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# The Effect of Aging Restorations on the Shear Bond Strength of Three Dental Adhesive Systems at Different Time Intervals. (An in Vitro Study).

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**الخلاصة**

**الهدف**:تهدف الدرا سة لقياس قوة الربط الانزلاقي لثلاث انواع من المواد اللاصقة للاسنان،الذاتية اللصق،الكلية اللصق و نظام اللاصق الكلي الواحد ولمقارنة تأثيرات تعتيق الحشوات لفترات زمنية مختلفة:(يوم واحد،اسبوع و شهر واحد) على قوة اللصق مع عاج السن وفي درجة حرارة الغرفة. **المواد و الطرائق المستخدمة** :مائة و ثمانية من الاضراس مثبتة بمادة الاكريليك بعد معالجة سطحها بالورق الصقيل وبدرجات مختلفة. تم تقسيم العينات الى 36سن لكل مجموعة من المجموعات، استعملت مادة الراتنج المركب لعمل عمود من الحشوات فوق المواد اللاصقة المستعملة، قسمت المجاميع الى مجموعات فرعية ( 12)سن لكل مجموعة قسمت على المواد الثلاثة المستعملة للفترات الزمنية المذكورة. اختبرت العينات بماكنة الاختبار العالمية واستخرجت النتائج باستعمال اختبار دنكان متعدد المدى. **النتائج**: اعلى قيمة لقوة الربط الانزلاقي للمواد اللاصقة الذاتية في فترة الاسبوع الواحد والقيمة الاقل تكون في اليوم الواحد من الخزن بينما القيمة الاعلى للمواد اللاصقة الكلية تكون في مدة اليوم الواحد من الخزن والقيمة الاقل تكون في الشهر الواحد. المقارنة بين أنظمة اللصق ذات قدرة الحفر الكلي و الذاتي للاسنان ، أنظمة اللصق الذاتية تمتلك قوة التصاق أعلى معنويا من أنظمة اللصق الكلية( ذات الحفر الكلي) في مستوى 0،05 النوع الثالث(( ذات الحفر الكلي الواحد) من أنظمة اللصق هذه له نفس قوة التصاق ذوات الحفر الكلي مع الاسنان. (ETH & Prime 3.0) **الاستنتاجات** :* أنظمة اللصق ذات الحفر الكلي و الذاتي تمتلك اعلى قيمة في قوة الربط الانزلاقي مع الاسنان بعد اسبوع واحد. * قوة الربط هذه للمواد اللاصقة المستعملة تتأثر بنوع الرابط المستخدم باختلاف الفترة الزمنية للاستعمال. *القيمة العالية لقوة الربط الانزلاقي مع الاسنان للأنظمة اللاصقة لايعني انه الافضل عند استعما له سريريا على المرضى لكن التوافق الكيميائي الحيوي،الحامضية و التسريب المجهري هي عوامل مهمة عمليا من الممكن ان تؤثر على قوة حشوات الاسنان في المستقبل.

## ABSTRACT

**Aims:** To measure the shear bond strength of three dental adhesive systems, self-etching priming system (Clearfil SE Bond), total-etch adhesive system (Prime &Bond NT) and all-in one adhesive (Etch & Prime 3.0 (E&P)), also to compare the effects of aging restorations with different time intervals (1-day, 1-week and 1-month) on dentin bond strength at room temperature. **Materials and Methods:** One hundred and eight human molars were embedded in self-cured acrylic resin, abraded on a water-cooled and polished with 80-400 grit sand papers to obtain standard dentin surfaces. The specimens were randomly assigned into three groups: (36 teeth) for Clearfil SE Bond, (36) for Prime&Bond NT and (36) for Etch & Prime 3.0 (E&P) adhesive systems were used at room temperature. These adhesive systems were applied to dentin surface according to the manufacturers' instructions. A composite resin (Tetric composite resin (USA)) cone was bonded to dentin surface. These groups were sub-divided into 3 subgroups (n=12), as in the following: (36) for Clearfil SE Bond; (12) were stored at 1-day, (12) at 1-week and (12) at 1-month time intervals, with the same criteria for Prime&Bond NT and Etch & Prime 3.0 (E&P). The specimens were stored in distilled water at room temperature and submitted to Universal Testing Machine at a crosshead speed of 0.5 mm/min. Means in MPa were analyzed statistically by Duncan′s Multiple Range Test at significant level of ($p>0.05$). **Results:** The results showed higher means of shear bond strength for Clearfil SE Bond and Prime &Bond NT were at (1-week) interval. While the higher bond strength for Etch & Prime 3.0 (E&P) were at (1-day) interval. The lower means of bond strength for Clearfil SE Bond were at (1-day). While for Prime &Bond NT and Etch & Prime 3.0 (E&P) were at (1-month) time interval. Etch & Prime 3.0 presented lower means of bond strength than Prime & Bond NT at ($p<0.05$). The results of this study revealed that Clearfil SE Bond showed the highest bond strength than other adhesives used in the study, which was significantly differ from Prime&Bond NT and Etch & Prime 3.0 (E&P) adhesive systems for the three time intervals used. **Conclusions:** In conclusion, a statistical difference between adhesive systems used in bond strength to dentin were observed when adhesives used with different aging intervals for (1-day, 1-week and 1-month). Self-etch bonding systems was showed a higher strength than total-etch adhesive systems. Etch & Prime 3.0 showed nearly equal bonding strength with the total-etch adhesives. The bonding strength

of adhesive system was influenced by the type of the bonding system used with different time intervals.

**Key Words:** Aging restorations, Bond Strength, Adhesive Systems , Different Time Intervals.

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## INTRODUCTION

Many investigators used extracted human teeth to evaluate the adhesive strength characteristics of dental adhesive and restorative materials. The widespread use of the teeth in vitro bond strength studies simulates in vivo situations. However, because of recent progress in conservative dental treatment, there is great difficulty in finding sound, non-carious human teeth for in vitro bonding studies. It was stated that the various structural components and properties of dentin could directly affect the adhesive bond [(1)] .

Adhesive systems bonding strength values to dentin may change due to location of the bonding area. Water content and permeability of dentin is not identical for all regions because of variations in the number of tubules per *mm.2*. Tubule number, density and peritubuler dentin area decreases with distance from the pulp, and intertubuler dentin area increases with distance from the pulp[(2)] .

The introduction of the acid etching technique and the development of hydrophilic monomers have made the use of adhesive systems possible in dentistry [(3,4)]. Currently, the use of adhesive systems on dentin substrate is more effective. The presence of a hybrid layer increases the bond strength and promotes the sealing of dentin surfaces by means of a resin-impregnated zone on decalcified dentin, preventing the microleakage of toxic products and consequently post-operative pain and restoration failure. The quality of the adhesion to the dentin substrate has been evaluated by laboratory tests such as tensile or shear tests. In 1991, ISO created a specification [Guidance on testing of adhesion to tooth structure. ISO/TC106/SC 1 N236, Resolution 61 – CD TR 11405, Trieste, October, 1991] for dentin bond tests which suggests a standard specimen storage period for which durability is analyzed. This standard demonstrates that specimens may be stored in distilled water at 37°C for few days to months. Bonding durability is an important factor for analysis[(5)].

Buonocore *et al*., (1956) [(6)] reported that after 1- month storage, a decrease in bond strength occurred. Kiyomura (1987) [(7)], Burrow *et al*., (1996) [(8)] and Sinhoreti *et al*., (2001) [(9)] found that bond strength decreased in vitro tests after long storage periods. Sano *et al*., (1996) [(10)] performed a study on monkeys, which was recorded that the degradation of the hybrid layer was occured after 1- year, observing porosity at its base.

This study was carried out to evaluate the means of shear strength of Clearfil SE Bond, Prime&Bond NT and Etch & Prime 3.0 (E&P) adhesive systems, an in vitro, also to determine the effects of aging restorations for (1-day, 1-week and 1-month) intervals on the bond strength of human dentin substrate of adhesive systems used.

## MATERIALS AND METHODS

Three commercially available adhesive systems and Tetric Ceram composite resin (Ivoclar Vivadent AG,FL-9494 Schaan /Liechtenstein) were used according to manufacturer instructions.

The selected human teeth were thoroughly cleaned and washed under running tap water and all adherent soft tissues were removed. Teeth were scaled with a periodontal scaler to remove organic debris before cleaning with water/pumice slurry. The teeth were stored in distilled water at room temperature until the time of testing for (1-day, 1-week and 1-month) intervals [(11)].

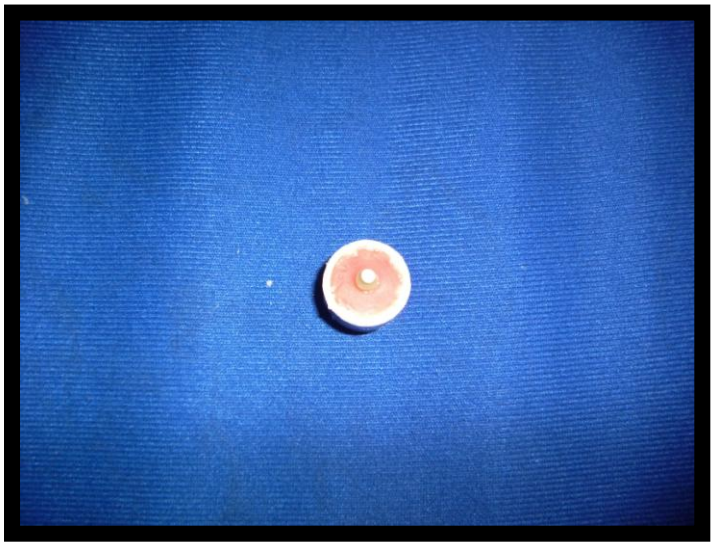
After cleaning, all human teeth roots were embedded into an autopolymerizing acrylic resin formed by a standard size plastic ring (15 mm height and 25 mm diameter). Crowns were cut at approximately 0.5 cm from the cementum-enamel edge

and polished using 80, 120, 220, 320 and 400 grit sand papers (Carborundum Abrasivos, Recife, PE, Brazil) on an automated polisher APL-4 (Arotec Ind. Com. Ltda., São Paulo, SP, Brazil) under water-cooling until a (5mm area in diameter) of dentin was obtained. Adhesive tape with a hole in the center (4mm in diameter) was adhered to the prepared dentin surface, delimiting the area to be used to bond the composite resin. The specimens were randomly divided into three equal groups (36 teeth for each) as in the following: (36) for Clearfil SE Bond group and (36 teeth) for each Prime&Bond NT and Etch & Prime 3.0 (E&P). The adhesive systems were applied according to manufacturer instructions [(11)]. Adhesive system used in this study was shown in Table (1).

Table (1): Adhesive system used in this study and their respective manufacturers.

| Adhesive system | Manufacturer |
|---|---|
| Clearfil SE Bond (CLSE) 2-step self-etching primer. | Kuraray Osaka, Japan |
| Prime & Bond NT (PBNT), one componant total etch. | Dentsply Milfort, DE, USA |
| Etch & Prime 3.0 (E&P),self conditioning all-in one adhesive. | Degussa Hülls Hanau, Germany |

To build the restoration, a plastic round mould (5 mm in height) with a central hole of (4 mm in diameter) with longitudinal cut was positioned over the specimens coinciding the central hole with the delimited area on the dentin. Composite resin was inserted in three increments, each one was light-cured for 40s with a light-curing unit XL-1500 (3M Dental Products, St. Paul, MN, USA). This was shown in Figure (1).



Figure(1): A figure represent sampling method of composite core on dentin surface of tooth build up with acrylic resin

At this point each of the three groups were sub-divided into 3 sub-groups (n=12), as in the following: (36) for Clearfil SE Bond group; (12 teeth) were stored for 1-day,(12) were stored for 1-week and (12) for 1-month. The same criteria (as mentioned before) were applied to Prime& Bond NT and Etch & Prime 3.0 (E&P) adhesive systems and were stored according to the same aging time intervals used

(1-day, 1-week and 1-month) in distilled water at 37°C (12).

After aging, the specimens were tested for shear bond strength in a Universal Testing Machine (Soil Test Co. Inc., USA) at a crosshead speed of 0.5 mm/min until failure occurred. The means of shear bond strength were determined by the following formula: S=T/A, where S= is the shear bond strength, T= is the tension applied, and A= is the bonded area. The shear bond strength was recorded in Newtons and converted into shear bond strength in (Mpa). When the shear test was finished, the specimens were examined by a stereomicroscope (Zeiss, model MC 63A, Germany) at 20X magnification (13). The results were submitted to Analysis of Variance and Duncan's Multiple Range Test at a significance level of ($p>0.05$). CLSE Bond demonstrated the highest mean shear bond strength at (1-week) which was significantly different at (1-day) interval ($p>0.05$). In addition, (CLSE) Bond had the highest means at all times tested. (E&P) had the lowest mean shear bond strength at (1-month), which was statistically lower than means at 1-day ($p<0.05$).

## RESULTS

As shown in Table 2, Clearfil SE Bond were statistically different from Prime & Bond NT and Etch & Prime 3.0 at all times ($p>0.05$). Clearfil SE Bond had the highest mean at 1-week (8.18), which was statistically different from 1-day value (4.98) at ($p<0.05$). The highest means for Prime & Bond NT were at 1-week (3.41) and the lowest means were at 1-month of aging (2.74). The highest means for and Etch & Prime 3.0 were at 1-day of aging (3.56) at ($p>0.05$) and the lowest means were at the 1-month (2.31).

Table(2): The mean of Shear Bond Strength in MPa, Standard Deviation, Minimum,Maximum and Duncan's Multiple Range Test after 1-day.

| Materials | N | Mean±SD | Minimum | Maximum |
|---|---|---|---|---|
| Clearfil SE Bond | 12 | 4.980±0.298 c | 4.50 | 5.43 |
| Prime & Bond NT | 12 | 2.952±0.097 a | 2.82 | 3.15 |
| Etch & Prime 3.0 | 12 | 3.569±0.276 b | 3.08 | 3.91 |

Means with different letters vertically have significant difference at $p\leq0.05$

Clearfil SE Bond presented the highest shear bond strength, which was significantly different ($p<0.05$) from the values obtained for the other systems of the other groups.

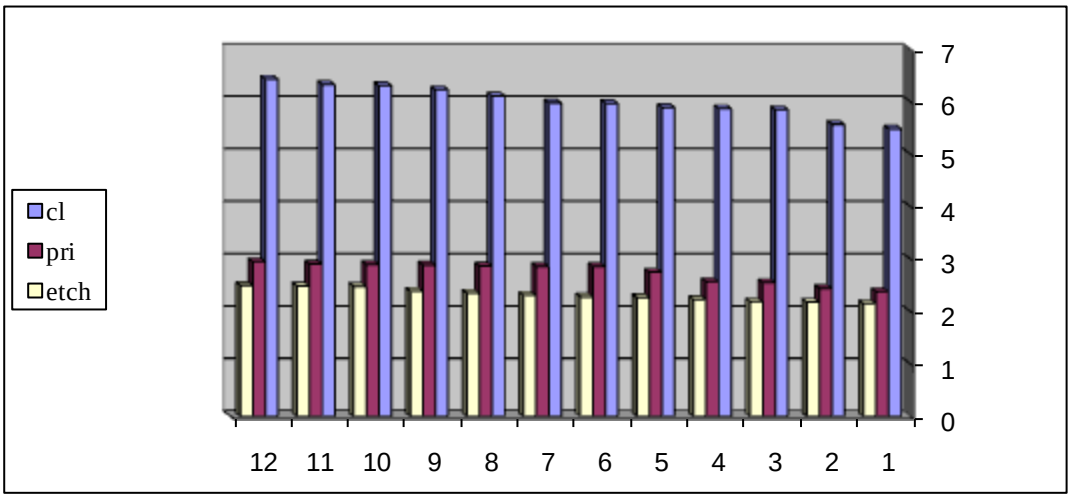
Etch & Prime 3.0 presented lower means of bond strength than Prime & Bond NT at ($p<0.05$).

After 1-month of aging, Clearfil SE Bond presented the highest means (6.0), which were statistically different from those of the other systems ($p<0.05$), followed by Prime & Bond NT (2.74) and then Etch & Prime 3.0 (2.31) at ($p>0.05$). This was shown in Table (4) and Figure (4).

Table(4): The mean of Shear Bond Strength in MPa, Standard Deviation, Minimum,Maximum and Duncan's Multiple Range Test after 1-month.

| Materials | N | Mean±SD | Minimum | Maximum |
|---|---|---|---|---|
| Clearfil SE Bond | 12 | 6.001±0.295 c | 5.48 | 6.43 |
| Prime & Bond NT | 12 | 2.744±0.202 b | 2.38 | 2.95 |
| Etch & Prime 3.0 | 12 | 2.318±0.124 a | 2.15 | 2.50 |

Means with different letters vertically have significant difference at $p \leq 0.05$



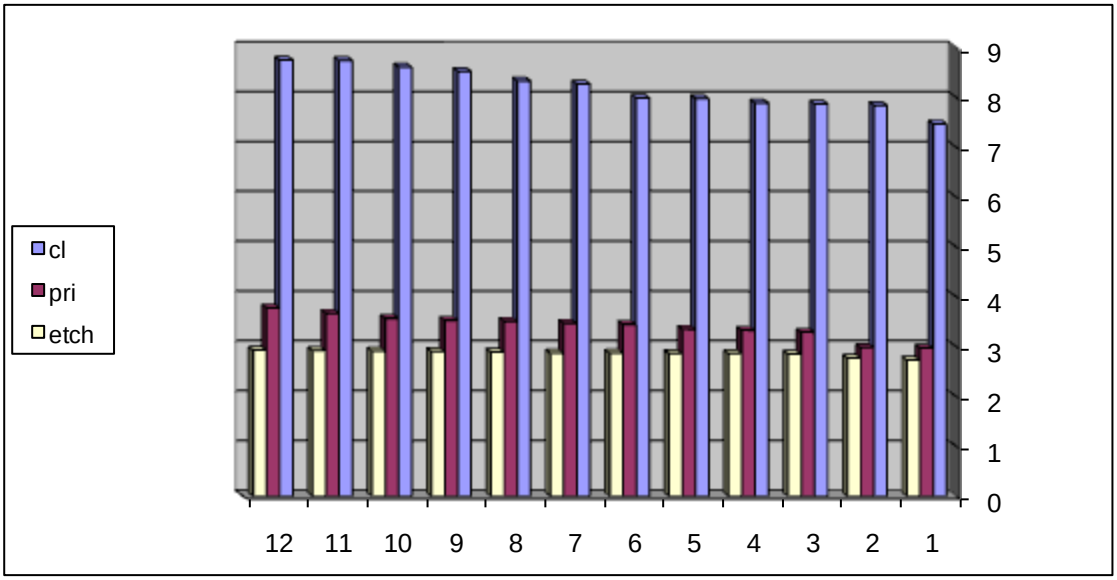

Figure(4): A histogram representing the mean of shear bond strength (Mpa) of the three adhesive system used after 1-month time interval.

After 1-week of aging, Clearfil SE Bond presented the highest means (8.18), which were statistically different from those of the other systems ($p<0.05$), followed by Prime & Bond NT (3.41) and then Etch & Prime 3.0 (2.86) at ($p>0.05$). This was shown in Table (3) and Figure (3).

Table(3): The mean of Shear Bond Strength in MPa, Standard Deviation, Minimum,Maximum and Duncan's Multiple Range Test after 1-week.

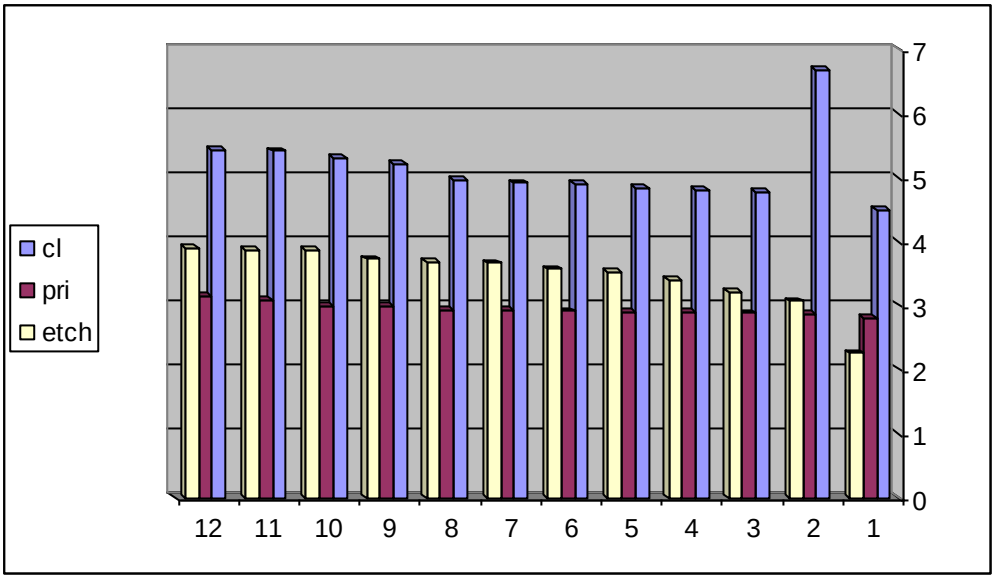
| Materials | N | Mean±SD | Minimum | Maximum |
|---|---|---|---|---|
| Clearfil SE Bond | 12 | 8.188±0.409 c | 7.47 | 8.75 |
| Prime & Bond NT | 12 | 3.410±0.246 b | 2.97 | 3.78 |
| Etch & Prime 3.0 | 12 | 2.863±0.059 a | 2.73 | 2.93 |

Means with different letters vertically have significant difference at p≤0.05




Figure(3): A histogram representing the mean of shear bond strength (Mpa) of the three adhesive system used after 1-week time interval.

After 1-day of aging, Clearfil SE Bond presented the highest means (4.98) , which were statistically different from those of the other systems (p<0.05), followed by Etch & Prime 3.0 (3.56) and then Prime & Bond NT (2.95) at (p>0.05). This was shown in Table (2) and Figure (2).




Figure(2): A histogram representing the mean of shear bond strength (Mpa) of the three adhesive system used after 1-day time interval.

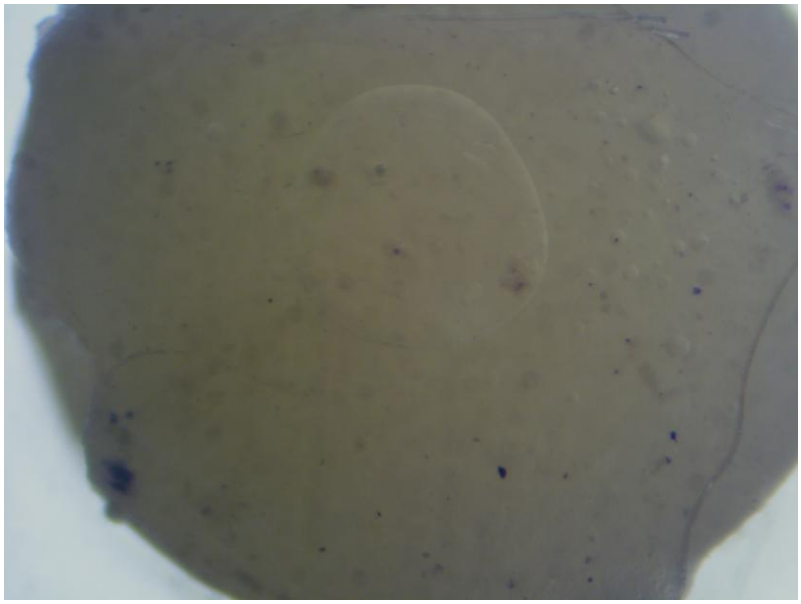
The examination of the debonded specimens under a stereomicroscope at 20X magnification showed that the majority of failures were adhesive for Prime & Bond NT and Etch & Prime 3.0 adhesive systems at (1-day, 1-week and 1-month). This was shown in Table (5), Figure (5 and 6) For Clearfil SE Bond adhesive system, the majority of failures were mixed at (1-day, 1-week and 1-month). This was shown in Table (5), Figure (7).

Table (5): Modes of Failure of Adhesive used in this study (in percentage).

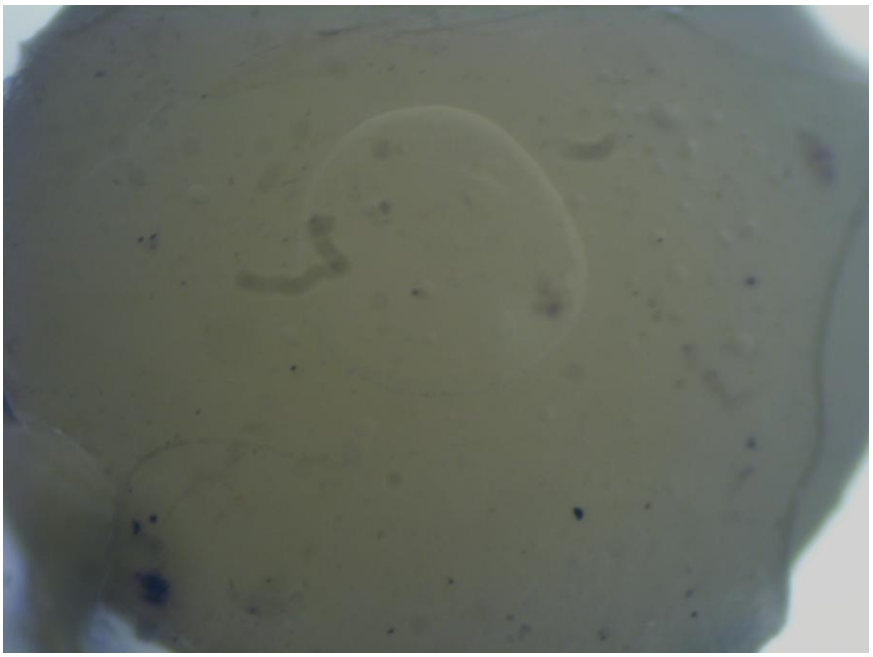
| | **1 day** | | **1week** | | **1 month** | |
|---|---|---|---|---|---|---|
| | **Adhesive** | **Mixed** | **Adhesive** | **Mixed** | **Adhesive** | **Mixed** |
| CLSE | 40 | 60 | 0 | 100 | 0 | 100 |
| PBNT | 90 | 10 | 90 | 10 | 90 | 10 |
| E&P | 100 | 0 | 100 | 0 | 100 | 0 |

Percentages of occurrence values were approximated to nearest number.

Figure(5): A figure representing adhesive fracture for Prime & Bond NT bonding agent at 1-month interval in stereomicroscope at 20X magnification.



Figure(6): A figure representing adhesive fracture for Etch & Prime 3.0 bonding agent at 1-month interval in stereomicroscope (20X magnification).



Figure(7): A figure representing mixed fracture for Clearfil SE Bond at 1-month interval in stereomicroscope at 20X magnification.

## DISCUSSION

In vitro tests attempt to simulate the clinical situation under laboratory conditions. Some times immediate results are obtained, but the effect of storage over time is not investigated, erroneously evaluating efficiency of the adhesive system. Therefore, tests that try to age the specimens by storing immersed in water are necessary [14] .

In this study, the lowest shear bond strengths were obtained for Etch & Prime 3.0 and they were similar to those obtained with Prime & Bond NT after 1-month (2.31 and 2.74 Mpa) respectively. This result may be due to an incomplete infiltration of the acidic monomer and dissolution of the smear layer occurred in specimens.

Sinhoreti *et al.*, [15] related partial dissolution of the smear layer with some closed dentinal tubules, resulting in low shear bond strength. Watanabe and Nakabayashi [16] used an experimental primer containing phenyl-P dissolved in TEGDMA (triethyleneglycol dimethacrylate) trying to eliminate the collagen-rich zone, but its mechanical properties were weakened after storage in water and results obtained were not satisfactory.

Burrow *et al.*, [17] used tensile and microtensile tests, with different types of mechanical load on the substrate to compare shear strength. It appears to be evident that the histological complex of the bonded areas is probably more resistant to shear load than tensile load.

The lower values obtained in this study may be explained by the methodology used. The creation of a less complex mechanical load and debonding occurs as a result of sliding along the interface between the adhesive layer and dentin as a result of the high concentration of the tangential force, similar to that found in the inclined plane[18,19].

Prime & Bond NT had lower means of shear bond strength (2.95,3.41 and 2.74 Mpa) compared to Clearfil SE Bond for (1-day, 1-week and 1-month) respectively, probably due to that self-etching primers, Clearfil SE Bond (CLSEB) create diffusion channels into intact calcium-rich dentin. This prevents the loss of dentin mass but solubilizes enough apatite crystals from around collagen fibrils to permit infiltration of adhesive monomers. Therefore, hybridization created by self-etching primers is free from defects and is conti-

nuous from resin to calcium rich dentin. Furthermore, the bonding mechanism provided by self-etching primers may be more stable with time because collagen fibers are surrounded by hydroxyapatite crystals which might protect it against hydrolysis and early degradation of the bond. In terms of aging (storage) time, shear bond strength decreased over time. Some studies explain that the presence of water may degrade polymeric material and collagen fibrils that are not involved by the bond system contains colloidal silica in micrometric size which impedes penetration into demineralized dentin. Therefore, water may have penetrated and degraded the MDP (10 methacrylodecamethylene phosphoric acid) [20].

Prime & Bond NT (PBNT) is an acetone based solution of phosphoric acid esters containing PENTA monomer which possesses acidic properties with a PH of (2.2). Therefore, PBNT may present mild self-etching characterictics when applied to dentin with intact smear layer and produce bond strength value similar to acid etched dentin [21]. However, after long storage periods in water these properties may be affected, decreasing durability. Some authors claim that the collagen-rich zone is the weakest point of the bond. Since, this zone is unprotected by minerals or resin, it is more susceptible to proteio hydrolysis. Sano *et al.,* [22] observed that high collagen fibrils and resinous material and photomicrographs further support this explanation, since there were no cohesive failures in dentin .

Two of the three adhesive systems used (Clearfil SE Bond and Prime & Bond NT Bond) showed a tendency toward higher means (8.18 and 3.41 Mpa) respectively at (1-week). This was shown in Table (3). It seems that this aging period is not sufficient to cause perceptible bonding degradation. However, this tendency towards higher strength means at (1-week) of storage may be attributed to the release of stress generated during polymerization contraction. Thus, the present study showed that the bond strength after different aging periods in distilled water was influenced by type of adhesive system used [23].

The failure types found in this study may be a result of the methodology used. The examination of the debonded specimens by a stereomicroscope (Zeiss, model MC 63A, Germany) at 20X magnification showed that the majority of failures were adhesive for Prime & Bond NT Bond and Etch & Prime 3.0 at (1-day,1-week and 1-month), because of mild self-etching characterictics when applied to dentin and may produce bond strength value similar to acid etched dentin. However, after long storage periods in water these properties may be affected, decreasing durability. While, for Clearfil SE Bond adhesive system, the majority of failures were mixed failure at the same aging time intervals due to the bonding mechanism provided by self-etching primers may be stable with time as collagen fibers are surrounded by hydroxyapatite crystals which protect it against hydrolysis and early degradation of the bond. The results of this study showed that higher bonding strength was for self-etch adhesives when compared to total-etch and all-in-one adhesive systems and they eliminate post-treatment sensitivity because they etch and prime simultaneously but, from the clinical point, when evaluating microleakage using self-etching bonding systems Hanning found that self-etch sealing ability is less effective as compared to the conventional acid etching technique [24]. However Tay found that self-etch adhesives are potentially useful for bonding as fissure sealants [25]. Because of contraversy, further studies are needed for the recommondations of the use or non-use of these adhesive systems clinically, in regarding to sealing ability, acidity, strength, microleakage and biocompatibility .

## CONCLUSIONS

Comparison among total-etch and self-etch adhesive systems, self-etch systems bonding strength was found statistically higher than total-etch adhesive systems at ($p<0.05$). Etch & Prime 3.0 showed nearly equal bonding strength with the total-etch adhesives.

• Among total-etch and self-etch adhesive systems, the highest shear bond strength value was at (1-week) interval**.**

• The bonding strength of adhesive system was influenced by the type of the bonding system used in respective to different aging peroid.

• The higher value of shear bond strength of adhesives not mean the recommomdations for or against its use clinically; but biocompatibility, acidity and microleakage are an important factors in practice and may affects the strength of the restorations in the future.

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