

Does Bonding Approach Influence the Bond Strength of Universal Adhesive to Dentin of Primary Teeth?

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Objective: To evaluate the effect of bonding strategy on microtensile bond strength (μ TBS) of a new universal adhesive system to primary tooth dentin. **Study design:** Flat dentin surfaces from 25 primary molars were assigned to 5 groups according to the adhesive and bonding approach: Adper Single Bond 2 (two-step etch-and-rinse adhesive) and Clearfil SE Bond (two-step self-etch system), as controls; Scotchbond Universal Adhesive–self-etch, dry or wet-bonding etch-and-rinse strategies. Composite buildups were constructed and the teeth were sectioned to obtain bonded sticks (0.8 mm^2) to be tested under tension at 1 mm/min . The μ TBS means were analyzed by one-way ANOVA and Tukey's tests ($\alpha = 0.05$). Failure mode was evaluated using a stereomicroscope ($400\times$). **Results:** Universal adhesive applied following both dry and wet-bonding etch-and-rinse strategies showed similar bond strength compared with control adhesive systems. Self-etch approach resulted in the lowest μ TBS values. For all groups, adhesive/mixed failure prevailed. The percentage of premature debonded specimens was higher when the universal adhesive was used as self-etch mode. **Conclusion:** The universal adhesive does not share the same versatility of being used in the etch-and-rinse and self-etch approaches; however, the use of the new adhesive following either wet or dry-bonding may be a suitable option as alternative to two-step etch-and-rinse adhesive protocol.

Key words: dentin; microtensile; etch-and-rinse; self-etch; universal simplified adhesive system

INTRODUCTION

Considering the great number of commercially available adhesive systems, the choice of bonding strategy and number of steps has been often a matter of personal preference. Based on that, manufacturers have released adhesive systems more versatile that include etch-and-rinse and self-etch options, aiming to make the clinical procedure more user-friendly. These new materials are so-called “Universal” or “Multi-purpose” adhesives and present similar composition relative to one-step self-etch systems.

Etch-and-rinse adhesive approach requires previous dentin demineralization with phosphoric acid in order to expose collagen fibrils for resin infiltration. The main disadvantage of this protocol is that there is the risk of collagen fibrils collapse during the procedure of dentin drying, which reduces the infiltration of resin monomers and leads to a decrease in bond strength¹⁻². In fact, the adequate moisture is still a clinical challenge, since it depends on both the solvent composition³ and on the overall interpretation of manufacturers' instructions. Interestingly, the universal adhesives could be applied on either wet or dry demineralized dentin, eliminating thus, procedure errors.

Likewise, based on the current trends toward ease-of-use and faster clinical application steps, the use of simplified self-etch adhesives seems to be an attractive choice, since these systems are less technique-sensitive than etch-and-rinse systems, due to simultaneous demineralization and infiltration into the dentin substrate⁴. Nevertheless, it has been demonstrated that one-step self-etch dentin bonding systems exhibit lower bond strengths and are less predictable than etch-and-rinse and two-step self-etch systems⁵⁻⁶. In this context, the use of universal adhesives following self-etch strategy without compromising the bonding effectiveness to dentin would be very desirable.

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There is little information in the literature about performance of the universal adhesive systems applied under different bonding strategies, and the results obtained are still not consensual⁷⁻¹¹. Moreover, these investigations were performed using permanent teeth. Since primary and permanent dentin present differences in microstructure and composition¹²⁻¹³, and these characteristics may interfere with the adhesive performance¹⁴⁻¹⁶, the findings reported cannot be directly extrapolated to primary teeth.

To the best of our knowledge, this is a pioneering investigation that assessed if a universal adhesive system can be applied in a multi-mode manner, following either a self-etch, dry or wet-bonding etch-and-rinse approaches on dentin of primary teeth. Therefore, this study aimed to investigate the impact of bonding strategy on microtensile bond strength (μ TBS) of a new universal adhesive system to primary tooth dentin.

MATERIAL AND METHOD

Twenty-five sound, naturally exfoliated second primary molars were collected after the patients' informed consent had been obtained under a protocol reviewed and approved by the local Research Ethics Committee. The teeth were disinfected in 0.5% aqueous chloramine and stored in distilled water at 4°C until use.

Flat midcoronal dentin surfaces were exposed after removal of occlusal enamel using a water-cooled diamond saw in a cutting machine (Labcut 1010, Extec Co., Enfield, CT, USA). The surrounding enamel was removed with a diamond bur in a high-speed handpiece (# 3195, KG Sorensen, Barueri, Brazil) with water spray. The specimens were carefully examined under a stereomicroscope at 30 \times magnification to confirm the absence of enamel islets. The exposed dentin surfaces were further polished with 600-grit silicon carbide abrasive paper under running water for 30 s to standardize the smear layer.

The teeth were randomly assigned into five groups (n=5) according to the different bonding strategies of the selected adhesive system. Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA) was applied on dentin surfaces following either a self-etch, a dry-bonding or a wet-bonding etch-and-rinse adhesive protocol. As control materials, the two-step etch-and-rinse adhesive, Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA); and two-step self-etch system, Clearfil SE Bond (Kuraray Medical Inc., Okayama, Japan) were used.

The adhesive systems were applied strictly in accordance with the respective manufacturers' instructions, as described in Table 1. After the bonding procedures, resin composite (Filtek Z250, shade A1, 3M ESPE, St. Paul, MN, USA) was incrementally placed on dentin surfaces, and each layer was light-cured for 20 s with a light emitting diode curing unit (Emitter B, Schuster, Santa Maria, RS, Brazil) with a light output of at least 1250 mW/cm². Light intensity output was monitored with a Demetron Curing Radiometer (Demetron Research Corporation, Danbury, CT). All bonded specimens were stored in distilled water at 37°C for 24h.

Microtensile bond strength (μ TBS)

Teeth were sectioned longitudinally in the mesio-distal and buccal-lingual directions across the bonded interface using a water-cooled diamond saw in a cutting machine (Labcut 1010, Extec Co., Enfield, CT, USA) to obtain sticks with a cross-sectional area of approximately

0.8 mm². The cross-sectional area of each stick was measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) for calculating bond strength (in MPa). The sticks were carefully examined with a stereomicroscope at 30 \times magnification and those with defects at the resin-dentin interface were discarded.

Each resin-dentin bonded stick was attached to a modified device for μ TBS testing with cyanoacrylate resin and tested under tension (Emic, São José dos Pinhais, PR, Brazil) at a crosshead speed of 1mm/min until failure. The μ TBS values were calculated by dividing the load at failure by the cross-sectional bonding area.

Failure mode

Premature failure was defined as specimen debonding during preparation, which precluded the testing. After the test, both sides of all debonded sticks were observed in a stereomicroscope (HMV II, Shimadzu, Kyoto, Japan) at 400 \times magnification to determine failure mode: adhesive/mixed (failure at the resin-dentin interface or mixed with cohesive failure of the neighboring substrate) or cohesive (failure exclusively within the dentin or resin composite).

Statistical Analysis

The experimental unit in the current study was the tooth. Thus, the mean of the μ TBS values of all of the sticks from the same tooth were averaged for statistical analysis. The μ TBS means for every test group was expressed as the mean of the five teeth used per group. The prematurely debonded specimens were included in the tooth mean with an arbitrary value of 4.0 MPa (mean value between zero and the minimum bond strength value observed in this study)¹⁷.

The normal distribution of the data was confirmed using Kolmogorov-Smirnov test. The μ TBS means were analyzed by one-way ANOVA and Tukey's *post hoc* tests ($\alpha = 0.05$). Statistical analysis was performed using the Minitab software (Minitab Inc., State College, PA, USA). The failure mode was evaluated descriptively.

RESULTS

The microtensile bond strength means (MPa) and standard deviations for all experimental groups are shown in Table 2. Universal adhesive applied following both dry and wet-bonding etch-and-rinse strategies showed similar bond strength relative to control groups. Self-etch approach resulted in the lowest μ TBS values ($p = 0.000$; $F = 9.95$).

The distribution of the failure mode is summarized in Figure 1. For all groups, adhesive/mixed failure prevailed. The percentage of premature debonded specimens was higher when universal adhesive was applied as self-etch mode.

DISCUSSION

The new class of adhesive systems suggests the clinicians to opt either for an etch-and-rinse or a self-etch approach. However, the concept behind the multi-mode adhesives is novel; hence no previous study evaluated the bond strength of universal adhesive to primary tooth dentin under different bonding strategies.

Thereby, we tested the hypotheses that the universal adhesive applied to dentin according to the etch-and-rinse and self-etch strategies when compared their respective controls groups should not affect the immediate bond strength to dentin; and that the application mode (dry or wet-bonding) should not influence the resin-dentin bonds created when using etch-and-rinse protocol.

Table 1 – Composition and application mode of the adhesive system tested.

Adhesive system (batch number)	Main components	Self-etch strategy	Etch-and-rinse strategy
Scotchbond Universal Adhesive	Etchant: 34% phosphoric acid, water, synthetic amorphous silica, polyethylene glycol, aluminium oxide. MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1. Keep dentin dry, do not overdry 2. Apply the adhesive for 20 s with vigorous agitation 6. Gentle air thin for 5 s 7. Light-cure for 10 s	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry to remove excess of water 4. Keep dentin moist (wet-bonding approach) or Keep dentin dry, do not overdry (dry-bonding approach) 5. Apply the adhesive as for the self-etch mode
Adper Single Bond 2	Etchant : 35% phosphoric acid HEMA, water, ethanol, Bis-GMA, dimethacrylates, amines, methacrylate-functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles	N.A	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Blot excess water 4. Apply 2 consecutive coats of adhesive for 15 s with gentle agitation 5. Gently air dry for 5 s 6. Light-cure for 10 s
Clearfil SE Bond	Primer: MDP, HEMA, hydrophilic dimethacrylate, dl-campherquinone ,N,N-diethanol-p-toluidine, water Bonding: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-campherquinone, N,N-diethanol-p-toluidine, silanated colloidal silica	1. Apply primer on dry dentin surface and left undisturbed for 20 s 2. Dry with air stream for 5 s to evaporate the volatile ingredients 3. Apply bond and gently air dry 4. Light-cure for 10 s	N.A

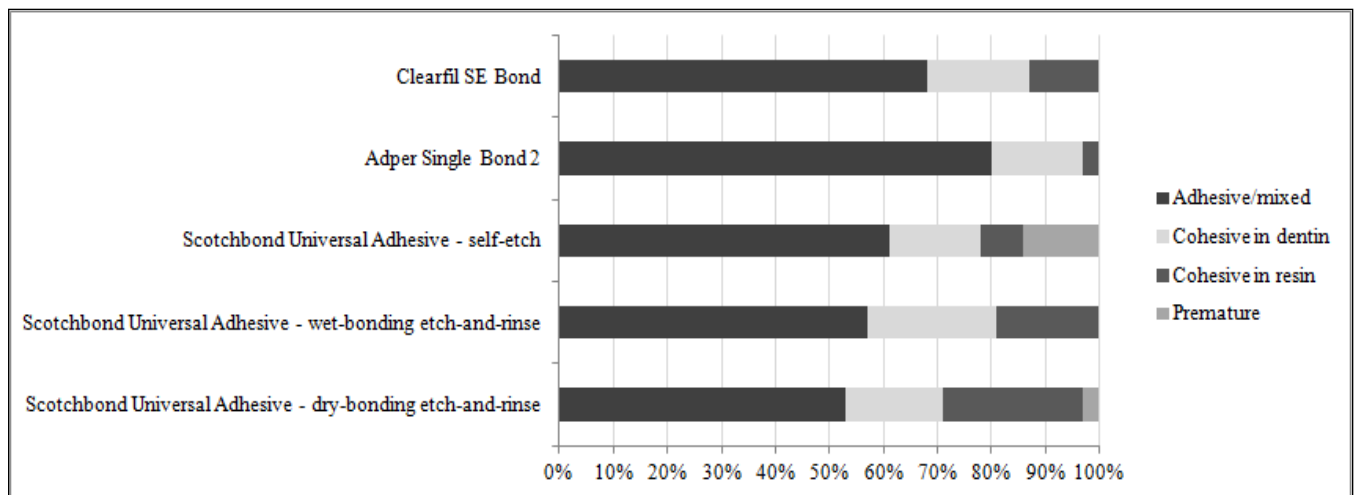
MDP: 10-methacryloyloxydecyl-dihydrogen-phosphate; Bis-GMA: bisphenyl-glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate

Table 2–Microtensile bond strength means and standard deviations for all experimental groups (*)

Adhesive System	Application mode	µTBS (MPa)
Scotchbond Universal Adhesive	Dry-bonding etch-and-rinse	50.1 ± 7.4 ^A
	Wet-bonding etch-and-rinse	47.3 ± 5.5 ^A
	Self-etch	27.8 ± 5.9 ^B
Adper Single Bond 2	Etch-and-rinse	43.2 ± 5.6 ^A
Clearfil SE Bond	Self-etch	44.1 ± 6.0 ^A

(*)Different superscript capital letters indicate significant differences (n=5; p <0.05).

Figure 1–Failure mode distribution (%).



In our study, the universal adhesive did not perform with the same versatility of being used in the etch-and-rinse and self-etch approaches. Lower μ TBS values, accomplished of a predominance of debonded premature specimens were found when universal adhesive was applied on dentin surfaces following the self-etch mode compared with other groups.

Scotchbond Universal Adhesive present similarities with self-etch adhesive system (Clearfil SE Bond) tested as control related to pH range (around 2), besides the presence of MDP (10-methacryloxydecyl dihydrogen phosphate) as acidic polymerizable monomer. Due to their mild aggressiveness, the smear layer and the underlying dentin are only partially dissolved, leaving hydroxyapatite remnants available for chemical interaction with a functional monomer¹⁸⁻¹⁹. Based on that, one would expect that μ TBS values of these adhesive would be statistically equivalent. However, this was not verified, corroborating the findings of a previous study⁹. The presence of polyalkenoic acid copolymer in the composition of Scotchbond Universal Adhesive may explain the worse bonding performance verified in our study. This copolymer may have competed with the MDP by blinding to the calcium of the hydroxyapatite^{9,20}. Additionally, the polyalkenoic acid copolymer could prevent the monomer approximation during polymerization, due to its high molecular weight⁹.

For some one step-self etch adhesives, performance may depend on the application method, as the number of coats recommended by the manufacturer may not suffice²¹⁻²². It has been evidenced that the application of the one-step self-etch adhesive multiple layers results in higher bond strengths²³⁻²⁴. Further studies are necessary to assessed if an additional layer would increase the bonding of the universal adhesive used with self-etch strategy.

Regarding etch-and-rinse approach, the universal adhesive showed similar μ TBS values compared with control groups. No difference in bonding effectiveness was also observed between Adper Single Bond 2 and Clearfil SE Bond, in line with previous reports²⁵⁻²⁶.

Furthermore, the application mode (dry or wet-bonding etch-and-rinse) did not affect the bond strength of Scotchbond Universal Adhesive to primary tooth dentin. This adhesive present “VMS Technology”, that consist in union of Vitrebond copolymer, MDP and silane in its composition, permitting the rehydration of collagen fibrils and the hybrid layers creation even with dry demineralized dentin. Hanasuba et al.⁷ reported that the dry-bonding etch-and-rinse protocol was more effective than its wet-bonding version. Nevertheless, other multi-purpose adhesive was tested (G-Bond

Plus; GC, Tokyo, Japan). The authors attributed the lower bond strength for the wet-bonding approach to the presence of too much water at the interface, resulting in inhibition of resin polymerization and/or monomer dilution⁷.

A previous study²⁷ evaluated the influence of dentin moisture on bond strength of an etch-and-rinse bonding agent to primary tooth dentin under laboratory and clinical conditions. Wet bonding technique provided higher μ TBS values to primary dentin for a two-step etch-and-rinse adhesive.

The preliminary etching time when using etch-and-rinse adhesives is considered critical, due the necessity of the washing procedure to remove the conditioner agent and the requirement for maintenance of the intrinsic moisture of the dentin¹. The overdrying of dentin substrate promotes the breakdown of collagen fibrils, jeopardizing the adhesive effectiveness¹⁻². Likewise, excessive residual moisture may hinder the impregnation of acid monomers on demineralized substrate by dilution of these components¹.

Indeed, the discrimination of the proper moisture degree is still a challenge, because the criteria used to guide this procedure are subjective and depends on operator skills. Since the dentin condition (wet or dry) seems did not impact the performance of universal adhesives, and considering that any simplification is of interest in pediatric dentistry, this universal adhesive may be suitable option as substitute to earlier two-step etch-and-rinse adhesive systems.

Recently, it was reported that the 18-month clinical behavior of Scotchbond Universal Adhesive in permanent dentition does not depend on the bonding strategy¹⁰. Primary teeth have greater density of dentinal tubules¹³ and lower mineral content¹² in comparison with permanent ones. These characteristics may interfere in the adhesive performance to this substrate, resulting in thicker hybrid layers²⁸⁻²⁹, lower bond strength values^{14-15,30} and subsequently, higher prone to undergone degradation over time. Future investigations are required to evaluate the effect of bonding approaches on bond durability of the new class of adhesive to primary dentin.

CONCLUSION

The self-etch approach negatively influences the bond strength of universal adhesive system to primary tooth dentin. However, the universal adhesive can be applied on dry or wet demineralized dentin without compromising the etch-and-rinse bonding quality.

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REFERENCES

1. 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*;28(3):215-35. 2003.
2. Nakajima M, Kanemura N, Pereira PN, Tagami J, Pashley DH. Comparative microtensile bond strength and SEM analysis of bonding to wet and dry dentin. *Am J Dent* 13(6):324-8. 2000.
3. Reis A, Grande RH, Oliveira GM, Lopes GC, Loguercio AD. A 2-year evaluation of moisture on microtensile bond strength and nanoleakage. *Dent Mater*;23(7):862-70. 2007.
4. Salz U, Bock T. Testing adhesion of direct restoratives to dental hard tissue—a review. *J Adhes Dent*;12(5):343-71. 2010.
5. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. *Dent Mater*;21(9):864-81. 2005.
6. Ozer F, Blatz MB. Self-etch and etch-and-rinse adhesive systems in clinical dentistry. *Compend Contin Educ Dent*;34(1):12-4, 16, 18; quiz 20, 30. 2013.
7. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Ende A, Van Meerbeek B, et al. Bonding effectiveness of a new ‘multi-mode’ adhesive to enamel and dentine. *J Dent*;40(6):475-84. 2012.
8. Perdigao J, Sezinando A, Monteiro PC. Laboratory bonding ability of a multi-purpose dentin adhesive. *Am J Dent*;25(3):153-8. 2012.
9. Munoz MA, Luque I, Hass V, Reis A, Loguercio AD, Bombarda NH. Immediate bonding properties of universal adhesives to dentine. *J Dent*;41(5):404-11. 2013.
10. Perdigao J, Kose C, Mena-Serrano A, De Paula E, Tay L, Reis A, et al. A New Universal Simplified Adhesive: 18-Month Clinical Evaluation. *Oper Dent*. 39:113-127, 2014.
11. Mena-Serrano A, Kose C, De Paula EA, Tay LY, Reis A, Loguercio AD, et al. A new universal simplified adhesive: 6-month clinical evaluation. *J Esthet Restor Dent*;25(1):55-69. 2013.
12. Angker L, Nockolds C, Swain MV, Kilpatrick N. Quantitative analysis of the mineral content of sound and carious primary dentine using BSE imaging. *Arch Oral Biol*;49(2):99-107. 2004.
13. Lenzi TL, Guglielmi Cde A, Arana-Chavez VE, Raggio DP. Tubule density and diameter in coronal dentin from primary and permanent human teeth. *Microsc Microanal*;19(6):1445-9. 2013.
14. Senawongse P, Harnirattisai C, Shimada Y, Tagami J. Effective bond strength of current adhesive systems on deciduous and permanent dentin. *Oper Dent*;29(2):196-202. 2004.
15. Uekusa S, Yamaguchi K, Miyazaki M, Tsubota K, Kurokawa H, Hosoya Y. Bonding efficacy of single-step self-etch systems to sound primary and permanent tooth dentin. *Oper Dent*;31(5):569-76. 2006.
16. Lenzi TL, Soares FZ, Rocha RD. Degradation of resin-dentin bonds of etch-and-rinse adhesive system to primary and permanent teeth. *Braz Oral Res* 2012.
17. Reis A, Loguercio AD, Azevedo CL, de Carvalho RM, da Julio Singer M, Grande RH. Moisture spectrum of demineralized dentin for adhesive systems with different solvent bases. *J Adhes Dent*;5(3):183-92. 2003.
18. Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. *Dent Mater*;17(5):430-44. 2001.
19. Sanabe ME, Kantovitz KR, Costa CA, Hebling J. Effect of acid etching time on the degradation of resin-dentin bonds in primary teeth. *Am J Dent*;22(1):37-42. 2009.
20. Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, et al. Self-assembled Nano-layering at the Adhesive interface. *J Dent Res*;91(4):376-81. 2012.
21. De Munck J, Vargas M, Iracki J, Van Landuyt K, Poitevin A, Lambrechts P, et al. One-day bonding effectiveness of new self-etch adhesives to bur-cut enamel and dentin. *Oper Dent*;30(1):39-49. 2005.
22. Sensi LG, Lopes GC, Monteiro S, Jr., Baratieri LN, Vieira LC. Dentin bond strength of self-etching primers/adhesives. *Oper Dent*;30(1):63-8. 2005.
23. Ito S, Tay FR, Hashimoto M, Yoshiyama M, Saito T, Brackett WW, et al. Effects of multiple coatings of two all-in-one adhesives on dentin bonding. *J Adhes Dent*;7(2):133-41. 2005.
24. Frankenberger R, Perdigao J, Rosa BT, Lopes M. “No-bottle” vs “multi-bottle” dentin adhesives—a microtensile bond strength and morphological study. *Dent Mater*;17(5):373-80. 2001.
25. Soares FZ, Rocha Rde O, Raggio DP, Sadek FT, Cardoso PE. Microtensile bond strength of different adhesive systems to primary and permanent dentin. *Pediatr Dent*;27(6):457-62. 2005.
26. Botelho Amaral FL, Martao Florio F, Bovi Ambrosano GM, Basting RT. Morphology and microtensile bond strength of adhesive systems to in situ formed caries-affected dentin after the use of a papain-based chemomechanical gel method. *Am J Dent*;24(1):13-9. 2011.
27. Chibinski AC, Stanislawczuk R, Roderjan DA, Loguercio AD, Wambier DS, Grande RH, et al. Clinical versus laboratory adhesive performance to wet and dry demineralized primary dentin. *Am J Dent*;24(4):221-5. 2011.
28. Nor JE, Feigal RJ, Dennison JB, Edwards CA. Dentin bonding: SEM comparison of the resin-dentin interface in primary and permanent teeth. *J Dent Res*;75(6):1396-403. 1996.
29. Nor JE, Feigal RJ, Dennison JB, Edwards CA. Dentin bonding: SEM comparison of the dentin surface in primary and permanent teeth. *Pediatr Dent*;19(4):246-52. 1997.
30. Bordin-Aykroyd S, Sefton J, Davies EH. In vitro bond strengths of three current dentin adhesives to primary and permanent teeth. *Dent Mater*;8(2):74-8. 1992.