

Influence of Adhesive Systems on Micro-leakage Pattern of Composite Resin Restorations

Eman A Abdulkadir
BDS

Department of Conservative Dentistry
College of Dentistry, University of Mosul

Ma'an M Nayif
BDS, PhD (Asst. Prof)

Department of Conservative Dentistry
College of Dentistry, University of Mosul

الخلاصة

الاهداف: ان للتسرب المجهرى اهمية في تقييم نجاح الحشوات ذات اللاصق. ان الهدف من هذه الدراسة هو تقييم كمية التسرب المجهرى الحاصل في حشوات الراتنج المركب والملصقة بثلاث انواع من نظم اللاصق. **المواد وطرائق العمل:** تم اختيار ٢٤ سن من اسنان العقل المقلوعة والخالية من التسوس والصدوع. وتم تحضير ٢٤ حفرة من الصنف الخامس على السطح الوجهي لكل سن باستخدام diamond cylinder bur No. (1543M). قسمت العينات عشوائيا الى ثلاث مجاميع اعتمادا على نوع المادة اللاصقة (لكل مجموعة منها ٨ اسنان). المجموعة الاولى: تم معالجة الحفرة بالاصق كلياالتخريش (Adper Single Bond (USA) المجموعة الثانية: تم معالجة الحفرة بالاصق ذاتي التخريش ذو المرحلة الواحدة (OptiBond All In One, Kerr, USA) المجموعة الثالثة: تم معالجة الحفرة بالاصق ذاتي التخريش ذو المرحلتين (CLEARFIL SE BOND, Kuraray, Japan) جميع الحفر تم حشوها بالراتنج المركب (Ceram X, DENTSPLY, USA) وبعد تلميع الحشوات تم حفظ الاسنان في المايكروويف لمدة ٢٤ ساعة وبدرجة حرارة ٣٧ درجة مئوية ثم تم تعريض الاسنان الى حمام حراري بين ٥ درجة مئوية و ٥٥ درجة مئوية ل ٣٠٠ دورة باستخدام حمام مائي. تم غمر الاسنان بصبغة المثلين الزرقاء لمدة ٢٤ ساعة وتم تحليل مدى اختراق الصبغة بجهاز stereomicroscope بعد شطر الاسنان طوليا وتكبير 10X. تم تحليل البيانات باستخدام اختبار -Kruskal Wallistest واختبار Mann-Whitney test عند مستوى معنوية ٥%. **النتائج:** لوحظ وجود اختلاف معنوي في التسرب المجهرى بين الحشوات ($p \leq 0.05$). الحشوات ذات اللاصق ذاتي التخريش ذو المرحلة الواحدة اظهرت قيمة تسرب عالية من الحشوات ذات اللاصق ذاتي التخريش ذو المرحلتين. ولوحظ عدم وجود اختلاف معنوي في التسرب المجهرى بين الحشوات ذات اللاصق كلي التخريش والحشوات ذات اللاصق ذاتي التخريش ($p > 0.05$). **الاستنتاجات:** التسرب المجهرى في حشوات الراتنج المركب تتأثر بنوع اللاصق. الحشوات ذات اللاصق ذاتي التخريش ذو المرحلة الواحدة اكثر عرضة للتسرب من الحشوات ذات اللاصق الكلي التخريش ومن الحشوات ذات اللاصق ذاتي التخريش ذو المرحلتين.

ABSTRACT

AIMS: Microleakage is important for assessing the success of adhesive restorative materials. The aim of this study was to evaluate the microleakage of resin composite restorations bonded with three types of adhesive systems. **MATERIALS AND METHODS:** Twenty four sound human third molars were used. Class V cavities were prepared on the buccal surface by a diamond cylinder bur No. (1543M). Samples were assigned randomly to three groups (N = 8): Group I: Cavities were treated with total etch adhesive system (Adper Single Bond 2Adhesive, 3M ESPE, USA). Group II: Cavities are treated with one step self-etch adhesive system (OptiBond All In One, Kerr, USA). Group III: Cavities were treated with two step self-etch adhesive system (CLEARFIL SE BOND, Kuraray, Japan). Cavities were restored in bulk technique with resin composite (Ceram X, DENTSPLY, USA). Restorations were polished and after storage in water for 24 hours at 37°C the samples were thermocycled between 5-50°C for 300 cycles in water baths then immersed in methylene blue solution for 24 hours. Dye leakage was assessed by examining longitudinal sections in a stereomicroscope at X10 magnification. Data were analyzed with Kruskal-Wallis test and Mann-Whitney test at 5% significant level. **RESULTS:** A significant differences in microleakage were observed among restorations ($p \leq 0.05$). One step adhesive bonded restorations demonstrate significantly higher leakage value than two step. No significant differences were observed between total etch adhesive and self-etch adhesive ($p > 0.05$). **CONCLUSIONS:** The microleakage of resin composite restoration was influenced by adhesive type. One step self-etch bonded restoration was more prone to leakage than total etch and two step bonded restoration.

Keyword: Adhesive systems, microleakage, resin composite.

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INTRODUCTION

In 1968, Buonocore *et al.* introduced the so called 'acid-etch technique' to micro mechanically interlock resin within

enamel upon impregnation into the acid produced etch pits. Today, this technique is still the most effective for reliable and durable bonding to enamel.⁽¹⁾ The integrity

and durability of the marginal seal is an important factor in the longevity of adhesive dental restorative materials, particularly for resin composites. The absence of a seal at restoration margins permits the entry of oral bacteria and fluids, which can result in postoperative sensitivity, adverse pulpal responses and recurrent caries.⁽²⁾

Technological advancements of dentin adhesives have evolved into two trends: total acid-etching techniques (5th generation dentin bonding agents) and self-etching primer technique (6th and 7th generation).⁽²⁾ Total etch adhesives are available as a three stepsystems (etchant, primer and adhesive) and two-step systems (combine the primer and adhesive materials into one component). Total etch adhesive system contains hydrophilic/ hydrophobic primers with the ability to penetrate into the demineralized enamel and dentin created after phosphoric acid etching and smear layer removal. A zone of interdiffusion is formed, and the resin dental substrate is defined as the hybrid layer. If the surface to which the adhesive will be applied consists of significantly more enamel than dentin total etch system is preferred. In comparison, self-etch adhesives generally demonstrate better adhesion to dentin than to enamel.⁽³⁾

The basic mechanism for bonding with total etch system consists of demineralization of the enamel and dentin surfaces by the acid (etchant) then adhesive applied to penetrate the microscopic spaces created by the etchant. Curing of the adhesive will result in formation of resin tags that microscopically provide a mechanical bond and seal to dentin and enamel.⁽⁴⁾ While self-etch system does not require a separate etching procedure. Thus there is no need to rinse and then dry the preparation prior to application of the adhesive. For these reasons, their popularity is increasing.⁽⁴⁾ Self-etch adhesives, composed of aqueous mixture of acidic functional monomers that are generally phosphoric acid esters. Phosphoric acid esters, with a pH relatively higher than that of phosphoric acid etching gels.⁽⁵⁾ Self-etch adhesives produce simultaneous conditioning and priming effects on dental substrates and do not remove the smear layer, but penetrate and modify it, creating a thin

hybrid layer dependent on pH, composition, and concentration of polymerizable acids and/or acidic resin monomers.⁽⁶⁾ Self-etch adhesives are available as two step systems and one step systems. In two step systems etch primer is applied without rinsing then a layer of adhesive resin is applied.⁽⁷⁾ A possible advantage of applying the etchant and primer in a premixed solution is that the demineralization of dentin occurs concurrently with primer infiltration and the risk of leaving demineralized dentin that was insufficiently infiltrated by resin is thought to be much smaller.⁽⁸⁾ One step self-etch systems is more simplified including a single application to the tooth.⁽⁷⁾ This system is extremely hydrophilic as they contain high concentrations of both ionic and hydrophilic monomers. Due to their high hydrophilicity, one step self-etch adhesives behave as semipermeable membranes, allowing fluids to pass through and seriously jeopardizing bond durability.^(9,10)

The hypothesis to be tested in this study is that no differences in the microleakage among the different adhesive systems. The objective of this study was to evaluate the microleakage in resin composite restoration following bonding with three different adhesive systems (Total etch, One and two step self-etch adhesives).

MATERIALS AND METHODS

Samples collection and mounting:

Twenty four extracted sound human third molar were stored approximately for one week in distilled water until their use. Teeth were cleaned and polished. Roots of the teeth were covered with two layers of nail varnish and apices sealed with a sticky wax. Roots were embedded in cold cure acrylic resin (Ivoclarvivadent, Liechtenstein) inside polyvinyl plastic rings up to a cemento-enamel junction.

Cavities preparation:

Class V cavities (4 mm width and 2 mm in height and depth) were prepared in the middle third of the buccal surfaces of the teeth with abut joint margins using a diamond cylindrical bur No. (1543M). In order to standardize cavity preparation the outline of the cavity was drawn on the tooth surface with the help of digital vernia to measure the dimensions Figure (1).

The handpiece of turbine was adapted to the arm of surveyor (Qualye Dental, England) in such a way that the long axis of the bur being perpendicular to the buccal

surfaces of the teeth and moves at a fixed horizontal and vertical plane within a certain space Figure (1).

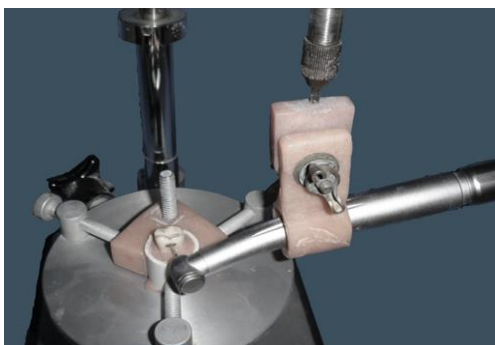


Figure (1): Sample fixed on the base of surveyor and turbine handpiece fixed on surveyor

Cavity restoration:

Samples were washed thoroughly under running water for one minute after preparation. Samples were dried and randomly allocated into three groups according to the bonding agents systems used (n=8). Three adhesive systems used in this study: A total etch system (Adper Single Bond 2 Adhesive, 3m ESPE, USA); one step self-etch system (OptiBond All In

One, kerr, USA); and two step self-etch system (CLEARFIL SE BOND, kurary, Japan) Figure (2).

Group I: Cavities were treated with total etch adhesive system.

Group II: Cavities are treated with one step self-etch adhesive system.

Group III: Cavities were treated with two step self-etch adhesive system.



Figure (2): Adhesive systems used in the study: a) Adper Single Bond 2 Adhesive; b) OptiBond All In One adhesive; c) CLEARFIL SE BOND adhesive.

The application technique, time and curing protocol of bonding systems were used in accordance to the manufacturer's instructions. Cavities were restored with nanoceramic hybrid resin composite in bulk technique (Ceram X, DENTSPLY, USA) Figure (3). Restorations were light cured for 40 seconds using LEDition light cure device at 530mw/cm (Ivoclar, vivadent,

Austeria). The intensity of light monitored with curing radiometer (Cromatest 7041-Megaphysik, Germany). Restorations were finished and polished using finishing disks of composite (KENDA DENTAL POLISHERS, Liechtenstein) from tooth surface toward restoration direction for 5 minutes. Table(1) show the composition of the materials used in the study.



Figure (3): Resin composite restorative material used in the study.

Table (1): Demonstrates compositions of the adhesives and resin composite used in this study.

Materials	Compositions
Adper Single Bond 2Adhesive	Silica, BisGMA, HEMA, dimethacrylates, ethanol and a methacrylate functional copolymer of polyacrylic and polyitaconic acids.
OptiBondAll.In .One	Monomers: Glycerol phosphate dimethacrylate (GPDM), Solvents: water, acetone, and ethanol , Fillers: including a nano_silica, Fluoride_releasing and Sodium hexafluorosilicate
CLEARFIL SE BOND	Primer: MDP, HEMA, dimethacrylate monomer, water, catalyst. Bond: MDP, HEMA, dimethacrylate monomer, microfiller, catalyst.
Ceram X , Duo	Methacrylate modified polysiloxane, ethyl-4(dimethylamino) borosilicate glass methacrylate functionalised silicon dioxide nano filler, Iron oxide , Glass filler size is 1.1-1.5µm ,nanofiller size is 10 nm,nanoparticle size is 2.3 nm, Filler content 76 Wt.% , 57 Vol.%.

Thermocycling:

Samples were stored in water at 37°C for 24 hours then subjected to a thermal cycling of 300 cycles between 5°C (±2°C) and 55°C (±2°C) using water baths with 30 seconds in each bath . Samples were covered with two coats of nail varnish leaving a 1 mm window around the cavity margins and immersed in 2% methylene blue solution (HiMedia lab. Mumbia, india) at 37°C temperature for 24 hours. Dye was carefully rinsed under tap water.

Sectioning and scoring:

Samples were fixed to plastic ring fixture which is adapted on the surveyor to ensure uniform sectioning of the teeth. The samples were bisected longitudinally in a buccolingual direction with a low speed diamond disc (HoRico, Italy) under water coolant. The sectioned teeth were evaluated under Stereomicroscope (Motic, Italy) at x10 magnification. The degree of microleakage using dye penetration was scored in a blinded manner as show in the Table (2).

Table (2): The Microleakage scores of dye penetration

Scores	Content
0	No leakage
1	Leakage through the cavity margin within the enamel only.
2	Leakage within the enamel and dentin not reaching axial cavitywall.
3	Leakage reaching axial wall.

Statistical analysis:

A statistical analysis was performed using Statistical Package for the Social Sciences (SPSS 13.0, SPSS Inc. USA).

1. Descriptive statistics including mean, standard deviation (SD) value.
2. Kruskal-Wallis test used to test the difference in microleakage among adhesive types.
3. Mann-Whitney test used to test the

difference in microleakage between each two adhesives.

All the statistical tests were computed at 5% significant level.

RESULTS

Mean and standard deviation (SD) of microleakage for restorations are shown in Table (3).

Table (3): Microleakage of bur prepared restoration bonded with three adhesives.

Type of adhesives	Total etch adhesive	One step self-etch adhesive	Two step self-etch adhesive
No.	8	8	8
Mean	0.25	0.5	0.031
SD	0.377	0.267	0.231

Kruskal-Wallis test show a significant difference among the groups ($p \leq 0.05$) Table (4). Mann Whitney test comparison of microleakage between adhesives show that there was significant difference in micro-

leakage between one step self-etch adhesive and two step self-etch adhesive only ($p \leq 0.05$) while no differences between other groups ($p > 0.05$) Table (5).

Table (4): Result of Kruskal-Wallis test leakage comparison among different adhesives

Group	Kruskal-Wallis Test value	df	p-value
cavities bonded with different adhesives	6.167	2	0.046*

*significant difference existed at $P \leq 0.05$.

Table (5): Result of Mann-Whitney test leakage comparison between different adhesives

Groups comparison	Mann-Whitney test value	p-Value
Group I X Group II	18.000	0.105
Group I X Group III	27.000	0.519
Group II X Group III	11.000	0.013*

*significant difference existed at $p \leq 0.05$.

The restorations bonded with one step show higher microleakage than restorations bonded with two step. While those bonded with total etch adhesive demon-

strate no significant differences in microleakage than restorations bonded with self-etch adhesives regardless of number of step application Figure (4).

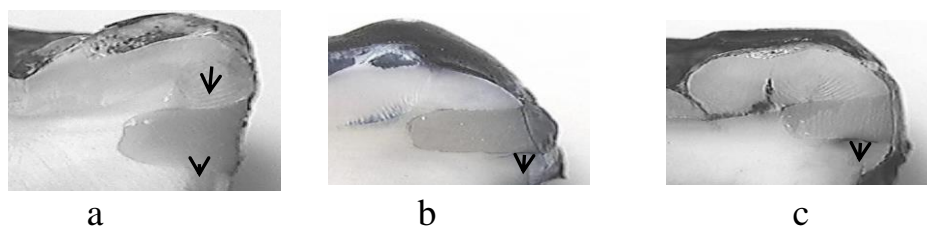


Figure (4): Representative images of microleakage scores for all types of adhesives used in the study. a) Score 0 of cavity restored with two step self-etch adhesive (CLEARFIL SE BOND). b) Score 1 of cavity restored with total etch adhesive (Adper Single Bond 2). c) Score 2 of cavity restored with one step self-etch adhesive (Optibond All In one).

DISCUSSION

Cavities prepared with a high-speed drill have a layer of debris (smear layer).⁽⁵⁾ This smear layer can be removed or modified/ infiltrated to achieve micromechanical retention of composite restorative ma-

terial to the dental substrate, depending on the adhesive protocol used and the bonding mechanism.⁽⁵⁾

Microleakage is one of the possible drawbacks of composite restoration due to polymerization shrinkage of the composite

resin result in stresses on the bond between composite and tooth structure result in gap formation and leakage.⁽¹¹⁾ The clinical symptoms associated with the occurrence of microleakage are breakdown and discoloration of margins, secondary caries, increase in postoperative sensitivity, and pulp pathology. Although many techniques have been used to demonstrate microleakage, such as chemical and radioactive tracers, electrochemical investigations; scanning electron microscopy, however the most common of all is dye penetration which is applied in our study because of easy use and reliable results imply the obvious improvement in the bonding process of current adhesives systems.⁽¹²⁾

The results of our study showed that most of the scores were within enamel or show no penetration; this low level of microleakage in all types of adhesives tested in this study may be due to a significant factor which is the presence of fillers in adhesives used in this study. Reduction in microleakage score has been reported by previous studies when using filled adhesives.^(13, 14) Because the adhesive layer obtained with these types of adhesives was thicker, with better ability of the interfaces to maintain adhesion during the critical early stages of polymerization so improving the resistance to dimensional changes.⁽¹⁵⁾ In addition this resin coat helps to reduce the amount of hydrophilic and acidic resin component in the bonded dentin interface, rendering these adhesives less permeable.^(13, 14)

On the other hand, Owens and Johnson⁽¹⁶⁾ found that none of the dentin bonding agents (SINGLE BOND total etch adhesive, ADPER PROMPT two step self-etch adhesive, CLEARFIL S3 one step self-etch adhesive and G-BOND one step self-etch adhesive) can completely prevent the microleakage at the restoration-tooth interface. Moezizadeh and Moayedi⁽¹⁷⁾ concluded that none of the latest generations of adhesive resin bonding systems (one and two step self-etch adhesive systems) can create gap free margins in class V composite restorations.

Although our study shows low possibility of the microleakage meanwhile when comparing the microleakage pattern

among the adhesive systems the results show a lowest microleakage value associated with two step (CLEARFIL SE BOND). The probable explanation may be related to the fact that although Clearfil SE Bond is a mild two step self-etch adhesive with a pH very close to 2⁽¹⁾ mild self-etch adhesives that produce thin hybrid layer with smear plugs occlude the orifice of the dentinal tubules which are partially infiltrated by resin. Such observation was reported by various studies.^(5, 18) Generally in self-etch adhesives collagen fibrils within the hybrid layer are not completely deprived of hydroxyapatite; it was hypothesized that the residual hydroxyapatite may serve as a receptor for additional intermolecular and chemical interaction with monomers of the mild self-etch adhesive,⁽¹⁾ therefore improving bonding effectiveness due to a combined micromechanical and chemical interaction with tooth substrate.^(1, 19) The chemical component may be able to compensate for the reduced bonding effectiveness from decreased micromechanical interlocking as the chemical interaction may result in bonds that better resist hydrolytic degradation (microleakage).^(20, 1)

The results show more leakage in one step self-etch system (Optibond All In One adhesive) than two step self-etch adhesive system (CLEARFIL SE BOND). Such finding was supported by Nayif *et al* when compared the nanoleakage of same self-etch adhesive systems with similar results.⁽¹⁸⁾ Authors observed multiple droplets within the adhesive and hybrid layer of one step system during SEM examination. Upon polymerization, the droplets become entrapped within the adhesive, potentially jeopardizing bond durability.⁽²¹⁾ However, increased hydrophilicity and water permeability of one step self-etch adhesive system due to hydrophilic monomer included act as semi permeable membrane. There are water channels that originate from the surface of the hybrid layer and extend through the adhesive layer to reach the adhesive composite interface. These water channels have been given the term water trees by Tay and Pashley in 2004.^(10, 22)

Results show no significant difference in leakage between total etch (Adper Sin-

gle Bond 2) and one step self-etch (Opti-bond All In) adhesives the probable explanations were associated with both adhesives permeability were more extensive due to the presence of higher concentrations of hydrophilic monomers due to mixing of bonding agent with primer in total etch adhesive and with acidic monomer and primer in one step self-etch adhesive lead to incomplete polymerizations. As partially cured adhesives were more permeable to fluid movement⁽²³⁾ they may expedite water sorption and compromise the long term integrity of the adhesive-composite bond. Conversely, dentin bonding systems that utilize the separated non solvated hydrophobic bonding agents as in two step self-etch adhesive system showed higher extents of polymerization and were correlated with less permeability to water.⁽²⁴⁾ Leakage of total etch adhesive may be associated with excessively air-dried dentin.^(1, 21) The major reason for this is presumed to be the effect of collapsing the collagen network at the bonding interface. The collapsed collagen prevents complete infiltration of the resin monomers into the demineralized dentin, leading to gaps and voids within the adhesive interface. Also several studies have reported the discrepancy between etching depth and adhesive penetration for etch & rinse adhesives.⁽²³⁾

On the other hand no significant difference between total etch adhesive and two step self-etch adhesive. These adhesive systems contain HEMA which is a hydrophilic monomer that penetrates into the collagen network. HEMA molecules are usually dissolved in different solutions with acetone, alcohol and/or water which work as chasers. These chasers compete with water present at the dentin surface by promoting a union of the water molecules and displacing water when compressed air is applied, permitting the penetration by the monomer.⁽²⁵⁾ The removal of water from the collagen fibrils may stabilize the structure by increasing the amount of interaction of weak forces between adjacent collagen molecules. Water removal may also permit additional hydrogen bonds to form between collagen molecules that were previously bonded to water molecules.⁽²⁶⁾ The results of our study was in

agreement with Deliperiet *al* that reported one-step self-etching adhesives exhibited a significant level of microleakage compared to total etch and two-step self-etching adhesive systems.⁽²⁷⁾ Also when considering the adhesives behavior regarding bond strength Sensi et al reported that one-step self-etching adhesives exhibited a lower bonding force to dentine compared to total-etch and two step self-etching adhesive systems.⁽²⁸⁾

CONCLUSIONS

Microleakage of class V resin composite restorations was influenced by the type of adhesive system. One step self-etch adhesive was more prone to microleakage than two step self-etch and total etch adhesive systems. Two step self-etch adhesive has lowest leakage when compared with total etch and one step self-etch adhesives.

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