Available online at www.sciencedirect.com

ScienceDirect

Tanta Dental Journal 11 (2014) 47–55

www.elsevier.com/locate/tdj

The effect of water storage on micro-shear bond strength of contemporary composite resins using different dentin adhesive systems

M.M. Kamel*, H.Y. Elsayed, A.I. Abdalla, A.M. Darrag

Conservative Dentistry Department, Faculty of Dentistry, Tanta University, Egypt

Received 11 December 2013; revised 16 March 2014; accepted 16 March 2014
Available online 27 May 2014

Abstract

Purpose: To evaluate effect of water storage on micro-shear bond strength of adhesives to class I cavity-bottom dentin using two types of composites resin.

Materials and methods: Ninety teeth were divided into three groups I,II&III (thirty molars each) according to the adhesive used, either total-etch 2-step (Adper Single Bond, 3M ESPE), self-etch 2-step(Clearfil SE Bond, Kuraray), or self-etch 1-step(Futura Bond, Voco) respectively. Each group was subdivided according to type of composite restoration used, either Hybrid (Clearfil APX, Kuraray), and Packable (Filtek P60, 3M ESPE). All teeth were thermocycled for 500 cycles,and subjected to occlusal load cycling for 120.000 cycles corresponding to 6 months clinical use. Bonding effectiveness was assessed by micro-shear bond strength test (μSBS) after 1 day, 3 months, and 6 months water storage.

Results: The mean μSBS values (±SD) for Subgroup IA(SB2-APX) were 32.58 ± 1.416, 31.820 ± 2.119, and 30.910 ± 1.393 MPa after 24 h, 3 month, and 6 month respectively; while for Subgroup I B(SB2-P60) were 31.960 ± 1.659, 31.350 ± 1.765, and 30.380 ± 1.773 MPa respectively. Subgroup II A (CSE-APX) recorded 37.28 ± 1.061, 36.77 ± 2.32, 36.21 ± 1.964 MPa, while Subgroup II B(CSE-P60) recorded 37.0 ± 2.115, 36.460 ± 1.727, and 36.080 ± 1.910 MPa after 24 h, 3 month, and 6 month respectively. Subgroup III A (FB-APX) showed 30.550 ± 2.088, 26.890 ± 1.533, and 21.590 ± 1.784 MPa, while subgroup III B (FB-P60) showed 29.790 ± 1.172, 25.960 ± 2.672, and 21.410 ± 2.126 MPa after 24 h, 3 month, and 6 month respectively.

Conclusion: Two-step Total-etch and Two-step self etch adhesives showed better tolerance to water storage compared to One-step self-etch adhesive. However, the type of composite restoration had no significant effect on the microshear bond strength of dental adhesives.

Keywords: Water storage; Microshear bond strength; Adhesives; Composite resin

* Corresponding author.
E-mail addresses: osamanada@hotmail.com, mohmagdikamel1@gmail.com, mohamed.kamel@dent.tanta.edu.eg (M.M. Kamel), hussien.abdrabo@dent.tanta.edu.eg (H.Y. Elsayed), ali_abdalla79@yahoo.com, ali.elsayed@dent.tanta.edu.eg (A.I. Abdalla), adarrag@hotmail.com, abeer.darag@dent.tanta.edu.eg (A.M. Darrag).

Peer review under the responsibility of the Faculty of Dentistry, Tanta University

ELSEVIER Production and hosting by Elsevier

http://dx.doi.org/10.1016/j.tdj.2014.03.004

1. Introduction

Lately, restorative dentistry has undergone an important paradigm shift. The concept of drill & fill by G.V. Black, has been replaced by the current trend of 'minimally invasive' dentistry, which is based upon minimizing the loss of sound tooth structure [1].

Resin bonded-composite has been introduced as a restorative material for posterior teeth [2,3]. The success of these restorations depends on bonding them to hard tooth tissue that will retain the restoration to the cavity preparation and prevent microleakage [4].

The principles of adhesive dentistry date back to 1955 when using techniques of bonding, postulated that acids could be used as a surface treatment before application of the resins [5], and found that etching enamel with phosphoric acid increased the duration of adhesion under water. However, bonding to dentin has a less reliable result due to its characteristics {collagen content, variable tubular structure, and outward dentinal fluid movement} [2]. Dentin bonding was further complicated by the presence of smear layer [6], age of teeth, direction of tubules and type of dentin [7].

Dentin bonding agents have been introduced to improve the adhesion to tooth structures, and to overcome these difficulties. Now, they are available in single-bottle systems to facilitate their use [8].

Manufacturer have improved the clinical performance of resin composite as posterior restorative materials; a recent type is *Packable composite*, in which there is incorporation of modified ceramic fibers (aluminum oxide & silicon dioxide) in addition to, or in place of, conventional inorganic filler particles. The ceramic fibers conduct light and allow curing depth up to 6 mm, thus allow for bulk placement of material and less curing time at chairside. Additionally, Packable resin composites have decreased polymerization shrinkage and increased wear resistance [9,10].

Studies evaluating the bond strength of different adhesive materials showed divergent findings. While some studies reported high bond strength [11–13]; other, however, showed lower values [14–16]. An explanation was given to the variation in the test methods between these studies.

The durability of the adhesive bond between resin and tooth structure is of significant importance for longevity of adhesive restorations. Long term stability of resin bonded dentin remains questionable. Hashimoto et al 2000 [17]demonstrated that the resin-dentin bond structures degraded in particular at the area of the hybrid layer when subjected to aging. *In vitro* laboratory studies reported decrease in bond strength after long water storage [18,19].

Cycling masticatory function in oral environment may fatigue the integrity of resin-tooth bonds, thereby permitting micro- or nanoleakage [20,21]. Other degradation promoting factors are residual solvent of the adhesive or insufficiently removed surface water [22]. Water was suggested to be incompletely removed and resulted in regions of incomplete polymerization and/or hydrogel formation making the hybridized adhesive–dentin interface more degradation sensitive. Clinically, marginal deterioration of resin composite remains problematic and forms the major factor that dramatically shorten the lifetime of composite-tooth bond [21].

Therefore, this research evaluate and compare the effect of water storage on the micro-shear bond strength of contemporary composite resins using three adhesives systems, **[etch-and-rinse]**, and **[self-etch] "one" & "two" step.**

2. Materials

The materials used in this study are shown in Table 1:

2.1. Methods

2.1.1. Specimen preparation

After obtaining signed written consent from each patient to use their own teeth in current research, ninety sound human third molar teeth were recently extracted in out-patient clinic of faculty of dentistry, Tanta University, and stored in 0.5% chloramine solution at 4 °C were used within 1 month after extraction. All the teeth were mounted in acrylic blocks (2 mm below cementoenamel junction) for ease of manipulation. For each tooth, a standardized box-shaped Class I cavity (4.5 × 4.5 mm) was prepared at the occlusal surface with the pulpal floor ending at mid-coronal dentin (depth 4 mm from cavity outline borders), using a high-speed hand piece with a cylindrical flat end carbide fissure bur (# 2, Dentsply Mailfere, Swiss) under water coolant [23].

The teeth were divided into *three equal groups* according *to type of adhesive* used (thirty teeth each):

- **Group I**: A two-step etch-and-rinse (total etch) adhesive *"Single bond 3M, EPSE, USA"*
- **Group II**: A two-step self-etch adhesive *"Clearfil SE bond, Kuraray, Japan"*.
- **Group III**: A one-step self-etch adhesive *"Futura bond NR,* Voco Cuxhaven, Germany".

Table 1
The materials used in this study.

Material	Components	Manufacture
Adper Single Bond 2 "Total-etch" (5th generation). Light cured	*Acid*: 37% phosphoric acid *Adhesive*: Bis-GMA,HEMA,dimethacrylates, polyalkenoic acid copolymer, initiators, water and ethanol Filled with 10% colloidal filler(5 nm).	3M, EPSE, USA
Clearfil SE bond "self-etch two step" (5th generation). Light cured	*Primer*: 10-MDP, HEMA, DHEPT, hydrophilic dimethacrylate, CQ, water. *Bond*: 10-MDP , HEMA, DHEPT, hydrophilic dimethacrylate, CQ, hydrophobic dimethacrylate, Filled with 10% silanated colloidal silica filler.	Kuraray, Japan
Futurabond NR "self-etch one step" 6th generation). Light cured	*Liquid A* Water, ethanol, silicium dioxide *Liquid B* Acid modified methacrylate (methacrylate ester), HEMA, camphorquinone Filled with nanoparticles.	Voco Cuxhaven, Germany
"Clearfil APX" hybrid type Light-cured composite resin (shade A3)	Bis-GMA, TEGDMA, filler (Barium, SiO_2), approximately 2 nm in size.	Kuraray, Japan
"Filtek P60" A packable type Light cured composite resin (shade A3)	61% Vol. zirconia/silica Inorganic fillers (Approx. 0.01–3.5 μm). -The monomer consists of BIS-GMA, UDMA & BIS-EMA	3M, EPSE, USA

10-MDP = 10-mthacryloyloxydecyle dihydrogen phosphate, HEMA = 2–hydrodxy ethylemethacrylate, DHEPT = N, N-diethanol p-toluidine, CQ = Camphorquinone .

Then each group was further divided into two subgroups ($n = 15$) according to Class I *composite resin type* that was applied in 3 horizontal increments:

- **Subgroup A:** Hybrid type composite resin "Clearfil APX, Kuraray, Japan".
- **Subgroup B:** Packable type composite resin "Filtek P60, 3M, EPSE, USA".
- The materials were handled according to manufacturer's instructions.

2.1.2. Occlusal loading & thermo-cycling procedures

The restored teeth were subjected to a maximum vertical load of 10 kg with cyclic frequency of 1.7 Hz for 120.000 cycles which simulates 6 months clinical use [24]. Attempts were made to assure that all specimens were kept wet during loading procedures.

After load cycling, all teeth were thermo-cycled in thermo-cycling apparatus for 500 cycles from 5 °C to 55 °C with 30 s dwell time, 20 s transfer time, corresponding to 6 months clinical use [25].

After thermal and mechanical load cycling, the teeth of each subgroup were divided into three equal divisions (five teeth each), according to storage time in 0.5% chloramine in distilled water (which is changed periodically every day), at 37 °C, in an incubator:

Division 1: water storage for 24 h (base line),
Division 2: water storage for 3 months, and
Division 3: water storage for 6 months.

2.1.3. Micro-shear bond strength test

After thermo-mechanical load cycling and water storage, the restored teeth were sectioned perpendicular to the composite-tooth interface with intervals of 1 mm using an Isomet diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) under water coolant, starting at resin composite side through dentin substrate. The cutting was advanced to Cemento-enamel junction to keep the slabs fixed in position. Then each tooth was rotated 90° and sectioned again perpendicular to the adhesive–tooth interface to obtain rectangular sticks, then sectioned at its cervical portion to separate the microspecimens [23].

These serial sectioning led to formation of numerous rectangular microbars or slabs in the form of beams with cross-sectional bonded areas of approximately 1 mm^2. Each microbar was formed of two different substrates: resin composite and dentin.

As a result of this cutting procedure, premature failure or debonding occurred; these were discarded (3–4 per tooth). Intact microslabs (180 microslabs, 2 from each tooth) with proper dimensions were selected

Table 2
Microshear bond strength (mean ± SD) of tested Adhesives/Composites resin combinations at 24 h.

Groups (adhesives) subgroups (composites)		Group I SB2	Group II CSE	Group III FB	ANOVA $F(P)$
Subgp.A (APX)		32.58 ± 1.42[a]	37.28 ± 1.06[a]	30.55 ± 2.09[a]	45.92 (0.000*)
Subgp.B (P60)		31.96 ± 1.66[b]	37.00 ± 2.12[b]	29.79 ± 1.17[b]	45.33 (0.001*)
T-test (*p*-value)	*T*	1.623	0.347	1.004	
	P	0.122	0.713	0.329	
Between groups regardless of sub-groups $F(P)$		4.40 (0.02)*			

Different groups in each subgroup which have the same letter (a or b) are significant to each other.
Tested subgroups in each group which have the same letter (a or b) are significant to each other.
*Significant at 95% level of confidence ($P \leq 0.05$).

using micrometer device to an accuracy of 0.001, to perform the microshear bond strength test.

An attachment for micro-shear bond strength test was especially designed to facilitate accurate alignment of microbar with the applied force during testing.

Using this especially designed attachment, the sticks will be mounted to a universal testing machine,[1] and stressed at a crosshead speed of 0.5 mm/min until failure occur.

Then the micro-shear bond strength (μSBS) was expressed in MPa, as derived from dividing the imposed force (N) at the time of fracture by the bonded area (mm2), according to the following formula:-

$$\text{MPa} = \text{Kg}/\text{cm}^2 \times 0.09807.$$

The data obtained from the test were collected, tabulated, and statistically analyzed using SPSS version 16, IBM Corporation.

3. Results

3.1. Micro-shear bond strength test results

The mean and standard deviation (±SD) of the microshear bond strength (μSBS) of different tested adhesives and/or composites at the tested storage time (24 h "base line", 3 months, and 6 months) were recorded.

The statistical analysis was performed using independent samples *T*-test at 95% level of confidence (Tables 2–4).

The results showed that composite type had no statistical significant effect on the microshear bond strength of tested adhesives at different water storage periods. On the other hand it was necessary to compare the μSBS of different tested adhesives (groups) at each subgroup at different storage time. Statistical analysis was performed using Analysis of Variance (ANOVA), whenever a statistical significant difference was recorded, Pairwise comparisons between tested adhesives was performed using Tukey's HSD pairwise comparison test, at 95% level of confidence (Table 5).

F test revealed a statistical significance difference among the tested groups at subgroup A, after 24 h recording F (P) value of 45.92(0.000). Thus Tukey's HSD pairwise comparison test was performed and recorded statistical significant differences among all tested groups (adhesives) ($P \leq 0.05$). Similarly at 3 months and 6 months water storage in subgroup A. Tukey's HSD pairwise comparison test recorded statistical significant differences among all tested groups ($P = 0.000$) at both storage times.

Regarding subgroup B, Tukey's HSD pairwise comparison test revealed a high statistical significance difference among all tested groups ($P = 0.000$).

Moreover similar findings were obtained, at both 3 months and 6 months water storage. Analysis of variance revealed a statistical significant difference among the adhesives used [F(P) = 62.48(0.001) and 45.33(0.001) at 3 and 6 month respectively], therefore Tukey's HSD pairwise comparison test was performed recording a high statistical significant difference among all tested adhesives ($P = 0.000$) at 3 and 6 months storage time.

However the composite type did not affect the μSBS of the tested adhesives at different water storage times, thus it was necessary to compare the results obtained from tested adhesives (groups) regardless the subgroups. Analysis of variance demonstrated a statistical significant difference among different tested adhesives recording F (P) values of = 4.40(0.02), 9.49(0.003) and 22.64(0.000) at 24 h (Table 2), 3 months (Table 3) and 6 months (Table 4) respectively.

On the other hand, to evaluate the effect of storage time on the bond strength, one way analysis of variance

[1] Buehler Ltd., Lake Bluff, IL, USA.

Table 3
Microshear bond strength (mean ± SD) of tested Adhesives/Composites resin combinations at 3 months.

Groups (adhesives) subgroups (composites)		Group I SB2	Group II CSE	Group III FB	ANOVA $F(P)$
Subg A. (APX)		31.82 ± 2.12[a]	36.77 ± 2.32[a]	26.89 ± 1.53[a]	59.88 (0.000)*
Subg B. (P60)		31.35 ± 1.76[b]	36.46 ± 1.73[b]	25.96 ± 2.67[b]	62.48 (0.001)*
T-test (*p*-value)	*T*	0.539	0.339	0.955	
	P	0.597	0.739	0.352	
Between.groups Regardless of sub-groups $F(P)$		9.49 (0.0003)*			

Different groups in each subgroup which have the same letter (a or b) are significant to each other.
Tested subgroups in each group which have the same letter (a or b) are significant to each other.
*Significant at 95% level of confidence ($P \leq 0.05$).

(ANOVA) was used to compare the μSBS among the different storage time of each composite and adhesive used in this study (Table 5). Whenever a statistical significant difference was recorded, Tukey's HSD pairwise comparison test was done between each two tested storage periods.

Since no significant difference among different tested storage periods (divisions) at both groups I & II was recorded, thus student *T*-test was used to compare different tested subgroups at each group regardless tested division. Table 6, demonstrated that no statistical significant difference was recorded between both types of composites.

4. Discussion

The tested Adhesives *were Single Bond 2, Clearfil SE Bond and Futura Bond NR*, representing different types of adhesives. "Two-step total-etch" filled adhesive system, which utilize acid etching before bonding procedure, "Two-Step, Self-etch" filled primer, and One-Step Self-Etch nano-filled adhesive respectively. In addition, composite resins tested represented different categories, *Hybrid composite* (Clearfil APX) based on Bis-GMA, TEGDMA and inorganic filler "Barium& SiO_2" (approximately 2 μm in size). The second was a *packable composite* (Filtec P60) based on Bis-GMA, UDMA, Bis-EMA, and Zirconia/silica inorganic filler (approximately 3.5 μm in size).

In the present study, LED curing unit was used to overcome the decrease of light intensity of the halogen light curing units over time due to bulb and filter aging 26. Also, LEDs have a working lifetime of over 10,000 h, compared to 40–100 h for halogen bulb [27], and wavelength peaks around 470 nm, which is nearly similar to the most commonly used photoinitiator camphorquinone (CQ) in dental composites so negating the need for filters. Furthermore, the thermal emission of the LED light curing units is significantly lower than that of halogen light curing units. It was concluded that the degree of conversion and depth of cure of LEDs were higher compared to than halogen light curing unit [28,29].

Most *in-vitro* studies evaluating bonding performance of adhesive materials use a flat dentin surfaces that did not resemble the clinical condition and have a low C-factor of 1/5 [19,30,31]. Clinically in a tooth cavity, shrinkage stress is generated during polymerization of the composite, pulling the adhesive from the cavity wall [32,33], and inducing gaps between the restoration and the cavity wall/floor that must result in micro-leakage [34]. This phenomenon is especially pronounced in a Class-I cavity with five bounded walls and only one free surface, revealing a C-factor of 5/1. In addition, the occlusal seal produced by bonding the adhesive to outer enamel margin of the occlusal class I cavities may have protected the bond of the adhesive to the class I bottom dentin against degradation [23].

Table 4
Microshear bond strength (mean ± SD) of tested Adhesives/Composites resin combinations at 6 months.

Groups (adhesives) subgroups (composites)		Group I SB2	Group II CSE	Group III FB	ANOVA $F(P)$
Subg A. (APX)		30.91 ± 1.39[a]	36.08 ± 1.91[a]	21.59 ± 1.78[a]	183.0 (0.001)*
Subg B. (P60)		30.38 ± 1.77[b]	35.94 ± 2.18[b]	21.41 ± 2.13[b]	45.33 (0.001)*
T-test (*p*-value)	*T*	0.743	0.150	0.205	
	P	0.467 (Not sig.)	0.882 (Not sig.)	0.840 (Not sig.)	
Between groups regardless of sub-groups $F(P)$		22.64 (0.000)*			

Different groups in each subgroup which have the same letter (a or b) are significant to each other.
Tested subgroups in each group which have the same letter (a or b) are significant to each other.
*Significant at 95% level of confidence ($P \leq 0.05$).

Table 5
Effect of storage time on the microshear bond strength of tested adhesives/composites combinations.

Groups & subg divisions	Group I (SB2)		Group II (CSE)		Group III (FB)	
	Subg A (APX) mean ± SD	Subg.B (P60) mean ± SD	Subg.A (APX) mean ± SD	Subg.B (P60) mean ± SD	Subg.A (APX) mean ± SD	Subg.B (P60) mean ± SD
24 h μ ± σ	32.58 ± 1.42	31.96 ± 1.66	37.28 ± 1.06	37.00 ± 2.12	30.55 ± 2.09[a]	29.79 ± 1.17[b]
3 month μ ± σ	31.82 ± 2.12	31.35 ± 1.76	36.77 ± 2.32	36.46 ± 1.73	26.89 ± 1.53[a]	25.96 ± 2.67[b]
6 month μ ± σ	30.91 ± 1.39	30.38 ± 1.77	35.94 ± 2.18	36.08 ± 1.91	21.59 ± 1.78[a]	21.41 ± 2.13[b]
F(*p*)	**2.486 (0.102)**	**0.577 (0.568)**	**0.829 (0.447)**	**0.577 (0.568)**	**61.55 (0.000)***	**40.50 (0.000)***

The divisions in each subgroup which have the same letter a or b are significant to each other.
* significant at $P \leq 0.05$.

Thus currently, the performance of adhesives was evaluated in class I cavity design.

Aqueous Chloramine T solution was chosen as a storage solution because it has no adverse effect on the collagen of the dentin. On the hand, teeth stored in a refrigerator showed absence of bacteria [35,36].

To mimic the clinical situation more closely, artificial saliva solutions can also be used, but bond strength reductions obtained were similar to those obtained with pure water degradation [37].

Water storage is the most commonly used artificial aging technique. The bonded specimens are stored in fluid at 37 °C for a specific period. This period may vary from a few months [38]. So distilled water was used currently as storage media for bonded specimens, for 24 h, 3 months & 6 months.

In this study, specimens were subjected to thermocycling and cycling masticatory function to closely simulate the thermal and load cycling changes in the oral cavity. These changes may accelerate hydrolysis of interface components and subsequent uptake of water and extraction of breakdown products or poorly polymerized resin monomers [17,26].

In the present study micro-shear bond strength testing used ultra-small bonding areas which are believed to have fewer defects occurring at the resin-dentin interface. In addition, this technique allows several samples to be obtained from one tooth, therefore allowing for a better comparative test [39]. Despite several authors observed higher micro-shear bond strengths than conventional shear bond strengths because of the smaller surface area [30,33].

However, several authors revealed that, the results obtained by micro-shear bond testing did not differ substantially from those gathered following a micro-tensile bond strength protocol [40,41].

The current finding revealed that *Clearfil SE* bond recorded significantly the highest micro-shear bond strength at all tested water storage times. Clearfil SE Bond is a two-step mild self-etching adhesive, which comprises the application of an acidic primer and a hydrophilic adhesive resin.

This may be explained by the bonding mechanism of Clearfil SE bond which result from the simultaneous demineralization and infiltration of enamel and dentin. It does not remove the smear layer, it impregnates the smear plugs, fixing it at the tubules and form a continuum in the substrate incorporating the smear plug in the resin tag, which will lead to a shallow but uniform resin infiltrated dentin layer [42]. Furthermore, the simplification of the bonding technique, the elimination of both rinsing and drying steps, thus reduces the possibility of over-wetting or over-drying which have a negative effect on adhesion [21,43].

Also, the primer of Clearfil SE Bond contains 10-MDP *(10-mthacryloyloxydecyle dihydrogen phosphate)* which is a highly hydrophilic functional monomer dissolved in water resulting in a pH of 2 [44]. This monomer is believed to improve the wetting to moist tooth surface. In addition and has two

Table 6
Comparison among three tested adhesives with each composite regardless time of water storage ($n = 90$).

Subgroup group	Subgroup A (APX)	Subgroup B (P60)	*T*-test	
	Mean ± SD	Mean ± SD	T	*P*-value
I SB2	31.770 ± 1.760	31.230 ± 1.798	1.246	0.218
II CSE	36.753 ± 1.848	36.513 ± 1.896	0.497	0.621
III FB	26.343 ± 4.131	25.720 ± 4.023	0.592	0.556

hydroxyl groups that may chelate to calcium ions of dentin. Moreover; the residual hydroxyapatite around the exposed collagen fibrils remains available for additional chemical interaction with the functional monomers [21,43].

Another factor that may contribute to the favorable performance of Clearfil SE Bond is the fact that it is a nanofilled adhesive containing 10 wt% silica nanofiller, resulting in a thicker adhesive layer and thus a more flexible interface that may relieve interfacial tensile stress between the shrinking composite and the rigid dentin interface [45]. This was confirmed by Ikemura et al 2003 [46]who stated that addition of fillers reinforces the hybrid layer and to increase the bond strength and decrease the microleakage. In addition, Fanning et al 1995 [47] demonstrated that, the thick adhesive layer might also help to absorb the stresses caused by the occlusal loads and make the coefficient of thermal expansion of the adhesive resin closer to those of the dentin and resin based composites.

The current results confirmed the previous findings where the bond strength of all tested adhesives reduction was descending from 24 h to 6 months of water storage, which was confirmed by previous studies [17,19,20,39,40,42,44,48,49]. This reduction was significant only in case of Futura Bond NR, however it was not significant with Clearfil SE Bond and Single Bond 2.

This decrease in bonding effectiveness by time might be explained by degradation of interface components by water storage. Water sorption can decrease the mechanical properties of the polymer matrix, by swelling and reducing the frictional forces between the polymer chains '*plasticization*' [50], with passive hydrolysis and leaching effect of break-down products of previous mechanisms. This passive hydrolysis and leaching effect is the most important mode of degradation of resin-dentin bond over time [51,52].

Concerning the results of Clearfil SE Bond, similar data was obtained by Abdalla et al 2007a [51] who reported that Clearfil SE Bond was stable after direct and indirect water storage for 12 months. They explained this stability by the chemical bonding capacity of Clearfil SE Bond to remaining hydroxyapatite crystals, which might in turn create insoluble calcium salts. These insoluble salts may prevent the loss of resin over time [53].

Regarding total etch two-step "Single Bond 2" adhesive, the primary bonding mechanism is diffusion-based and depends on hybridization or infiltration of resin within the exposed collagen mesh as well as into dentin tubules. After *in situ* polymerization, this hybrid layer provided micromechanical retention 21. It is insignificant reduction after water storage for 6 months may be contributed to it requires a moist dentin surface before bonding. The rationale behind this is that as long as the dentin is kept fully hydrated, the dentin matrix does not collapse and free spaces are available allowing resin infiltration and good adhesion [12,54].

The microshear of *Futura Bond NR* (self-etch one-step) was drastically significantly decreased after 6 months water storage. This may be due to that hydrophilic and hydrophobic components present have antagonistic properties, which form a hybrid layer with incomplete adhesive infiltration into the dentin substrate. The formed hybrid layer exhibits microscopic water-filled channels that allow water movement from underlying dentin to the adhesive-composite areas [55]. Moreover, the water can diffuse back from the bonded dentin into hydrophilic adhesive resins after drying since hydrophilic resins attract water [56]. Thus, the increase in the amount of hydrophilic resin monomers in one-step self-etching adhesive compositions could have a negative effect on the durability of resin-dentin bonds [57].

These results were confirmed by Frankenberger & Tay 2005 [58] who describe the behavior of these materials as permeable membranes after polymerization. Water sorption and dissolution of the incompletely polymerized resin containing amphiphilic monomers may result in deterioration of the one-step self etch adhesive. In addition, the higher acidity and hydrophilicity of the acidic monomers increase the risk of hydrolytic degeneration [59,60].

On the other hand, Abdulla et al 2008 [50], found that microtensile bond strength of Futura bond NR was not significantly affected after 6 months of storage under continuous pulpal water pressure which might be due to difference of methodology.

5. Conclusion

1. The type of adhesive system plays an important role in durability and lifespan of composite restorations.
2. Two-step total-etch and two-step self etch primer adhesives showed better tolerance to water storage compared to one-step self-etch adhesive and the difference was statistically significant.
3. The type of composite restoration had no significant effect on the microshear bond strength of dental adhesives used in this study.

Further studies are needed to determine the clinical effectiveness using SEM studies of the contemporary adhesive systems and dental composites.

References

[1] Degrange M, Roulet JF. Minimally invasive restorations with Bonding. Chicago: Quintessence Publishing; 1997.

[2] Baratieri LN, Ritter AV. Four-year clinical evaluation of posterior resin-based composite restorations placed using the total-etch technique. J Esth Restor Dent 2001;13:50−7.

[3] Rosin M, Urban AD, Gärtner C, Bernhardt O, Splieth C, Meyer G. Polymerization shrinkage-strain and microleakage in dentin bordered cavities of chemically and light-cured restorative materials. Dent Mater 2002;18:521−8.

[4] Schneider BT, Baumann MA, Watanabe LG, Marshall Jr GW. Dentin shear bond strength of compomers and composites. Dent Mater 2000;16:15−9.

[5] Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955;34:849−53.

[6] Pashley D, Michelich V, Kehl T. Dentin permeability: effects of smear layer removal. J Prosthet Dent 1981;46:531−7.

[7] Duke ES, Lindemuth J. Variability of clinical dentin substrates. Am J Dent 1991;4:241−6.

[8] Miyazaki M, Tsubota K, Onose H, Hinoura K. Influence of adhesive application duration on dentin bond strength of single-application bonding systems. Oper Dent 2002;27:278−83.

[9] Leinfelder KF, Bayne SC, Swift EJ. Packable composites: overview and technical considerations. J Esthet Dent 1999;11:234−9.

[10] Jackson RD, Morgan M. The new posterior resins & a simplified placement technique. J Am Dent Assoc 2000;131:375−83.

[11] Schreiner RF, Chappell RP, Glaros AG, Eick JD. Microtensile testing of dentin adhesives. Dent Mater 1998;14:194−201.

[12] Cardoso PE, Braga RR, Carrilho MR. Evaluation of microtensile, shear and tensile tests determining the bond strength of three adhesive systems. Dent Mater 1998;14:394−8.

[13] Cardoso PE, Carrilho MR, Francci CE, Perdigao J. Microtensile bond strengths of one-bottle dentin adhesives. Am J Dent 2001;14:22−4.

[14] Chigira H, Manabe A, Hasegawa T, Yukitani W, Fujimitsu T, Itoh K, et al. Efficacy of various commercial dentin bonding systems. Dent Mater 1994;10:363−8.

[15] Chappell RP, Eick JD. Shear bond strength and scanning electron microscopic observation of six current dentinal adhesives. Quintessence Int 1994;25:359−68.

[16] Toba S, Veerapravati W, Shimada Y, Nikaido T, Tagami J. Micro-shear bond strengths of adhesive resins to coronal dentin versus the floor of the pulp chamber. Am J Dent 2003;16. Spec No: 51A-56A.

[17] Hashimoto M, Ohno H, Kaga M, Endo K, Sano H, Oguchi H. *In vivo* degradation of resin-dentin bonds in humans over J to 3 years. J Dent Res 2000;79:1385−91.

[18] Sano H, Shono T, Takatsu T, Hosoda H. Microporous dentin zone beneath resin-impregnated layer. Oper Dent 1994;19:59−64.

[19] Armstrong SR, Keller JC, Boyer DB. The influence of water storage and C-factor on the dentin-resin composite microtensile bond strength and debond pathway utilizing a filled and unfilled adhesive resin. Dent Mater 2001;17:268−76.

[20] Sano H, Yoshikawa T, Pereira PN, Kanemura N, Morigami M, Tagami J, et al. Long-term durability of dentin bonds made with a self-etching primer, in vivo. J Dent Res 1999;78:906−11.

[21] Abdalla AI, Felzer AJ. Morphological characterization of the interface between self-etching adhesives and vital dentin. Am J Dent 2007;20:305−8.

[22] Hashimoto M, Ohno H, Sano H, Tay FR, Kaga M, Kudou Y, et al. Micromorphological changes in resin-dentin bonds after 1 year of water storage. J Biomed Mater Res 2002;63:306−11.

[23] De Munck J, Shirai K, Yoshida Y, Inoue S, Van Landuyt K, Lambrechts P, et al. Effect of water storage on the bonding effectiveness of 6 adhesives to class I cavity dentin. Oper Dent 2006;31:456−65.

[24] Krejci I, Häusler T, Sägesser D, Lutz F. New adhesives in class V restorations under combined load and simulated dentinal fluid. Dent Mater 1994;10:331−5.

[25] Sibel A, Jason W, Donald E. Surface protection for newly erupting first molars. Compend Contin Educ Dent 2006;27:46−52.

[26] Miyazaki M, Hattori T, Ichiishi Y, Kondo M, Onose H, Moore BK. Evaluation of curing units used in private dental office. Oper Dent 1998;23:50−4.

[27] Ruggeberg FA, Twiggs SW, Caughman WF, Khajotia S. Lifetime intensity profiles of 11 light-curing units. J Dent Res 1996;75:380. Abstract no. 2897.

[28] Kurachi C, Tuboy AM, Magalhaes DV, Bagnato VS. Hardness evaluation of a dental composite polymerized with experimental LED-based device. Dent Mater 2001;17:309−15.

[29] Yap AUJ, Soh MS. Thermal emission by different light-curing units. Oper Dent 2003;28:260−6.

[30] Zheng l, Pereira PNR, Nakajima M, Sano H, Tagami J. Relationship between adhesive thickness and microtensile bond strength. Oper Dent 2001;26:97−104.

[31] De Munck J, Van Meerbeek B, Yudhira R, lambrechts P, Vanherle G. Micro-tensile bond strength of two adhesives to Er:Y AG-lased vs. Bur-cut enamel and dentin. Eur J Oral Sci 2002;110:322−9.

[32] Feilzer AJ, de Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoration. J Dent Res 1987;66:1636−9.

[33] Versluis A, Douglas WH, Cross M, Sakaguchi RL. Does an incremental filling technique reduce polymerization shrinkage stresses? J Dent Res 1996;75:871−8.

[34] Roulet JF. Marginal integrity: clinical significance. J Dent 1994;22:S9−22.

[35] Suzuki T, Finger WJ. Dentin adhesives: site of dentin vs. Bonding of composite resins. Dent Mater 1988;4:379−83.

[36] O'Brien JA, Retrief DH, Bradley, Denys FR. Shear bond strength of a new dentin bonding restorative system. Dent Mater 1988;4:179−83.

[37] Kitasako Y, Burrow MF, Nikaido T, Tagami J. The influence of storage solution on dentin bond durability of resin cement. Dent Mater 2000;16:1−6.

[38] Shono Y, Terashita M, Shimada J, Kozono Y, Carvalho RM, Russell CM, et al. Durability of resin-dentin bonds. J Adhes Dent 1999;1:211−8.

[39] De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, et al. Four-year water degradation of total-etch adhesives bonded to dentin. J Dent Res 2003;82:136−40.

[40] Burrow MF, Satoh M, Tagami J. Dentin bond durability after three years using a dentin bonding agent with and without priming. Dent Mater 1996;12:302−7.

[41] Burrow MF, Nopnakeepong U, Phrukkanon S. A comparison of microtensile bond strengths of several dentin bonding systems to primary and permanent dentin. Dent Mater 2002;18:239−45.

[42] Phrukkanon S, Burrow MF, Tayes MJ. Effect of cross-sectional surface area on bond strengths between resin and dentin. Dent Mater 1998;14:120−8.
[43] Phrukkanon S, Burrow MF, Tayes MJ. The influence of cross sectional shape and surface area on the microtensile bond test. Dent Mater 1998;14:212−21.
[44] Oliveira SS, Pugach MK, Hilton JF, Watanabe LG, Marshall SJ, Marshall Jr GW. The influence of the dentin smear layer on adhesion: a self-etching primer vs. A total-etch system. Dent Mater 2003;19:758−67.
[45] Abdalla AI, Garcia-Godoy F. Clinical evaluation of self-etch adhesives in class V non-carious lesions. Am J Dent 2006;19:289−92.
[46] Ikemura K, Tay FR, Kouro Y. Optimizing filler content in an adhesive system containing pre-reacted glass ionomer filler. Dent Mater J 2003;19:137−46.
[47] Fanning DE, Wakefield, Robbins JW. Effect of filled adhesive on bond strength in three dental bonding systems. Gen Dent 1995;43:256−62.
[48] Armstrong SR, Vargas MA, Fang Q, Laffoon JE. Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding system through 15-month water storage. J Adhes Dent 2003;5:47−56.
[49] Reis A, Rocha de Oliveira Carrilho M, Schroeder M, Tancredo LL, Loguercio AD. The influence of storage time and cutting speed on microtensile bond strength. J Adhes Dent 2004;6:7−11.
[50] Abdalla AI, Felzer AJ. Four-year water degradation of a total-etch and two self-etching adhesives bonded to dentin. J Dent 2008;36:611−7.
[51] Abdalla AI, El Eraki M, Felzer AJ. The effect of direct and indirect water storage on microtensile dentin bond strength of a total-etch and two self-etching adhesives. Am J Dent 2007;20:370−4.
[52] Carrilho MR, Carvalho RM, Tay FR, Pashley DH. Effects of storage media on mechanical properties of adhesive systems. Am J Dent 2004;17:104−8.
[53] Tay FR, Pashley DH. Aggressiveness of contemporary self-etching systems. I: depth of penetration beyond dentin smear layers. Dent Mater 2001;17:296−308.
[54] Garcia RN, de Goes MF, Giannini M. Effect of water storage on bond strength of self-etching adhesives to dentin. J Contemp Dent Pract 2007;7:046−53.
[55] Tay FR, Pashley DH. Have Dentin adhesives become too Hydrophilic? J Can Dent Assoc 2003;69:726−31.
[56] Pashley DH, Carvalho RM, Tay FR, Agee KA, Lee K-W. Solvation of dried dentin matrix by water and other polar solvents. Am J Dent 2002;15:97−102.
[57] Ferrari M, Tay FR. Technique sensitivity in bonding to vital, acid-etched dentin. Oper Dent 2003;28:3−8.
[58] Frankenberger R, Tay FR. Self-etch vs etch-and-rinse adhesives: effect of thermo-mechanical fatigue loading on marginal quality of bonded resin composite restorations. Dent Mater 2005;21:397−412.
[59] Van Dijken JW. Durability of three simplified adhesive systems in class V non-carious cervical lesions. Am J Dent 2004;17:27−32.
[60] De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 2005a;84:118−32.