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

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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 \( \mu \text{SBS} \) after 1 day, 3 months, and 6 months water storage.

Results: The mean \( \mu \text{SBS} \) values (±SD) for Subgroup I A (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.

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

* Corresponding author.
E-mail addresses: osamanada@hotmail.com, mohmagdkamel1@gmail.com, mohamed.kamel@dent.tanta.edu.eg (M.M. Kamel), hussien.abd Rabo@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).

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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”.

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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 mm². 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$^b$ | 30.55 ± 2.09$^b$ | 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)                      | $T$-test (p-value) | $T$-test (p-value) | $T$-test (p-value) | $T$-test (p-value) |
| $T$-test (p-value)                      | 1.623 | 0.347 | 1.004 |
| $P$-test (p-value)                      | 0.122 | 0.713 | 0.329 |
| Between groups regardless of subgroups $F(P)$ | 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.

$^a$Significant at 95% level of confidence ($P ≤ 0.05$).

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) ($P ≤ 0.05$). 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 (ANOVA) was used to compare the μSBS among the different storage time of each composite and adhesive

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

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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& SiO2” (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]. Thus currently, the performance of adhesives was evaluated in class I cavity design.

| 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* | 36.77 ± 2.32* | 26.89 ± 1.53* | 59.88 (0.000)* |
| Subg B. (P60) | 31.35 ± 1.76b | 36.46 ± 1.73b | 25.96 ± 2.67b | 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 ≤ 0.05).

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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 microtensile 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-methacryloyloxydecyl 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 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.
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