength values ± SD	20.9 ± 2.4	16.3 ± 2.7	18.4 ± 4.9	18.4 ± 1.7	13.1 ± 1.2	14.7 ± 2.4

Excite (Ex), Adper L-Pop (APL), G-Bond (GB) and Thermocycling (Th)

Figure 3

Table 3: Tukey's comparisons between different groups

Groups	Ex	APL	GB	Ex-Th	APL-Th	GB-Th
Ex	0	0.04493*	0.5731	0.5489	0.00022*	0.00255*
APL		0	0.7234	0.7456	0.2805	0.8783
GB			0	1	0.01245*	0.1495
Ex-Th				0	0.01376*	0.1611
APL-Th					0	0.8932
GB-Th						0

* Significantly different groups

Figure 4

Figure 1: Percentage of bond failure

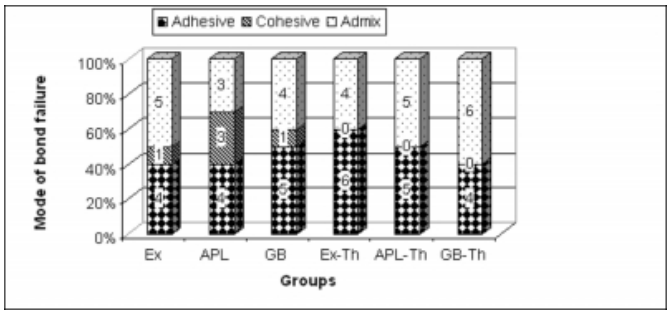


Figure 5

Figure 2: SEM Photographs (x 1000) of specimens after ÂµTBS testing demonstrating a mixed failure mode (adhesive/cohesive). (a) GB-bonded specimen. (b) APL-bonded specimen showing cracks in the adhesive layer (pointer) as well as voids at the interface (arrows). (c) Ex-bonded specimen. Hybrid layer (H) Adhesive layer (A)

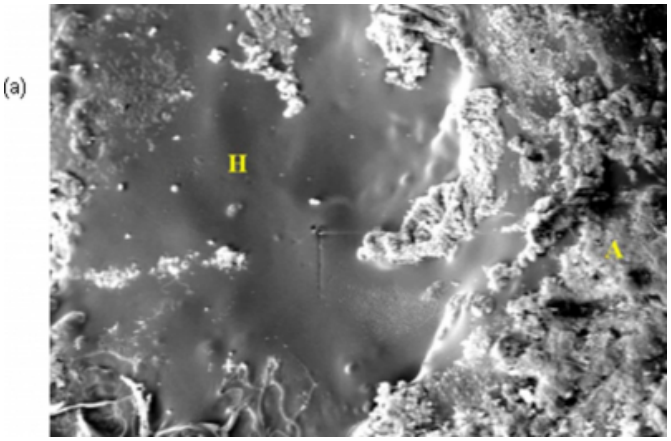


Figure 6

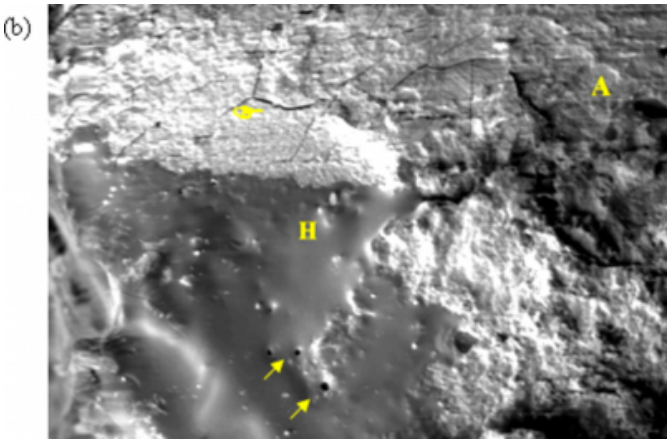
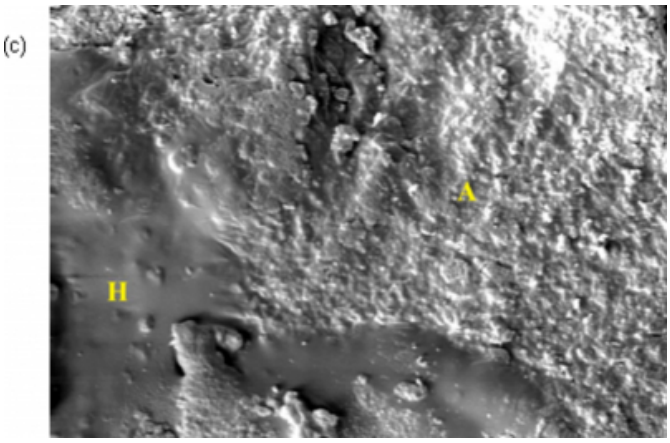


Figure 7



DISCUSSION

Adhesion to tooth structure usually provides a great opportunity for more conservative restorations. [2] However, the successful adhesive should fulfill the minimal acceptable level of bond strength that helps to resist both polymerization and thermal stresses at the bonding interface. [22] Recently, a great concern about technique sensitivity and time consuming of adhesives has been developed and this has initiated the development of newer generations with reduced application steps. [23] Accordingly, this in vitro study is concerned with the evaluation of micro-tensile bond strength of two one-step adhesive systems, APL and GB, in comparison to a one two-step type, Ex. The micro-tensile bond strength test was selected for that purpose because of its expected accuracy in comparison to other bond strength tests. [12,13]

The recorded data revealed that the µTBS value of GB adhesive was comparable to that of the Ex (Control) and to that of APL, whereas the µTBS value of APL adhesive was

lower than that of Ex (Tables 2, 3). These findings coincide with the results of many studies [2,7,24,25] and may be explained as follows; the higher bonding efficiency of Ex is almost dependent on the formation of hybrid layer, in addition to the direct penetration of both dentin micro-irregularities and the opened dentinal tubules. [26] Presence of alcohol in the Ex formulation also helps the infiltration of adhesive resin into the collapsed collagen network and accordingly improves the adhesive's bond strength. [3] Combining both the etching and bonding procedures in one step can deteriorate the bond as recorded in previous studies [7,24] Those studies related the reduction in bond strength to the presence of etching material and outcomes within the formed dentin/adhesive interaction layer. However, with using GB, functional monomers contained in the bonding material react with hydroxyapatite to form insoluble calcium, forming a thin transitional zone that may be responsible for improving its bond strength. [9,27,28,29]

Subjecting some specimens to thermal cycling before performing such bond strength testing has been recommended in many in vitro studies. [7,19,30] Application of heat and cold alternatively usually gives a crude indication about the efficiency and longevity of such bonds in service. Similar to other studies, [7,19,31] a reduction in bond strength values of the tested adhesive systems was found after thermal cycling, but the difference in this study was not statistically significant (Tukey's comparison, $P > 0.05$) probably because of the relatively short-term application of thermal cycling. The reduction in μTBS values could be related to the fatigue of the already existed bond. Bond fatigue may be developed as a result of thermal stresses that developed at the bonding interfaces because of the differences in coefficient of thermal expansion of materials sharing those interfaces. [32] However, the little effect of both thermocycling and water immersion on the bond strength values of GB could be attributed to the minimal thickness of the adhesive junction. [2,5,20]

The adhesive and admix (adhesive/cohesive) modes of bond failure were revealed in the results of this study (Figure 1), which coincide with other studies. [4,33,34,35] The suggestion is that the presence of both etching material and products of the etching process within the adhesive layer weaken mechanical properties of such resin. These remnants own different elastic moduli and coefficients of thermal expansion than those of dentin and composite material. These differences could initiate microcracks within the adhesive layer at the time of specimens' cutting and testing. Therefore fracture of the adhesive body could be expected more frequently with the self-etching adhesives in comparison to the 2 step adhesives those always free of etching remnants. [7]

CONCLUSION

Within the limitation of this study, the one-step adhesive APL does not perform consistently worse regarding μTBS (16.3 ± 2.7) than the newer one GB (18.4 ± 4.9). The μTBS of GB is also comparable to that of the control, Ex two-step adhesive (20.9 ± 2.4). Thermal cycling has no statistically significant deteriorating effect on the performed bond of different adhesives under investigation, APL-Th (13.1 ± 1.2), GB-Th (14.7 ± 2.4), Ex-Th (18.4 ± 1.7).

References

1. Nikolaenko SA, Lohbauer U, Roggendorf M, Petschelt A, Dasch W, Frankenberger R (2004): Influence of c-factor and layering technique on microtensile bond strength to dentin. Dent Mater., 20:579-585.
2. Shirai K, De Munck J, Yoshida Y, Inoue S, Lambrechts P, Suzuki K, Shintani H, Van Meerbeek B (2005): Effect of cavity configuration and aging on the bonding effectiveness of six adhesives to dentin. Dent Mater., 21:110-124.
3. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G (2003): Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent., 28:215-235.
4. El Zohairy AA, De Gee A J, Mohsen MM, Feilzer AJ (2005): Effect of conditioning time of self-etching primers on dentin bond strength of three adhesive resin cements. Dent Mater., 21:83-93.
5. Armstrong SR, Vargas MA, Fang Q, Laffoon JE (2003): Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentinbonding system through 15-month water storage. J Adhes Dent., 5:47-56.
6. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B (2006): Bond strength of a mild self-etch adhesive with and without prior acid etching. J Dent., 34:77-85.
7. Frankenberger R, Tay RF (2005): Self-etch versus etch-and-rinse adhesives: effect of thermo-mechanical fatigue loading on marginal quality of bonded resin composite restorations. Dent Mater., 21:397-412.
8. Ozok AR, Wu MK, De Gee AJ, Wesselink PR (2004): Effect of dentin perfusion on the sealing ability and microtensile bond strengths of a total-etch versus an all-in-one adhesive. Dent Mater., 20: 479-486.
9. Product pamphlet, GC G-Bond, Advanced Seventh Generation Bonding, GC America Inc., USA
10. Sidhu, S K, Omata Y, Tanaka T, Koshiro K, Spreafico D, Semeraro S, Mezzanzanica D, Sano H (2007): Bonding characteristics of newly developed all-in-one adhesives. J Biomed Mater Res B Appl Biomater., 80: 297-303.
11. Perdigao, J., Gomes G, Gondo R, Fundingsland JW (2006): In vitro bonding performance of all-in-one adhesives. Part I--microtensile bond strengths. J Adhes Dent., 8: 367-373.
12. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernands CA, Tay FR (1999): The

microtensile bond test: a review. J Adhes Dent., 1:299-309.
13. Sano H, Shono T, Sonoda H, Pashley DH (1994): Relationship between surface area for adhesion and tensile bond strength-evaluation of a microtensile test. Dent Mater., 10:236-240.
14. Purk JH, Healy M, Dusevich V, Glaros A, Eick JD (2006): In vitro microtensile bond strength of four adhesives testedat the gingival and pulpal walls of Class II restorations. J Am Dent Asso., 137:1414-1418.
15. Sano H (2006): Microtensile testing, nanoleakage, and biodegradation of resin-dentin bonds. J Dent Res., 85:11-15.
16. Betamar, N., G. Cardew, Van Noort R (2007): Influence of specimen designs on the microtensile bond strength to dentin. J Adhes Dent., 9: 159-168.
17. Yoshiyama M, Carvalho RM, Sano H, Horner JA, Brewer PD, Pashley DH (1996): Regional bond strength of resin to human root dentin. J Dent Res., 74:435-442.
18. Yoshiyama M, Sano H, Ebisu S, Tagami J, Ciucchi B, Carvalho RM, Johnson MH, Pashley DH (1996): Regional bond strength of bonding agents to cervical sclerotic root dentin. J Dent Res., 75:1404-1413.
19. Nikaido T, Kunzelmann KH, Chen H, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R, Tagami J (2002): Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin. Dent Mater., 18:269-275.
20. Price RB, Derant T, Andreou P, Murphy D (2003): The effect of two configuration factors, time, and thermal cycling on resin to dentin bond strengths. J Biomaterials., 24:1013-1021.
21. El-Kholany NR, Abdelaziz KM, Zaghloul NM, Aboulenien N (2005): Bonding of single-component adhesive to dentin following chemomechanical caries removal. J Adhes Dent., 7:281-287.
22. Feilzer AJ, De Gee AJ, Davidson CL (1987): Setting stress in composite resin in relation to configuration of the restoration. J Dent Res., 66:1636-1639.
23. Koh SH, Powers JM, Bebermyer RD, Li D (2001): Tensile bond strengths of fourth-and fifth-generation dentin adhesives with packable resin composite. J Esthet Restor Dent., 13:379-386.
24. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M , Suzuki K, Lambrechts P, Vanherle G (2003): Four-year water degradation of total-etch adhesives bonded to dentin. J Dent Res., 82:136-140.
25. Hannig M, Bock H, Bott B, Hoth-Hannig W (2002): Inter-crystallite nanoretention of self-etching adhesives at enamel imaged by transmission electron microscopy. Eur J Oral Sci., 110:464-470.
26. Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P, Vanherle G (1992): Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems. J Dent Res., 72:1530-1540.
27. Hiraishia N, Breschi L, Prati C, Ferrari M, Tagami, King NM (2007): Technique sensitivity associated with air-drying of HEMA-free, single-bottle, one-step self-etch adhesives. J Dent Mater., 23:498-505.
28. Spencer P, Wang Y (2002): Adhesive phase separation at the dentin interface under wet bonding conditions. J Biomed Mater Res., 62: 447-456.
29. Tay FR, King NM, Chan KM, Pashley DH (2002): How can nanoleakage occur in self-etching adhesive systems that demineralize and infiltrate simultaneously? J Adhes Dent., 2: 255-269.
30. Lee SY, Chiang HC, Lin CT, Huang HM, Dong DR (2000): Finite element analysis of thermo-debonding mechanism in dental composites. J Biomaterials., 21:1315-1326.
31. Mitsui FH, Peris AR, Cavalcanti AN, Marchi GM, Pimenta LA (2006): Influence of thermal and mechanical load cycling on microtensile bond strengths of total and self-etching adhesive systems. Oper Dent., 31:240-247.
32. Craig RG, Powers JM (2002): Restorative Dental Materials. 11th ed. Mosby, Inc., 53-54.
33. Carvalho RM, Pegoraro TA, Tay FR, Pegoraro LF, Silva NRFA, Pashley DH (2004): Adhesive permeability affects coupling of resin cements that utilize self-etching primers to dentin. J Dent., 32:55-65.
34. Cho BH, Dickens SH (2004): Effect of the acetone content of single solution dentin bonding agents on the adhesive layer thickness and the microtensile bond strength. Dent Mater., 20:107-115.
35. Jacques P, Hebling J (2005): Effect of dentin conditioners on the microtensile bond strength of a conventional and a self-etching primer adhesive system. Dent Mater., 21:103-109.

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