©Operative Dentistry, 2012, **37-1**, 37-44

Cross-Compatibility of Methacrylate-Based Resin Composites and Etch-and-Rinse One-Bottle Adhesives

C Sabatini • M Campillo • S Hoelz
EL Davis • CA Munoz

Clinical Relevance

Cross-compatibility between different combinations of methacrylate-based resin composites and etch-and-rinse one-bottle adhesives was evidenced for products from different manufacturers. This represents a desirable property of adhesives as it allows the flexibility to select different composite systems based on the specific restorative needs.

*Camila Sabatini, DDS, MS, assistant professor, SUNY at Buffalo, School of Dental Medicine, Department of Restorative Dentistry, Buffalo, NY, USA

Marc Campillo, PhD, senior research scientist, SUNY at Buffalo, School of Dental Medicine, Department of Restorative Dentistry, Buffalo, NY, USA

Shane Hoelz, student dentist, SUNY at Buffalo, School of Dental Medicine, Buffalo, NY, USA

Elaine L. Davis, PhD, professor, SUNY at Buffalo, School of Dental Medicine, Department of Oral Diagnostic Sciences, Buffalo, NY, USA

Carlos A. Munoz, DDS, MSD, professor, SUNY at Buffalo, School of Dental Medicine, Department of Restorative Dentistry, Buffalo, NY, USA

*Corresponding author: SUNY at Buffalo, School of Dental Medicine, Department of Restorative Dentistry, 3435 Main Street, 215 Squire Hall, Buffalo, NY 14214; e-mail: cs252@buffalo.edu

DOI: 10.2341/11-095-L

SUMMARY

Objective: To compare dentin shear bond strength (SBS) of four combinations of light-activated one-bottle adhesives and composites to determine if cross-compatibility exists, and to determine if the use of the same manufacturer's adhesive and composite results in higher SBS than systems that combine different manufacturers' products.

Methods: One hundred sixty human third molars were used for bonding (n=10). Specimens were treated with 37% phosphoric acid and one of four etch-and-rinse adhesives. Specimens were placed in a bonding jig, which was filled with one of four composites. Adhesives PQ1 (Ultradent), Excite (Ivoclar-Vivadent), Optibond Solo Plus (Kerr), and Single Bond (3M-ESPE) and composites Vit-l-Escence

(Ultradent), Four Seasons (Ivoclar-Vivadent), Premise (Kerr), and Filtek Supreme Plus (3M-ESPE) were tested. SBS was measured at 24 hours and three months with a testing machine at a speed of 1 mm/min and expressed in MPa. A three-way analysis of variance and Tukey tests were used for data analysis.

Results: Significant differences were evidenced among composites for each adhesive system ($p<0.001$) and among adhesives for each composite system ($p<0.001$). Optibond Solo Plus and PQ1 yielded significantly higher bond strengths than Single Bond and Excite for all composite systems ($p<0.05$). All combinations, with the exception of two, demonstrated a decrease in bond strength values after aging.

Conclusions: Cross-compatibility was demonstrated, indicating that etch-and-rinse one-bottle adhesive systems can be safely used with composites from different manufacturers without a compromise to the bond strength. Moreover, even higher mean SBS values were demonstrated for selective combinations of different manufacturers' products.

INTRODUCTION

A belief has been promoted by manufacturers from dental companies encouraging dentists to use their adhesive systems in combination with their resin composites as they claim that greater strength and longevity of the restorations can be obtained when using products from the same manufacturer. Clinicians often face the question as to whether incorporating an adhesive and composite from the same manufacturer will result in greater strength and longevity of restorations.

While it is well known that certain bonding agents are not compatible with self- and dual-polymerized resin composites,[1–5] it remains unclear whether compatibility issues exist between adhesives and light-activated composites. Adverse acid-base reactions as well as adhesive permeability may be responsible for any incompatibility issues that may exist.[6–8] Acidic monomers in etch-and-rinse adhesives can adversely react with basic initiators (tertiary amines) of self- or dual-polymerized composites, preventing their polymerization.[2–5] Although light-activated composites also undergo polymerization through the generation of free radicals by tertiary amines, they appear to be less affected by the acidic monomers on etch-and-rinse adhesives than self- or dual-polymerized composites. This may be the result of a more rapidly occurring initiation process, which may interrupt the acid-base reaction.[6,9]

Cross-compatibility between adhesives and resin composites made from different manufacturers represents a desirable property as it allows the dentist the flexibility to select different composite systems based on specific restorative needs. A few studies have reported higher bond strengths when an adhesive was used with a composite from the same manufacturer rather than a composite from a different manufacturer.[2–3,10] However, these findings may be the result of differences in strength among the various types of composite materials (ie, hybrids vs microfills) rather than caused by compatibility issues between the adhesive and composite systems, as suggested by some studies.[11,12] Not enough evidence is available to support the claim that combining an adhesive and a composite with somewhat different monomeric composition and made from different manufacturers will result in lower bond strengths than the use of products made from the same manufacturer. In general, it appears that cross-compatibility exists and that adhesives and composites from different manufacturers can be combined without a compromise to the bond strength.[13,14]

The literature on the topic is scarce. A literature search dating back to 1980 was conducted to identify studies related to compatibility issues between light-activated composites and adhesive systems. A few studies were identified that revealed no compatibility issues between adhesives and light-activated composites.[13–15] However, these early studies report on products that have either disappeared from the market, changed in technology, or were made by a manufacturer that is no longer in business. The authors are not aware of recent studies reporting on the cross-compatibility properties of newer commercially available adhesive systems.

The objective of this study was to compare dentin shear bond strength (SBS) of different combinations of light-activated methacrylate-based resin composites and etch-and-rinse adhesive systems made from four different manufacturers at 24 hours and three months to determine if cross-compatibility exists. Furthermore, the study aimed to determine if using an adhesive and composite from the same manufacturer results in higher bond strengths than the use of an adhesive and composite made from different manufacturers.

Table 1: *Tested Materials, Manufacturers, Category, Composition, and Batch Number as per Manufacturers' Descriptions*

Adhesive Systems

Product (Manufacturer)	Category	Organic Composition	Filler	Batch Number
Optibond Solo Plus (Kerr, Orange, CA, USA)	Etch and rinse	Alkyl dimethacrylate resins, barium aluminoborosilicate glass, silicon dioxide, sodium hexafluorosilicate Solvent: ethanol	Barium aluminoborosilicate glass, silicon dioxide sodium hexafluorosilicate Filled 15% by wt	3267789
Excite (Ivoclar-Vivadent, Amherst, NY, USA)	Etch and rinse	HEMA, Bis-GMA, Phosphonic acid acrylate Solvent: ethanol	Silicon dioxide Filled 0.5% by wt	M06539
Single Bond (3M-ESPE, St Paul, MN, USA)	Etch and rinse	HEMA, Bis-GMA, water, PAA, Solvent: ethanol	Silica Filled 10% by wt	20090625
PQ1 (Ultradent, South Jordan, UT, USA)	Etch and rinse	Bis-GMA, methacrylate-based hydrophilic monomers Solvent: ethanol	Silica dioxide and FluorUtite Filled 40% by wt	B46NM

Resin Composites

Product (Manufacturer)	Category	Organic Composition	Filler	Batch Number
Premise (Kerr, Orange, CA, USA)	Nanohybrid	Bis-EMA, TEGDMA	Prepolymerized filler, barium glass, silica nanoparticles (0.02-0.4 μm); filled 84% by wt	3204945
Four Seasons (Ivoclar-Vivadent, Amherst, NY, USA)	Hybrid	Bis-GMA, UDMA, TEGDMA	Barium glass, ytterbium trifluoride, Ba-Al fluorsilicate, dispersed silicon dioxide spheroid mixed oxide (0.6 μm); filled 76% by wt	L47114
Filtek Supreme Plus (3M-ESPE, St Paul, MN, USA)	Nanohybrid	Bis-EMA, Bis-GMA, UDMA, TEGDMA, Water	SiO2 Nanosilica filler, ZrO2/SiO2 Nanoclusters (0.02-0.075 μm) Filled 78.5% by wt	20080827
Vit-l-Escence (Ultradent, South Jordan, UT, USA)	Hybrid	Bis-GMA	Barium alumina silicate (0.7μm)	B4869

Abbreviations: Bis-MA, bisphenol A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; CQ, camphorquinone; PAA, polyalkenoic acid copolymer.

MATERIALS AND METHODS

Table 1 summarizes the composites and adhesives tested in this study. Adhesives and composites from four different manufacturers were selected, for a total of 16 combinations, which were all tested both at 24 hours and three months of 100% humidity storage at 37°C and thermocyling. A sample size of 10 (n=10) was selected after performing power analysis based on previous studies. This yielded a total of 320 specimens.

Specimen Preparation

One hundred sixty noncarious, unrestored human third molars were used to obtain dentin substrates for bonding. The molars were used within three months of extraction and stored in an aqueous disinfectant (0.5% chloramine T solution at 4°C) until ready to be used. The teeth were sectioned longitudinally in a mesiodistal direction using a band saw (Isomet, Buehler, Lake Bluff, IL, USA). The sectioned teeth were embedded in a chemically polymerized methacrylate (Fastray, HJ Bosworth, Skokie, IL, USA) with the outer surface exposed. The exposed molar surface was ground flat on a model trimmer until an adequate surface of dentin was revealed. The exposed dentin was finished with 400- and 600-grit abrasive paper (Silicon carbide abrasive paper, Buehler). The prepared specimens were stored in deionized water at 4°C until ready to be bonded.

Bonding and Testing

Study groups were randomized to avoid bias relative to sequence of tooth restoration. One hour prior to bonding, the specimens were acclimatized to room temperature (23 ± 2°C). Immediately before bonding, the specimens' surfaces were slightly refinished with a 600-grit abrasive paper to expose fresh dentin. Dentin was etched with 37% phosphoric acid (Ultra-etch, Ultradent, South Jordan, UT, USA) for 15 seconds, then rinsed and blot dried for moist bonding. Each adhesive was applied following the manufacturer's instructions and polymerized for 20 seconds with a light-curing unit (Bluephase C8, Ivoclar-Vivadent, Amherst, NY, USA). A minimum power density of 800 mW/cm^2 was ensured by periodically monitoring the unit's output with a radiometer (Demetron, Kerr, Orange, CA, USA). The specimens were placed on a specially fabricated bonding jig (Ultradent) with a cylindrical mold of 2.38 mm in diameter. The mold was filled with the corresponding composite in increments no greater than 2 mm and polymerized for 40 seconds. Immediately after bonding, the specimens were stored for 24 hours in an incubator at 37°C and 100% humidity.

SBS was measured using a testing machine (Ultratester, Ultradent) at a test speed of 1 mm/min. A notched crosshead designed to match the diameter of the bonded specimen was used to apply the testing load. Specimens were stabilized in a testing jig, which was free to move to facilitate positioning under the load. The test base was then positioned so that the notched crosshead was placed against the specimen surface and the notch was fitted to the bonded specimen. The load required to debond the specimen was recorded and expressed in MPa by dividing the load by the surface area of the bonded specimen. SBS values were recorded at 24 hours, and the mean bond strength values for each study group were calculated.

Bonding procedures were repeated as previously outlined, and the specimens were stored under the same conditions for three months. Prior to bond strength testing at three months, the specimens were thermocycled for 500 cycles between 5°C and 55°C with a dwell time of 20 seconds as per recommendations from the International Organization for Standardization (ISO TR11405, 1994).

Statistical Analyses

A three-way analysis of variance (ANOVA) was used to evaluate the effect of the adhesive, composite, and time variables on bond strength. The presence of statistically significant interactions among these factors was also evaluated. A pairwise multiple comparison procedure Tukey test was used to identify differences among adhesives within the composite groups and among composites within the adhesive groups both at 24 hours and after three months of aging. In addition, a Student *t*-test was used to explore significant differences between bond strength values at 24 hours and after three months of aging for each composite-adhesive combination. A significance level of 0.05 was used for all tests.

RESULTS

Three-way ANOVA results are summarized in Table 2. Statistically significant differences were found among adhesives ($p<0.001$), composites ($p<0.001$), and the different testing times ($p<0.001$). Furthermore, statistically significant interactions between the adhesive and the composite ($p=0.003$) and between the adhesive and the testing times ($p<0.001$) were found, but no significant interaction was detected between the composite and the testing times ($p=0.092$).

Figure 1 summarizes the results of the pairwise comparison using Tukey tests. Mean SBS values for the different adhesive-composite combinations at 24 hours and three months are shown for each composite system. Significant differences were evidenced among adhesives for each composite system (letters in Figure 1). When the SBS values were compared within each adhesive system at 24 hours, Tukey tests revealed no statistically significant differences among the four composites for Optibond Solo. When bonding with Excite, a significant difference was observed between Filtek Supreme and Premise. For PQ1, Four Seasons was significantly different from Vit-l-Essence, and with Single Bond, Vit-l-Essence was found to be significantly different from Filtek Supreme and Four Seasons. The same comparison at three months revealed no statistically significant differences between the four composites for either adhesive system with the only exception of Single Bond, which showed a statistically significant difference between Filtek Supreme and Premise.

Table 2: *Three-Way Analysis of Variance Results*

Source of Variation	*df*	SS	MS	*F*	*p*
Adhesive	3	23,846.936	7948.979	152.569	<0.001
Composite	3	916.124	305.375	5.861	<0.001
Time	1	3391.059	3391.059	65.086	<0.001
Adhesive × Composite	9	1330.579	147.842	2.838	0.003
Adhesive × Time	3	2173.621	724.54	13.906	<0.001
Composite × Time	3	338.665	112.888	2.167	0.092
Adhesive × Composite × Time	9	331.666	36.852	0.707	0.702
Residual	288	15,005.043	52.101		
Total	319	47,333.692	148.381		

Abbreviations: df, *degrees of freedom; SS, sum of squares; MS: mean squares;* F, *f obtained;* p, *probability.*

Table 3 shows the differences between the mean bond strength values at 24 hours and after three months of aging for all composite-adhesive combinations. All groups showed a decrease in SBS after aging, with exception of the combinations Single Bond–Four Seasons and Single Bond–Vit-l-Essence. However, for these two groups, the differences did not show statistical significance. For the groups bonded using products from the same manufacturer, two out of four (Optibond-Premise and Excite-Four Seasons) showed significant differences in bond strength values before and after aging, and the other two (Single Bond-Filtek Supreme and PQ1-Vit-l-Essence) did not.

DISCUSSION

In this study, 16 different combinations of etch-and-rinse one-bottle adhesives and light-activated composites from four manufacturers were selected to evaluate cross-compatibility at 24 hours and three months. Cross-compatibility was demonstrated though pairwise comparisons using Tukey tests. As shown in Figure 1, even higher mean SBS values were observed for selective combinations of different manufacturers' products, indicating that cross-compatibility exists and that current etch-and-rinse one-bottle adhesive systems can be safely used with composites from different manufacturers without a compromise to the bond strength. At 24 hours, Optibond Solo Plus and PQ1 yielded significantly higher bond strengths than Single Bond and Excite for all composite systems. Optibond Solo Plus and PQ1 were not statistically different from each other. Similarly, Single Bond and Excite were not statistically different from each other. After aging, although the same behavior was observed for Optibond Solo Plus and PQ1, a few combinations remained not significant. Our findings are in agreement with studies that concluded that the use of products from the same manufacturer does not necessarily yield higher bond strengths.[13,14]

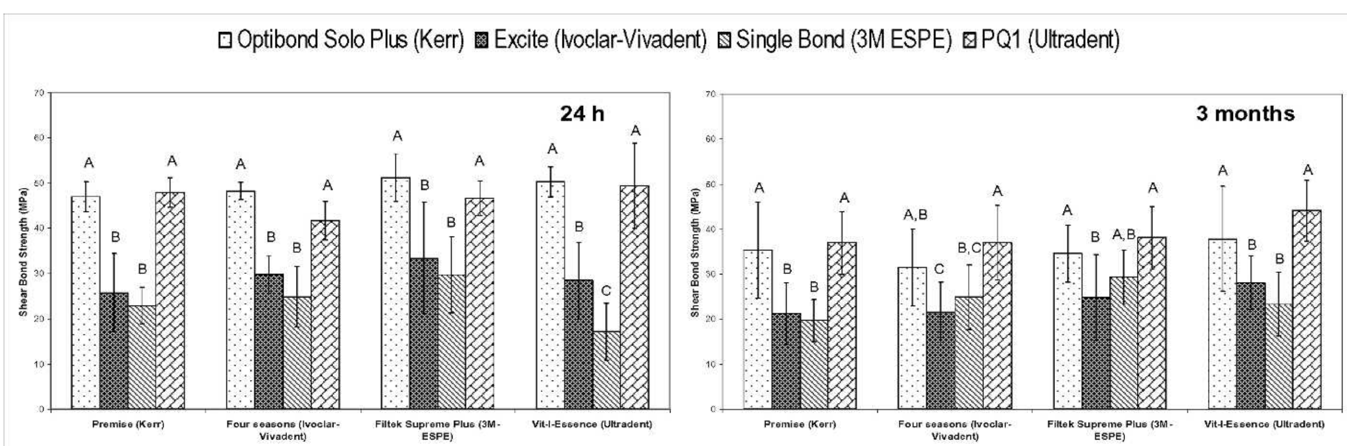

Figure 1. *Mean (SD) shear bond strength values for each of the adhesive-composite combinations. Different manufacturers are represented with different print patterns. Same letters indicate no significant differences among adhesives for each composite.*

Table 3: *Difference in Shear Bond Strength Values Between 24 Hours and Three Months of Aging (Student's t-test p-values)*[a]

	OptiBond Solo Plus (Kerr)		Excite (Ivoclar-Vivadent)		Single Bond (3M-ESPE)		PQ1 (Ultradent)	
	Difference	*p*	Difference	*p*	Difference	*p*	Difference	*p*
Premise (Kerr)	−11.6	**0.004**	−4.5	0.214	−3.2	0.119	−11.0	<**0.001**
Four Seasons (Ivoclar-Vivadent)	−16.8	<**0.001**	−8.2	**0.004**	0.1	0.987	−4.7	0.132
Filtek Supreme (3M ESPE)	−15.0	<**0.001**	−8.6	0.096	−0.4	0.911	−8.5	**0.003**
Vit-l-Escence (Ultradent)	−12.5	**0.005**	−0.4	0.910	6.2	0.053	−5.3	0.169

[a] Bold numbers indicate significant differences.

Compatibility might not be the only factor accountable for differences in bond strength values. Variations in the physical properties of the different types of composites may help explain the observed results.[11,12] Only two of the four groups that combined products from the same manufacturer (Filtek Supreme-Single Bond and Vit-l-Escence-PQ1) displayed the highest bond strength in their corresponding adhesive group.

While the debate is still ongoing as to how much strength is considered clinically acceptable bond strength, the scientific community has agreed on minimum bond strength values in the range of 17 MPa to 20 MPa in order to resist contraction stresses and obtain gap-free margins.[16] Despite the presence of statistically significant differences, all SBS values remained greater than 20 MPa, indicating that that cross-compatibility between products exists and that adhesives can be safely used with composites from different manufacturers without a compromise to their bond strength.

The formation of an adhesive interface is a complex phenomenon. The specific composition of the adhesive directly influences the quality of the resultant hybrid layer and hence its ability to resist shear forces. Compared with unfilled adhesives, filled adhesives create thicker interfacial layers, which help relieve the stresses generated during polymerization contraction, thermal changes, and occlusal loading[17] while yielding improved overall physical properties once it is polymerized. Conversely, the greater viscosity of filled adhesives may decrease momomer conversion[18] as well as limit their ability to penetrate into the demineralized dentin matrix[19] with the potential compromise to the bonded interface overtime.[20,21] Highly filled adhesives Optibond Solo Plus and PQ1 demonstrated significantly higher mean bond strength than lightly filled Excite and Single Bond irrespective of the brand of composite tested.

Current adhesives use primers based on either ethanol or acetone solvents. The type of solvent is also known to have an influence on the wetting capabilities of the adhesive and hence on the strength and stability of the hybrid layer overtime.[22] Although the four adhesives tested in this study were ethanol based, the comparatively lower bond strengths observed for Single Bond and Excite suggest that the type of solvent is only one of the many aspects influencing SBS results.

The effect of water storage on the degradation of adhesive interfaces has been the subject of numerous investigations.[23–25] Specifically, the formation of water blisters within the hybrid layer contributing to the degradation of the adhesive interface overtime has been extensively documented in the literature.[26–28] Our results are in agreement with these studies. As shown in Table 3, with the exception of two groups, all combinations showed a decrease in SBS values after three months of water storage. For seven of the 16 combinations, the differences between 24 hours and three months were statistically significant. Interestingly, the groups showing the highest bond strength values at 24 hours also showed the greatest amount of degradation after aging. However, the ranking remained the same when compared with the initial values.

Although valuable information can be obtained from laboratory studies, care should be taken not to overemphasize the results of *in vitro* investigations such as those reported in the present study. Numerous variables play a role in the immediate and long-term behavior of adhesive interfaces, and they all deserve careful consideration.

CONCLUSIONS

Within the limitations of this *in vitro* investigation, the following conclusions may be drawn:

- Cross-compatibility was demonstrated indicating that current etch-and-rinse one-bottle adhesive systems can be safely used with composites from different manufacturers without a compromise to the bond strength. Moreover, even higher mean SBS values were demonstrated for selective combinations of different manufacturers' products.
- Optibond Solo Plus and PQ1 demonstrated superior bond strength values to all resin composites irrespective of the manufacturer.
- A decrease in SBS values was observed at three months for most combinations. However, trends similar to those observed at 24 hours remained.

Acknowledgements

This project was supported in part by SUNY at Buffalo Student Research Group.

Note

This research work was presented at the 39th Annual Meeting of the American Association for Dental Research (AADR), Washington, DC, USA. March 2010.

(Accepted 6 May 2011)

REFERENCES

1. Walter R, Macedo GV, Oliveira GMS, & Swift EJ (2009) Adhesive bond strengths using self- and light-cured composites *Journal of Contemporary Dental Practice* **10(6)** E025-E032.
2. Carvalho RM, Garcia FCP, e Silva SMA, & Castro FLA (2005) Adhesive-composite incompatibility, part II *Journal of Esthetic and Restorative Dentistry* **17(3)** 191-195.
3. Carvalho RM, Garcia FCP, E Silva SMA, & Castro FLA (2005) Critical appraisal: adhesive-composite incompatibility, part I *Journal of Esthetic and Restorative Dentistry* **17(2)** 129-134.
4. Ishii T, Ohara N, Oshima A, Koizumi H, Nakazawa M, Masuno T, & Matsumura H (2008) Bond strength to bovine dentin of a composite core build-up material combined with four different bonding agents *Journal of Oral Science* **50(3)** 329-333.
5. Leevailoj C, Ua-Wutthikrerk P, & Poolthong S (2007) Shear bond strength of dual-cured and self-cured resin composites to dentin using different bonding agents and techniques *Operative Dentistry* **32(2)**149-159.
6. Suh BI, Feng L, Pashley DH, & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part III. Effect of acidic resin monomers *Journal of Adhesive Dentistry* **5(4)** 267-282.
7. Tay FR, Pashley DH, Yiu CKY, Sanares AME, & Wei SHY (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part 1. Single-step self-etching adhesive *Journal of Adhesive Dentistry* **5(1)** 27-40.
8. Tay FR, Suh BI, Pashley DH, Prati C, Chuang SF, & Li F (2003) Factors contributing to the incompatibility between simplified-step adhesives and self-cured or dual-cured composites. Part II. Single-bottle, total-etch adhesive *Journal of Adhesive Dentistry* **5(2)** 91-105.
9. Sanares AME, Itthagarun A, King NM, Tay FR, & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-556.
10. Leirskar J, Oilo G, & Nordbo H (1998) *In vitro* shear bond strength of two resin composites to dentin with five different dentin adhesives *Quintessence International* **29(12)** 787-792.
11. Hasegawa T, Itoh K, Koike T, Yukitani W, Hisamitsu H, Wakumoto S, & Fujishima A (1999) Effect of mechanical properties of resin composites on the efficacy of the dentin bonding system *Operative Dentistry* **24(6)** 323-330.
12. Perdigao J, Swift EJ, & Cloe BC (1993) Effects of etchants, surface moisture, and resin composite on dentin bond strengths *American Journal of Dentistry* **6(2)** 61-64.
13. Chan DCN, Reinhardt JW, & Boyer DB (1985) Composite resin compatibility and bond longevity of a dentin bonding agent *Journal of Dental Research* **64(12)** 1402-1404.
14. Thomsen KB, & Peutzfeldt A (2007) Resin composites: strength of the bond to dentin versus mechanical properties *Clinical Oral Investigations* **11(1)** 45-49.
15. Mallmann A, de Melo RM, Estrela V, Pelogia F, Campos L, Bottino MA, & Valandro LF (2007) Adhesives with different pHs: Effect on the MTBS of chemically activated and light-activated composites to human dentin *Journal of Applied Oral Science* **15(4)** 265-269.
16. Davidson CL, Degee AJ, & Feilzer A (1984) The competition between the composite-dentin bond strength and the polymerization contraction stress *Journal of Dental Research* **63(12)** 1396-1399.
17. Swift EJ, Perdigao J, Wilder AD, Heymann HO, Sturdevant JR, & Bayne SC (2001) Clinical evaluation of two one-bottle dentin adhesives at three years *Journal of the American Dental Association* **132(8)** 1117-1123.
18. Ferracane JL, Berge HX, & Condon JR (1998) *In vitro* aging of dental composites in water: Effect of degree of conversion, filler volume, and filler/matrix coupling *Journal of Biomedical Materials Research* **42(3)** 465-472.
19. Perdigao J, Baratieri LN, & Lopes M (1999) Laboratory evaluation and clinical application of a new one-bottle adhesive *Journal of Esthetic Dentistry* **11(1)** 23-35.
20. Swift EJ, Perdigao J, Heymann HO, & Ritter AV (1999) Shear bond strengths of one-bottle adhesives to moist enamel *Journal of Esthetic Dentistry* **11(2)** 103-107.

21. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, & Carvalho RM (1995) Adhesion testing of dentin bonding agents: A review *Dental Materials* **11(2)** 117-125.

22. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, & Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: Methods and results *Journal of Dental Research* **84(2)** 118-132.

23. 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 *Journal of Dental Research* **82(2)** 136-140.

24. Frankenberger R, Strobel WO, Lohbauer U, Kramer N, & Petschelt A (2004) The effect of six years of water storage on resin composite bonding to human dentin *Journal of Biomedical Materials Research Part B-Applied Biomaterials* **69B(1)** 25-32.

25. Reis A, Grandi V, Carlotto L, Bortoli G, Patzlaff R, Accorinte MDR, & Loguercio AD (2005) Effect of smear layer thickness and acidity of self-etching solutions on early and long-term bond strength to dentin *Journal of Dentistry* **33(7)** 549-559.

26. Tay FR, & Pashley DH (2003) Have dentin adhesives become too hydrophilic? *Journal of the Canadian Dental Association* **69(11)** 726-731.

27. Hashimoto M, De Munck J, Ito S, Sano H, Kaga M, Oguchi H, Van Meerbeek B, & Pashley DH (2004) *In vitro* effect of nanoleakage expression on resin-dentin bond strengths analyzed by microtensile bond test, SEM/EDX and TEM *Biomaterials* **25(25)** 5565-5574.

28. Sauro S, Pashley DH, Mannocci F, Tay FR, Pilecki P, Sherriff M, & Watson TF (2008) Micropermeability of current self-etching and etch-and-rinse adhesives bonded to deep dentine: a comparison study using a double-staining/confocal microscopy technique *European Journal of Oral Sciences* **116(2)** 184-193.