A Microleakage Evaluation of Two Generations of Bonding Agents Using a Novel Fluid Filtration Model

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ABSTRACT

Aim: The aim of the study was to evaluate the microleakage between two different generations of bonding agents using fluid filtration model.

Materials and methods: In total, 60 human extracted mandibular molars were used in the study. The samples were divided into three groups, group I—Adper Scotchbond primer and adhesive, group II—Single Bond Universal adhesive, and group III—control group. All the samples were restored and stored in distilled water for 24 hours followed by thermocycling in water bath for 1,000 cycles between 5 ± 2°C and 55 ± 2°C, for dwell time of 30 seconds and transfer time of 10 seconds. The samples were then tested for leakage under fluid filtration model.

Results: There was a statistically significant difference between the leakage in between the two groups. The difference was found to be statistically significant (p < 0.05). Single bond group showed better adhesion and less leakage compared to the Adper Scotchbond primer and adhesive.

Conclusion: Single Bond Universal adhesive (3M) showed the least amount of microleakage when compared to Adper Single Bond 2 and Adper Scotchbond primer and adhesive. Fluid filtration model was successfully constructed and worked efficiently and can be considered as one of the ideal methods for evaluation of microleakage.

Keywords: Adper Scotchbond primer and adhesive, Adper Single Bond, Microleakage, Single Bond Universal.

INTRODUCTION

A microscopic space always exists between the tooth restoration interface which results into microleakage responsible for breakdown at the marginal area of restoration and development of pulpal pathology that jeopardize the clinical longevity of restoration. The percolation of oral fluid and bacteria along the restoration margins may be responsible for pulp irritation, recurrent caries, hypersensitivity, and failure of restoration.

The evaluation of dental composite resin restorations over the past 20 years has centered primarily on issues of biomechanics,1–4 as these parameters are most immediate in determining short-term clinical restoration success. With advanced technologies, dental adhesives have evolved from no-etch to total-etch (fourth- and fifth-generation) and finally to self-etch (sixth-, seventh-, and eighth-generation) systems.5

However in the midst, other important issues concerning the biocompatibility of composite resins—though at times briefly acknowledged—have gone otherwise largely unaddressed.5,6 As can be expected of any synthetic material placed within a biological system, composite restorations are not completely inert.7–9 They interact dynamically with the host, forces, and conditions present in the oral environment.8,10 By the early 1990s, investigations into the durability of resin-based restorative materials in vivo were reporting material loss at a faster rate than could attribute purely to mechanical forces.11,12 Others had found material discoloration at the tooth–composite marginal interface,13,14 a clear marker of degradation at the adhesive–dentin interface over time.15,16 Release of toxic products from composite restorations such as methacrylic acid and formaldehyde,17,18 and other leachable by-products were also being detected.17,18

These early findings attested to the fact that resin-based dental materials are indeed subject to chemical degradation in the oral cavity.2,19 Polymers of resin-based composites are bound by unprotected ester linkages inherently prone to cleavage by water.17,19

Since the composition of human saliva is nearly 99% water, these materials are highly susceptible to hydrolytic degradation in vivo. More importantly, saliva also contains esterase-like enzyme activities capable of accelerating the hydrolytic process and, thus, the rate of chemical degradation.19,20–23

Very little research has focused on the impact of biodegradation along the tooth–resin interface. Of particular concern are proximal and cervical restorations where the cavosurface margin is formed against wet dentinal substrate.24,25 Under optimal bonding conditions, resin penetration of cut tubules and associated branches results in the formation of resin tags that provide mechanical interlocks, fusing the hybrid layer to the un-etched dentinal substrate below.25

Over the past years, microleakage studies determining the leakage of bonding agents have been published; however,
the methods used are varied and most of them present an nonstandardized method of microleakage evaluation. There are various methods to measure microleakage including chemical, bacterial and radioactive tracers, dye penetration, dye extraction, electrochemical, neutron activation, and micro-computed tomography; however, they present inherent drawbacks. The fluid filtration method has been widely used to measure microleakage. This method has several advantages over the commonly used methods: the samples are not destroyed, permits the evaluation of microleakage over time, operators bias, and most importantly the results are accurate since very small volume is recorded. Hence, this study is aimed at evaluating microleakage of two different generations of bonding using the fluid filtration method.

**Materials and Methods**

In total, 60 human extracted mandibular molars were used in the study. The teeth were cleaned by ultrasonic scaler and were washed thoroughly before being stored in normal saline solution at room temperature. Following which the teeth were mounted on acrylic block and standard class I cavity preparation of approximately 1.5 mm depth and 3 mm width were prepared with airotor and diamond bur no. 330 (SS White FG). The teeth were randomly divided into three groups (n = 20) according to the bonding agent used as follows:

- **Group I: Adper Scotchbond primer and adhesive**
- **Group II: Single Bond Universal**
- **Group III: Control group**

Positive control—specimens restored without bonding agents. Negative control—specimen restored with Adper Single Bond 2 (total-etch).

All the prepared cavities were rinsed with water from a syringe, for 30 seconds and dried with absorbent paper for 15 seconds, before the restoration of the cavities.

In group I, the cavities were etched using etchant (37% phosphoric acid) for 15 seconds followed by rinsing for 15 seconds and air-dry for 5 seconds. Application of primer was done followed by gentle dry for 5 seconds. Adhesive application was done and was light cured for 10 seconds.

In group II, Single Bond Universal adhesive was applied in the cavities agitated using applicator tip followed by light curing for 20 seconds.

In the control group, in the positive control group, the teeth were not restored with bonding agent, and in the negative control group, the samples were etched with etchant (37% phosphoric acid), rinsed with water for 15 seconds, air-dried for 5 seconds, followed by application of Adper Single Bond 2, and light cured for 20 seconds.

All the samples were then filled with Filtex Z350 XT nanocomposite in incremental layering technique, and each increment was cured for 40 seconds. Using a diamond disk, dentin disks were prepared by sectioning the tooth, each measuring 6 mm in diameter and 2 mm in thickness.

The samples were then stored in distilled water for 24 hours followed by thermocycling in water bath for 1,000 cycles between 5 ± 2°C and 55 ± 2°C, for dwell time of 30 seconds and transfer time of 10 seconds.

The samples were then placed in the fluid filtration model to evaluate microleakage (Figs 1 and 2). The statistical analysis was done using independent student t test to do intergroup comparison. The significant statistical difference was kept as p < 0.05.

**Results**

The intergroup distribution of microleakage was presented in Table 1. Self-etch adhesives showed least leakage compared to the total-etch adhesives (Fig. 3).

The intergroup statistical comparison of group I with control was presented in Table 2. The comparison of the group I with the positive controls showed that there was a decrease in the microleakage of group I compared to the positive control, which was found to be statistically significant (p < 0.005). Group I also showed less leakage compared to the negative controls, which was statistically significant (p < 0.005).

The intergroup statistical comparison of group II with control was presented in Table 3. The findings of group II were significantly lower compared to the positive controls, i.e., the controls showed more amount of microleakage, and this was found to be statistically significant (p < 0.005). Also, the group II observations were less than the negative control which was also found to be statistically significant (p < 0.005).

The intergroup statistical comparison of group I and group II with the positive and negative controls was presented in Table 4. The mean of the positive controls was higher than that of the group I and group II, and this difference was statistically significant. The mean of the negative control was lower than that of the mean score of group I and group II, and this difference was found to be statistically significant. When all the groups were compared using analysis of variance, the scores of the positive control were significantly higher than those of the two intervention groups as well as the negative controls.

**Discussion**

The success of any restorative therapy is to provide an absolute seal in order to prevent the further deterioration of the existing dental disease and prevent it from reaching the pulp. Microleakage of dental materials, especially composites, is the most important factor that affects the outcome of the treatment provided. It is a well-known and a well-established fact that the amount of microleakage and the failure rate are directly proportional to each other. It thus reduces the shelf life of the restoration, and hence, understanding the extent of its existence among the present materials being used on patients currently is essential for a more improved and long-term treatment.

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**Fig. 1:** Specimen testing using fluid filtration model
The dental literature supports the use of tooth bonding adhesives when used according to the manufacturer’s instruction unique for each product, as being effective in enhancing retention of restorations, minimizing microleakage, and reducing sensitivity. Microleakage at tooth dental restoration interface is the main factor affecting longevity, in which restorative margins could be colored or it might lead to secondary caries, increased sensitivity in the restored tooth, and also pulp.

In the present study, we used 60 extracted human mandibular molar teeth that were randomly divided into two groups for ease of comparison between the two types of restorative materials, i.e., group I: Adper Scotchbond primer and adhesive and group II: Single Bond Universal.

### Table 1: Intergroup distribution of microleakage

|                 | Group I  | Group II | Positive control | Negative control |
|-----------------|----------|----------|------------------|------------------|
| Microleakage    | Mean     | SD       | Mean             | SD               |
| (μL/minute/cm H₂O) | 3.265 ± 0.771 | 1.360 ± 0.305 | 5.642 ± 0.30672  | 1.079 ± 0.553723758 |

SD, standard deviation

### Table 2: The intergroup statistical comparison of group I with control

| Group                          | t value | p value  | Significance |
|--------------------------------|---------|----------|--------------|
| Group I with positive controls | 9.163   | <0.005   | Significant  |
| Group I with negative controls | 7.92    | <0.005   | Significant  |

### Table 3: The intergroup statistical comparison of group II with control

| Group                          | t value   | p value  | Significance |
|--------------------------------|-----------|----------|--------------|
| Group II with positive controls | 33.73     | <0.005   | Significant  |
| Group II with negative controls | 0.043415  | <0.005   | Significant  |

### Table 4: Intergroup statistical comparison of group I and group II with the positive and negative controls

| Group                          | F value   | p value  | Significance |
|--------------------------------|-----------|----------|--------------|
| Group I                        | 127.98    | <0.0001  | Significant  |
| Group II                       | 495.02    | <0.0001  | Significant  |
| All the groups                 | 168.55    | <0.0001  | Significant  |

The present study used two materials (i.e., Adper Scotchbond primer and adhesive and Single Bond Universal adhesive) that are routinely used in clinical settings and are found to be equally successful on the clinical evaluation for the long run by practitioners. Although enamel adhesion is a predictable and established entity, an adequate bond to dentin is more difficult to achieve. To overcome this challenge, technological advancements of dentin adhesives have, at this time, evolved into two trends: total acid-etching techniques.
The concept of total-etch adhesion for enamel and dentin is well accepted. Although new techniques with self-etching adhesives have been introduced, there is a need for more clinical trials before making a complete switch to these systems. Currently, the adhesive considered as gold standard are total-etch systems. In this study, a total-etch adhesive, i.e., Adper Single Bond 2 is used as a positive control, since studies have reported a superior adhesion of total-etch adhesive over self-etch. Hence, in order to compare multiple-step bonding adhesive over single step, total-etch adhesive is used as a control.

In this study, self-etch adhesive systems had the best performance. The utilization of these adhesives on dentin does not alter the microleakage values. In another study, the authors reported that there was no difference between the Scotchbond Multipurpose and the Adper Scotchbond, which is contrast to the findings of the present study. Even though they report of microleakage while using the two materials, there was no significant difference unlike the present study findings.

The current procedure was carried out using the fluid filtration technique. There are various studies that have been carried out similarly using the same technique, but a direct comparison cannot be made since there is a change in the methodology design, and also the exact location and amount of microleakage cannot be always accurately determined clinically. We used this technique because it is not destructive in nature. It also allows the samples to be investigated over a period of time regarding the leakage. It provides some level of qualitative and quantitative analysis since the photographs can be used as a record for further future evaluation.

Pashley et al. found no correlation between the bonds to enamel of these adhesive systems and their pH (weak, moderate or strong). So far, the literature does not provide a straightforward answer whether mild self-etch adhesives bonded to enamel can withstand the mechanical and chemical challenges of the oral cavity. In addition, Munoz et al., found using universal bonding with the self-etch approach that nanoleakage level was similar to Clearfil SE Bond, which is the gold standard of self-etch adhesives. They concentrated on instant characteristics of universal adhesives to dentin, including bond strength, nanoleakage, and conversion degree of Clearfil SE Bond, Adper Single Bond, Peak Universal Adhesive, Scotchbond Universal, and All-Bond Universal. They suggested that Scotchbond Universal showed the lowest microleakage between the seventh-generation universal adhesives.

However, other techniques like the dye penetration method according to Taylor and Lynch are destructive method. It also fails to demonstrate the nature and pattern of leakage. Also the technique has other disadvantages like size of the dye, the type of dye, and also its cross-reaction of the dentin. The other techniques such as three-dimensional micro-computed tomography, bacterial microleakage, and use of metal based like silver nitrate have not been that successful since long-term assessment of the specimens is not possible by using these techniques.

Also a study by Rueggeberg proved that the time after extraction does not have any effect on the bonding of the material to the teeth. It may vary from a few minutes to a few years also. Hence, we used the freshly extracted molars for the study purpose to avoid biological bias in the study.

The evaluation of microleakage is performed by different methods, such as air pressure, bacterial assessment, radioisotope studies, scanning electron microscopy, chemical identifiers, electrochemical studies, and measurement of dye penetration. Some studies have reported that different methods of microleakage evaluation do not differ in the final results.

The result of the current study are consistent with study carried out by Pradelle et al., the study stated that etch and rinse and self-etch systems do not differ in dentin margin microleakage.

In fluid filtration method of this study, the microleakage of self-etch adhesive was more significantly than total-etch adhesive. In contrast to our finding, it was indicated that Xeno III self-etch adhesive had less leakage than Prime and Bond NT total-etch adhesive using fluid filtration. This difference may be referred to the different self-etch and total-etch adhesives used in these studies and the laboratory assembly. Incomplete bonded restorations may produce changes in fluid flow and microleakage. In the present study, more microleakage was observed in the self-etch adhesive than in the total-etch adhesive that is in agreement with those of Loguercio et al.’s study.

One explanation for microleakage is the degree of conversion that is not occurred completely in self-etch adhesives due to the existence of water and more hydrophilic monomers in their contents.

Previous studies have evaluated the correlations among the three methods of microleakage assessment, fluid filtration method was defined as the control group and the correlations of the two other methods were assessed by it. Therefore, the study was not indicated any significant correlations between the qualitative method (dye penetration) and quantitative methods (fluid filtration and dye extraction) like the past paper, but there was a positive correlation between them.

This finding may be due the different test setup and separate samples prepared for each microleakage assessment method. Additionally, it is advisable to use fluid filtration methods instead of dye extraction in order to get reliable quantitative results. In the future, it would be advisable to assess the sealing ability of different adhesives with various methods and handling techniques for a long time and even in a in situ study.

Conclusion
Within the limitations of this in vitro study, it can be concluded that the single-step adhesive provided definite edge over multistep approach of adhesives. However, when comparing with the results of previous literature, it does not provide a straightforward answer to whether self-etch or etch and rinse adhesive; hence, more studies need to be emphasized in future.

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