

## EVALUATION OF THE ROLE OF DIFFERENT ADHESIVE SYSTEMS IN DURABILITY OF ENDODONTICALLY TREATED TEETH AFTER THERMOCYCLING

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### **ABSTRACT**

**Background:** The main reason suggested for the failure of endodontically treated teeth after a long period of time of intraoral performance is the lack of perfect coronal seal together with the presence of nanoleakage. Therefore, this study was conducted to evaluate to what extent the difference between adhesive systems may affect the seal achieved between pulp chamber dentin and the overlying coronal restoration together with nanoleakage evaluation.

**Materials and methods:** Two groups of root canal treated teeth were bonded with two types of adhesive systems (Total-etch and Self-Etch) and restored with nano-hybrid resin composite. All teeth were thermocycled (2,500 cycles, 5°C to 55°C, 20 seconds dwell time, and 5 seconds resting time). After thermocycling, teeth were sectioned into sticks of 1 mm<sup>2</sup> (15 sticks obtained from each group) for microtensile bond strength testing (mTBS). From each group, a slab of 1 mm<sup>2</sup> was obtained for micromorphological analysis to observe the quality of the resin-dentin interface by scanning electron microscope and to evaluate nanoleakage pattern through silver tracing as well. The collected data were expressed in MPa and analyzed by independent sample t-test.

**Results:** Regarding bond strength testing, highly significant difference was recorded between both adhesive systems ( $p < 0.05$ ). Total etch adhesive group showed higher results compared to the Self Etch one. Besides, SEM observations showed that all specimens showed different patterns of nanoleakage regardless of the type of adhesive system used.

**Conclusions:** Total etch adhesive system showed better results than self-etch adhesive one in achieving good seal to pulp chamber dentin in endodontically treated teeth after thermocycling.

**Keywords:** Microtensile Bond Strength; Self-etch Adhesive System; Total-etch Adhesive System; Nanoleakage; Thermocycling.

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## INTRODUCTION

Many patients receiving root canal treatment are wondering about how long this tooth will survive intraorally. In the past, it was known that root canal treated teeth don't survive for long periods, and the normal sequela was extraction. However, with the outstanding improvements of dental materials, these teeth were restored in many ways such as; resin-composite restorations, post and core systems, and endo-crowns.

Therefore, maintenance of a perfect coronal seal is of a great importance. *Torabinejad et al.* emphasized the importance of this seal and demonstrated that root canal fillings exposed to saliva may become contaminated regardless of the materials and obturation techniques used.<sup>(6)</sup> Where, the canal may be recontaminated in various ways such as contact between the oral bacterial flora and root canal tubule inlets which could retard healing and create infection in the periradicular, periodontal ligament or supporting osseous structures<sup>(5)</sup>. However, it most frequently occurs due to inadequate coronal sealing<sup>(4)</sup>. The placement of a suitable material over the coronal gutta-percha act as a barrier to coronal microleakage. Root canal treated teeth are subjected to multiple factors intruding within the root canal therapy that negatively affect bond strength durability such as irrigants causing dissolution of dentin<sup>(24,45)</sup> sealers, or temporary materials that change the surface wettability.

In the past, it was totally significant that endodontic failure resulted from leakage through obturated root canals, and especially in case of presence of accessory canals but nowadays, it is of a greater significance to complete the coronal restoration of a root canal treated tooth, especially if multi-rooted<sup>(7-9)</sup>.

This can be achieved through resin composites bonded directly to endodontically treated teeth through adhesives systems. The latter has to bond to pulp chamber dentin and create a defense line

against bacterial leakage in root canal treated teeth. These systems have undergone several changes lately, where All-in-One adhesives were introduced as time saving solution demineralizing and resin impregnating simultaneously, together with an excellent bonding strategy<sup>(2,3)</sup>. However, if these adhesives fail to resist polymerization stresses, it is inevitable that there will be micro-gaps between the tooth and composite<sup>(3)</sup>. Pulp chamber dentin is our major and influential problem due to inherent wetness<sup>(36)</sup> that negatively affects adhesion<sup>(27,28)</sup>. It differs from coronal dentin<sup>(33)</sup> in the high tubular density together with little intertubular dentin (permits hybrid layer formation). Moreover, using various irrigants/medicaments and temporary filling materials during endodontic treatment, stands against the success of the final restoration<sup>[10, 11]</sup>. Adhesives should provide sealing with pulp chamber dentin and strengthen root canal treated teeth through; coronal restoration leakage prevention<sup>(40,41)</sup>, increasing restorative materials retention<sup>(25)</sup> to dentin walls and increasing mechanical resistance to mastication stresses<sup>(25,26)</sup>.

Based on the previous data, the aim of this invitro study is to evaluate the influence of the type of adhesive systems, in terms of their bond strength and resistance to nanoleakage, on the durability of root canal treated teeth after Thermocycling.

## MATERIALS AND METHODS

Two commercially available light cured adhesive systems and a restorative resin composite were used in the present study. The materials' brand names, chemical compositions, and lot number are shown in table (1).

**Teeth selection:** Twenty extracted human mandibular molars were used in this study. Teeth were examined and thoroughly washed with water, scaled with periodontal scaler to remove any blood, attached periodontal tissues, plaque and calculus. The teeth were stored in saline solution at room temperature.

TABLE (1): Material name, composition, batch number and manufacturer:

Name	Composition	Batch Number	Manufacturer
<b>Adperä Single Bond2*</b> (total-etch adhesive)	BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acid	N716057	<b>(3M ESPE)</b>
<b>Single Bond Universal Adhesive*</b> (single-step self-etch adhesive)	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, copolymer, filler, ethanol, water, initiators, silane.	N601958	
<b>Filtek Z250 XT</b>	<b>The Filler System:</b> Surface-modified zirconia/silica with a median particle size of approximately 3 microns or less, Non-agglomerated/non-aggregated 20 nanometer surface-modified silica particles, filler loading is 82% by weight (68% by volume) <b>The Resin System:</b> BIS-GMA, UDMA, BIS-EMA, PEGDMA, TEGDMA	N721306	

*MDP:10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol-a-glycidyl methacrylate*

### Root canal treatment

After access cavity preparation, the working length was determined by measuring the length of a #10 K-file (Dentsply, Maillefer, Ballaigues, Switzerland) just visible at the apical foramen. Glyde path was achieved using Proglider (Dentsply, Maillefer, Ballaigues, Switzerland). The mesial canals were instrumented using ProTaper NEXT (Dentsply, Maillefer, Ballaigues, Switzerland) nickel titanium files X1, X2, while the distal canals were prepared to file X3. After each file 2ml, 5.25% NaOCl was used for irrigation. To eliminate the smear layer in the final irrigation 2ml 17% EDTA for 3 minutes and 2ml 5.25% NaOCl were used respectively. Following the preparation, the root canal was dried using paper points. Obturation was performed using master cone equivalent to the master apical files and Adseal as root canal sealer (Meta, Biomed, Korea) using # 25 spreader and #20 accessory gutta percha.

A small piece of cotton was placed over pulp orifices then teeth were restored with temporary restorative material. Teeth were kept in tap water

for 24 hours at room temperature. Then, temporary filling was removed and the pulp chamber was cleaned perfectly using an excavator followed by a cotton pellet with alcohol in order to remove the temporary filling and the remnant sealer on the wall of the access cavity<sup>14</sup>. The pulp chamber was finally cleaned with a cotton pellet with water and then blot dried

**Grouping of teeth:** Teeth were divided into two main groups of four molars each (n=10) according to the adhesive system used (A), namely A1; a Total-etch adhesive system (*Adper™ Single Bond 2*) and A2; and Self-Etch one (*Single Bond Universal*). Then, all teeth were restored with a light activated nano-hybrid resin-composite.

**Restoration of the prepared teeth:** Teeth were then restored using a composite resin (Filtek Z250 XT, shade A3, 3M ESPE, St. Paul, USA) after bonding with the assigned adhesive system.

**Thermocycling:** All samples were subjected to thermocycling (2,500 cycles, 5°C to 55°C, 20 seconds dwell time, and 5 seconds resting time (Mechatronic, Germany).

### Beam preparation for microtensile bond strength testing

Beam preparation was done in the Dental Research Centre, Cairo, Egypt. Each tooth containing acrylic block was mounted on **IsoMet 4000 micro saw buehler Germany** and sectioned to obtain sticks (beams) of 0.8-1 mm<sup>2</sup> cross-section area. As a protocol undertaken by *Takahashi et al. in 2010*, only the central sticks from each specimen were selected in order to eliminate substrate regional variability. Besides, sticks of similar length and remaining dentin thickness were tested, and their thickness was checked using a caliper (Mitutoyo, Tokyo, Japan). Specimens lost during manipulation or test preparation were not recorded and were not considered in the statistics as well.

### Microtensile bond strength testing

From each group 15 sticks were measured. Sticks were attached to a universal testing machine; **Wilson (Beuhler) micro hardness tester (Germany)** with cyanoacrylate adhesive (Zapit)\*. A tensile load with compression mode of force was applied at a crosshead speed of 0.5 mm/min with a load cell of 500N. The applied tensile force resulted in debonding along the dentin-adhesive interface for each stick was recorded in MPa (Newton divided by the area).

### Specimen preparation for nanoleakage

A 1mm thick slab was obtained from each group and immersed in 50 wt% ammoniacal silver nitrate solution in a small container and wrapped with aluminum foil paper. The container was then placed in a black photofilm container to ensure total darkness for 24 hours. The specimens were then rinsed thoroughly with distilled water and immersed in photo developing solution for 8 hours under a fluorescent light, to reduce the diamine silver ions into metallic silver grains, that cannot diffuse after reduction, within voids throughout

the resin dentin bonded interfaces<sup>(19-20)</sup>. Specimens were rinsed thoroughly with distilled water. Slabs were sandblasted using ascending grains and then polished with 3 alumina paste. Then, they were cleaned ultrasonically and air dried. Finally, specimens were coated with gold sputter.

### Nanoleakage assessment

Nanoleakage was qualitatively assessed using a scanning electron microscope (SEM) by backscattered electron image mode.

**Statistical analysis:** Microtensile bond strength data was collected tabulated, and analyzed statistically by independent sample t-test followed by two-tailed significance at  $p < 0.05$  level. The results were presented as means  $\pm$  standard deviations.

## RESULTS

### Statistical analysis of Microtensile bond strength (in MPa)

The data in *Table 2* and *figure 1* represent descriptive statistics and test of significance between the two adhesive systems tested. Independent t-test was used to compare mTBS values after Thermocycling. The test rendered highly significant difference at the level of significance  $p = 0.014$ . *Adper™ Single Bond 2* was significantly higher than *Single Bond Universal* with  $(25.72 \pm 7.09)$  and  $(19.88 \pm 4.90)$  respectively.

### Qualitative analysis of nanoleakage

The observation of the photomicrographs (figure 2) at 4000 $\times$  magnification by scanning electron microscope revealed that all the specimens showed nanoleakage manifested by silver penetration of different patterns and different degrees. However, not all identified shiny spots represent silver deposition. Moreover, a large gap was observed at the resin-dentin interface in all test groups.

TABLE (2) Microtensile Bond Strength Values (MPa) after thermocycling

	<i>Single Bond Universal</i>	<i>Adper™ Single Bond 2</i>
N	15	15
Minimum	12.79	17.97
Maximum	29.29	37.41
Mean	19.88	25.72
SEM	1.26	1.83
SD	4.90	7.09
Variance	24.00	50.25
Mean + SD	19.88 ± 4.90	25.72 ± 7.09
Mean + SEM	19.88 ± 1.26	25.72 ± 1.83
<b>Independent samples t-test</b>		
t-statistic		-2.62
df		28
Sig. (2-tailed)		.014
Mean Difference		-5.84
SE Difference		2.22

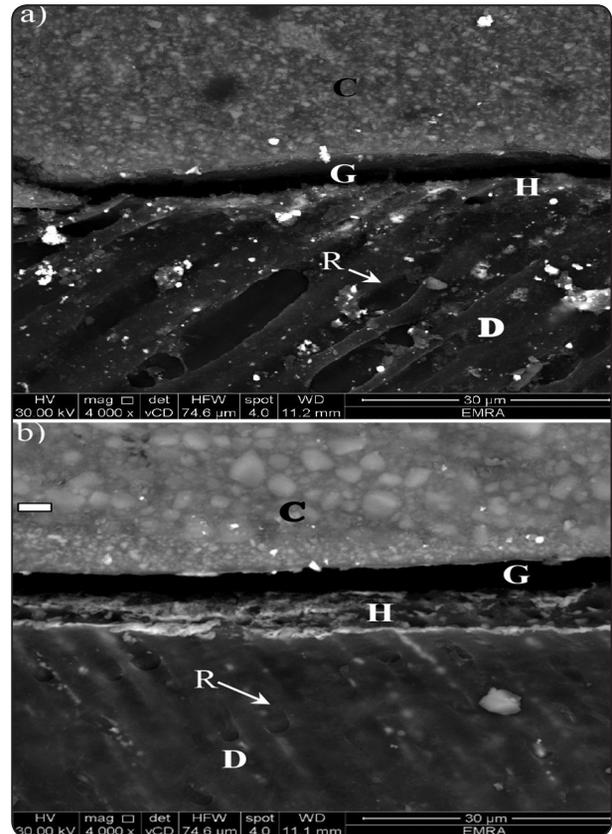


Figure (2): Representative SEM micrographs of resin-dentin interface (4000X magnification) after thermocycling (2,500 cycles). (a) Single Bond Universal Self-etch adhesive system. (b) Adper Single Bond2 Total etch adhesive system. C: Resin-Composite, G: Interface Gap, D: Dentin, R: Resin- tags, H: Hybrid Layer

**DISCUSSION**

**Thermocycling**

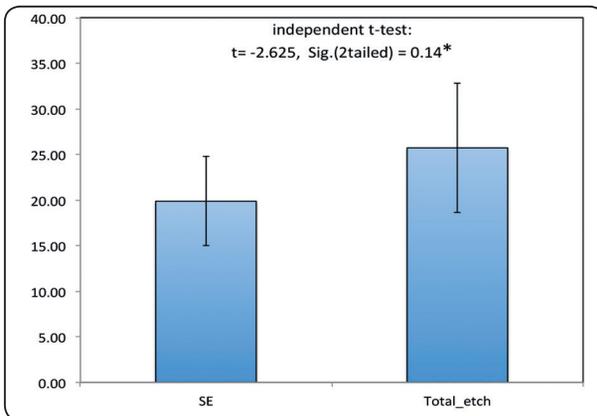


Fig. (1): Histogram showing the microtensile bond strength values for the two groups (in MPa)

Thermocycling was used to create an exact replica of the challenging intra oral environment including moisture, stress conditions, and variable temperature degrees. It is the most common way of stimulating the human's intake of food within the range of 5°C and 55°C, that are the minimum and maximum tolerable degrees respectively (17,31). Although more extreme temperatures could be encountered, but they cannot be taken as representative guidelines (6).

It has been reported that 10,000 cycles are equivalent to 1 year of intra oral performance (23). Therefore, specimens in the present study were

subjected to 2,500 cycles, for 20 seconds dwell time, and 5 seconds resting time representing 3 months of in vivo activity.

### Adhesive systems and how they influence bond strength

Microtensile bond strength testing was evaluated after thermocycling. More than seventy percent of published studies nowadays use microtensile bond test that was proposed by Sano *et al.* <sup>(7)</sup>. It has better use of specimens, greater control in determining the working area, and better stress distribution when compared with macro-tensile tests

Two adhesive systems were used due to different manipulation steps and compositions as shown in Table (1) and because of the successful results they proved in previous studies. After thermocycling, microtensile bond strength of *Adper™ Single Bond 2* was significantly higher than *Single Bond Universal*. This can be explained by the difference in composition where the latter contained MDP monomer which has strong affinity for calcium in its composition, and the dentin here had decreased calcium content which affects the bond strength negatively <sup>(12,13)</sup>. This decreased calcium content was due to the use of combined solution (2.5% hypochlorite and EDTA) during irrigation which was found to promote higher calcium removal from dentin <sup>(11)</sup>. Another explanation may be due to the presence of carboxylic group of the polyitaconic acids in *Adper™ Single Bond 2* which forms ionic bonds with hydroxyapatite on dentin causing a strong bond. <sup>(14,15)</sup> Besides, *Carvalho et al.* found that <sup>(46)</sup> using ZOE temporary material did not affect the bonding of the total etch system but affected the bonding of the self-etch system negatively. This happens if the surface is not properly cleaned, remnants of materials stand against good bonding and may also inhibit adhesive material polymerization through release of eugenol when exposed to water <sup>(46)</sup>.

Other studies were contradictory. Different results may be because of using different material content and different study methodology. They

stated that self-etch adhesives have better bond strength values than etch and rinse <sup>(24,32)</sup>. This may be explained by their application of 5% NAOCL to dentin prior to bonding. Besides, *Elbay and Tosun, Ozturk and Ozer* <sup>(24)</sup> and *Kijsamanmith et al.*, concluded that self-etch adhesives showed the highest significant mTBS values when compared with total etch adhesives. They explained this by that phosphoric acid application affected pre-dentin and collagen network, resulting in excessive demineralization and negatively affecting bonding. <sup>(48)</sup> Moreover, the high molecular weight MDP is able to promote an ionic bond to hydroxyapatite through the low solubility of the calcium salt on its surface, which organize themselves into highly hydrophobic nano-layers, thus protecting the hybrid layer from hydrolytic degradation <sup>(8)</sup>.

### Adhesive systems and how they influence nanoleakage

Although micro tensile bond strength testing is very important, nanoleakage measuring is considered as an important alternative <sup>(4)</sup> to evaluate the sealing of various adhesives to pulp chamber dentin, especially after Thermocycling.

Nanoleakage is defined as leakage pattern occurring within nanometer-sized spaces within the hybrid layer and the adhesive/resin interface. It is a crucial factor that leads to degradation of bonding to dental tissue <sup>(44)</sup>, and more specifically pulp chamber dentin. It occurs laterally, through submicron porosities (20-100 nm in width approx.) at the base of the hybrid layer, which has not been filled with adhesive resin or which have been left poorly polymerized <sup>(41)</sup>. These areas that are demineralized but not fully hybridized are considered very weak points in the adhesion mechanism. This may allow dentinal and oral fluid to slowly permeate the interface and degrade the adhesive resin <sup>(24)</sup>.

The commonly used tracer for nanoleakage assessment is Ammoniacal silver nitrate that

penetrates dentin because silver ion size is (0.059 nm in diameter). SEM micrographs (Fig.2) showed hybrid layer formation at the resin dentin interface in both adhesives groups after Thermocycling with the Total etch adhesive showing a thicker and more uniform layer with less evident resin tags.

An interfacial large gap was observed with both groups. This gap may be a result of the polishing of specimens before scanning. It might be also due to rupture of the interface from vacuuming <sup>(16)</sup>. Different nanoleakage patterns were observed in both groups. Similar to many versions of one step self etch adhesives, *Single Bond Universal* showed a spotted pattern of isolated islands of silver grains dispersed along both hybrid and adhesive layers. Deposits were also found along the interfibrillar spaces of mineralized collagen fibrils (pH=2.7)

In case of *Adper™ Single Bond 2* where it appeared as a thin continuous line at the bottom of the hybrid layer. This may explained by that the infiltration of BisGMA into acid etched dentin is less than that of HEMA. Therefore, the bottom of the hybrid layer is rich in HEMA which when mixed with water in dentin forms hydrogels manifested by silver uptake.

According to *Ito et al* <sup>(1)</sup>, self-etching single-bottle adhesives represented more nanoleakage than Scotch Bond Multipurpose (SBMP) because of the water content of self-etch adhesives compared to the water free SBMP. The difference from the results of the present study might be due to variation in specimen's treatment before nanoleakage assessment.

## CONCLUSION

The current study supports the idea of using Total-Etch adhesive system which positively affects bond degradation resistance when compared to Self-Etch one with a significant effect on microtensile bond strength to pulp chamber dentin in root canal treated teeth and a non-significant effect on nanoleakage profile of both adhesives.

The good strength properties provided by Total Etch adhesive, creates a barrier against bacterial ingress towards the periapical tissues. Besides, it increases the adaptation to pulp chamber dentin. Accordingly, this affects the durability of root canal treated teeth. Therefore, although Self Etch adhesives provides easier application, and less time consuming than Total etch, the latter remains to perform better intraorally especially after a long period of time.

## RECOMMENDATIONS

- 1- On clinical bases, more researches are recommended to evaluate the capabilities of recent techniques or up-to-date materials to overcome the negative effect of SEAS on bond strength to pulp chamber dentin.
- 2- Studies evaluating the correlation between the performance of SEAS and durability of root canal treated teeth are required.
- 3- Further studies might be beneficial if they aimed to measure other disciplines such as dentin permeability and elemental composition of adhesive layer instead of bond strength measuring.
- 4- Investigations are advised to extend the follow up interval more than three months period evaluated in the present study through Thermocycling.

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