

# Comparative Evaluation of Microleakage between Three Adhesive Agents

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## Abstract

**Introduction:** The aim of this study was to evaluate and compare the microleakage of three adhesives in class V composite restorations. **Methods:** 30 extracted third molars were divided into three groups based on the adhesive types. Two class V cavities were prepared on each tooth. An individual adhesive was used for bonding in each group and cavities were restored with resin composites. The specimens in each group were aged by thermal cycling and submerged in silver nitrate solution. Microleakage was assessed with a stereo microscope. Statistical analysis was performed to compare the extent of microleakage between the groups. **Results:** The microleakage of the universal adhesive G-Premio Bond was significantly lower than that of the other two bonding agents. No significant difference was found between G-Premio Bond and Iperbond Ultra ( $p > 0.99$ ), although the 6<sup>th</sup>-generation adhesive Quickbond had the highest microleakage compared to the other two. **Conclusion:** Microleakage was influenced by the type of adhesive. Lower levels of microleakage in the universal adhesives led to longevity and durability of the restoration.

**Keywords:** Microleakage, Adhesive, Composite Restoration, Thermal Cycling.

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## Introduction

The manufacture of resin composites is among the greatest achievements in dentistry of the last century, as the use of adhesives has revolutionized restorative dentistry (1, 2). One of the main challenges for dentists is the mechanical retention of the restorative materials. Adhesives have made tooth preparation in restorative dentistry less invasive and composite restoration more convenient (3,4). Adhesive systems are used to bond the resin composite to the enamel and dentin of the tooth. The use of adhesives for tooth restoration began in 1955 (5) with Buonocore observing that phosphoric acid can be used to prepare the tooth surface before applying the resin. He came to the conclusion that etching the enamel surface with phosphoric acid increases the life of restorative materials adhering to the tooth (6). The success of composite restoration depends on the ability of adhesive to create a good bond between the tooth and the restoration (7, 8). The development of adhesives has resulted in the reduction of microleakage in composite resin restoration, and thus to the longevity and optimization of the restoration. Preventing recurrent caries and improving adhesion are further examples of the influence of adhesive developments (9).

Nonetheless, the shortcomings of resin composites and adhesives still limit their use. While newly developed adhesives have unique features, there are some drawbacks. The shrinkage that occurs during resin composite polymerization leads to the formation of a gap

between the tooth and the restoration (10). Insufficient marginal adaptation between the adhesive and the tooth after the composite shrinkage can lead to the penetration of liquids, bacteria, molecules and ions into the space between the restoration and the wall or restoration cavity (11).

Ultimately, such a microleakage leads to secondary caries, color change, inflammation of the pulp tissue and consequently to a reduction in the life of the restoration, which in turn impairs proper adhesion to enamel and dentin (12, 13). Adhesives have been evolved and improved over the years to address shortcomings (7, 8). There are several generations of adhesives available on the market that can be used in different modes such as self-etch or total-etch. The 4<sup>th</sup>- generation adhesives, known as the three-step etch-and-rinse system, are considered the gold standard, especially for long-term performance (14–16). However, it has some disadvantages, such as the complexity of the application and long chairside time, which leads to problems with tooth isolation, especially with posterior teeth (14). These complications lead to the conclusion that adhesives with simpler technique and acceptable performance are required for a durable restoration (17).

The 6<sup>th</sup>-generation bonding systems introduced in the late 1990s and early 2000s, also known as the “self-etching primers”, have been a dramatic leap in technology (7). This generation of adhesives are two-step systems that contain two bottles, containing the acidic primer and the adhesive. Although such adhesives have shown acceptable performance, they have some limitations such as a prolonged bonding process and multiple steps (18). The 7<sup>th</sup>-generation, however, are self-etch adhesives with all components in a single bottle. Despite their ease of use, their effectiveness has been questionable (15).

The latest generation of bonding agents has been universal adhesives whose components are contained in a single bottle similar to the 7<sup>th</sup>-generation and,

depending on the situation, can be applied in self-etch or etch-and-rinse mode (19, 20). They make restorative dentistry easier, as they simplify the procedure, and shorten the chairside time (21).

As already mentioned, microleakage is one of the main causes of restoration failure, however, there has not been enough research on evaluating microleakage in dental adhesives. In addition, there has been a debate among researchers about the universal adhesives’ functionality, especially in the self-etch mode, to prevent microleakage and improve bonding effectiveness.

The aim of this study was therefore to evaluate and compare the microleakage of the universal adhesives G-Premio Bond and Iperbond Ultra and the 6<sup>th</sup>-generation adhesive Quickbond. The null hypothesis was that the microleakage of the three adhesives did not differ significantly.

## Materials and Methods

Given  $\alpha = 0.05$ ,  $\beta = 0.2$ , and 80% of power in this *in vitro* study, the minimum number of samples for each group was set to 10 (22). A number of 30 caries-free human third molars that had been extracted due to periodontal problems were collected. The teeth were rinsed and disinfected with Chloramine-T 0.5% for seven days and then stored in distilled water until use. The study protocol was approved by the Ethics Committee of the Tehran University of Medical Sciences (Ref: IR.TUMS.DENTISTRY.REC.1396.2106).

A standard class V cavity with 2 mm occlusogingival height (1 mm at the enamel-cement junction and 1 mm below), 3 mm mesiodistal width and 1.5 mm depth was prepared on the buccolingual surface of each tooth. The teeth were randomly divided into three groups (n=10). Each adhesive was applied to the teeth according to the manufacturer’s instruction. The materials used in the experiment and their composition are listed in Table I.

**Table I.** Manufacturer and composition of the materials used in this study.

Components	Manufacturer	Type	Material
4-methacryloyloxyethyl trimellitate anhydrate, 10-methacryloyloxydecyl dihydrogen thiophosphate, methacrylate adic ester, distilled water, acetone, photo initiators, silica fine powder	GC corporation, Tokyo, Japan	Adhesive	G-Premio Bond
Polymerization accelerators, 4-Methacryloyloxyethyltrimellitic acid, Dendritic Polymer, Aqua, Acetone, Urethandimethacrylate, Urethane modified dimethacrylate, Triethylglycoldimethacrylate, 2-Hydroxyethylmethacrylate, Multifunctional dimethacrylates, Ammonium quaternary alkylbentonite, Photoinitiators	Itena clinical products, Paris, France	Adhesive	Quickbond
Ethanol, 2-Propenoic acid, 2-methyl-, 7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxo-5,12-diazahexadecane-1,16-diyl ester, 2-Hydroxyethylmethacrylate, Silicone dioxide	Itena clinical products, Paris, France	Adhesive	Iperbond Ultra
Barium aluminosilicate, Fumed silica, Bis-GMA, Triethylglycoldimethacrylate,	Itena clinical products, Paris, France	Resin composite	Reflectys

In the first group, G-Premio Bond (GC Corporation, Tokyo, Japan) was gently applied to the surface of the cavity and dried for 5 seconds after 10 seconds with maximum pressure. The adhesive was cured with an LED light-curing device (BlueLex GT1-200, Monitex, at 800 mW/cm<sup>2</sup> light intensity) for 10 seconds and the cavity was restored with a composite.

In the second group, Quickbond (Itena Clinical, Paris, France) was used. First, its primer was applied to the cavity and dried with a heavy blast of air for 5 seconds. Next, two successive layers of adhesive were applied to the surface of the entire cavity and dried. Finally, it was cured with the LED light-curing device for 20 seconds and the cavity was restored with a composite.

In the third group, Iperbond Ultra (Itena Clinical, Paris, France) was used. A drop of adhesive was gently applied to the cavity surface with a disposable brush for 20 seconds. It was then dried for 5 seconds using oil-free compressed air. The adhesive was cured for 20 seconds with the LED light-curing device and the cavity was restored with the Reflectys composite (Itena Clinical, Paris, France).

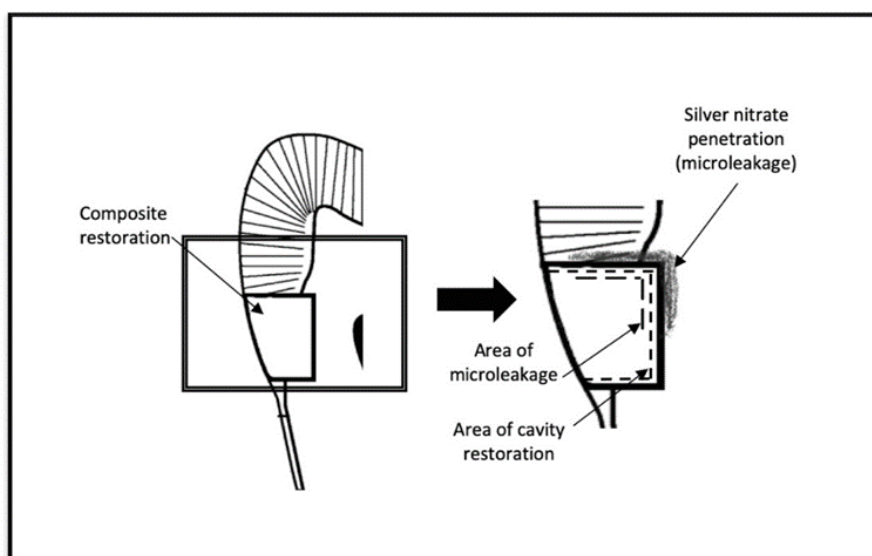
The cavities in each group were incrementally filled with the Reflectys composite and cured for 40 seconds. The restorations were then polished with a multipolish (resin-based acrylic polishing bur). The specimens were stored in deionized water for 24 hours. After incubation, fine diamond burs and Sof-Lex polishing discs (Sof-Lex, 3M ESPE, USA) was used for final finishing and polishing.

The end of each tooth root was sealed by composite resin and light cured. The specimens in each group were placed in dough in two rows of 5 numbers in the shape of a rectangular cube. The entire surface of the teeth was covered with nail polish, with the exception of the restoration and 1 mm of the surrounding, to prevent silver nitrate from penetrating the other parts of the tooth.

Next, the specimens were aged by thermal cycling between 5 and 55°C for 3000 cycles with 20 seconds dwell time and 5 seconds transfer time. We used the dye penetration technique to assess microleakage. The specimens were immersed in 1 mole of silver nitrate solution for six hours and then rinsed with water. The specimens were then immersed in a radiographic developer for 12 hours and placed under a fluorescent light for an additional 6 hours.

The teeth were embedded in clear, cold-curing methyl methacrylate resin (Heraeus Kulzer, Hanau, Germany) and a buccolingual section was made on each tooth with a diamond saw (Isomet Low-Speed Saw, Buehler, Lake Bluff, IL, USA). Microleakage was measured quantitatively on each side for to the total length of the interface between the tooth and the composite (23). The measurements were carried out with a stereo microscope (Olympus SZX9, Tokyo, Japan) and the total microleakage percentage was calculated as the sum of the measured length of penetrated silver nitrate divided by the sum of the measured length of the entire restoration (23) (Figure 1).

**Figure 1.** Schematic view of microleakage assessment.

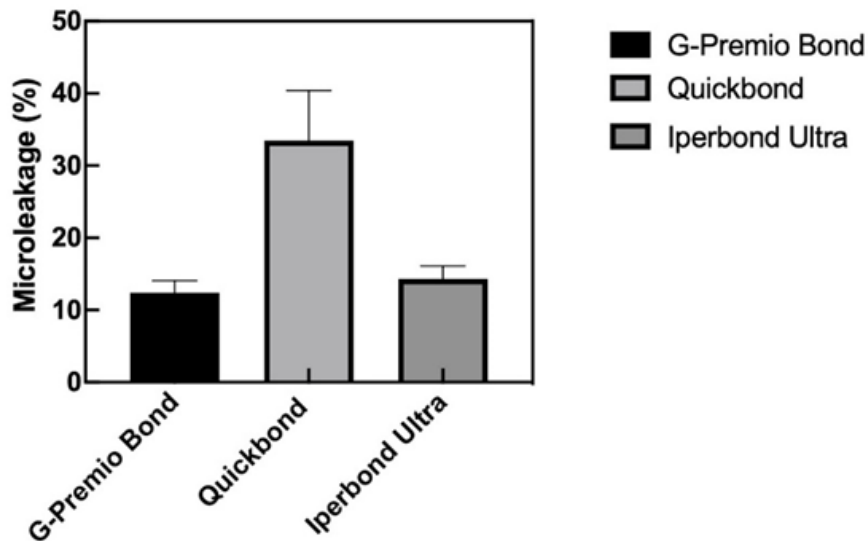


Statistical analysis was performed to compare the microleakages between groups ( $P < 0.05$ ).

### Results

After the specimens were examined with a stereo microscope, the data were collected and the microleakage in each specimen was determined. The microleakages in

the three adhesives varied from 12.41% in G-Premio Bond to 14.3% in Iperbond Ultra, and 33.47% in Quickbond (Figure 2). As given in Table II, there were no significant differences between the microleakage in G-Premio Bond and that in Iperbond Ultra ( $P > 0.99$ ). However, the microleakage in Quickbond was higher than two other adhesives.



**Figure 2.** Comparison of the microleakage between the three adhesives.

The analysis showed that the P-value of microleakage between the groups was 0.008, while that proportional to microleakage was 0.028.

**Table II.** Comparison of mean and confidence interval between the groups

	Upper CI (95%)	Lower (95%)	CI	Adjusted mean	Standard deviation
G-Premio Bond	16.16	8.67		12.41(a)	1.65
Quickbond	48.88	18.05		33.47(b)	6.91
Iperbond Ultra	18.30	10.31		14.30(a)	1.79

\*Mean and confidence intervals are displayed in percentage (%). Similar lowercase letters indicate no significant differences between the groups ( $P < 0.05$ ).

## Discussion

In this study, the microleakage of three adhesives was assessed after thermal cycling. Based on the results, the null hypothesis that the microleakage did not differ significantly between the adhesives was rejected. It was found that the universal adhesive G-Premio Bond had the lowest microleakage compared to the other two adhesives. This is particularly important in clinical dentistry, as microleakage is one of the main complications in composite resin restoration. Liquids, bacteria, molecules and ions that penetrate the space between the restoration and the tooth cause microleakage, and are the determining factor for restorative materials. The chemical, thermal, and mechanical stresses can lead to microleakage (24, 25). It can be the cause of postoperative tooth sensitivity and

secondary caries, which can lead to pulpal inflammation and restoration failure (7, 11).

The science and technology of materials in dentistry have advanced over the past few decades. Since there are so many options for dental adhesives, there has always been a debate among researchers as to which adhesives are more efficient. Based on the previous studies, etch-and-rinse adhesives are still the gold standard in terms of bond strength and microleakage. However, they are not as popular because of their complex approach (14, 16). The 6<sup>th</sup>-generation adhesives, which are two-step self-etch adhesives, were the first adhesives introduced that did not require acid etching prior to adhesion (18).

The 7<sup>th</sup>-generation adhesive systems, also known as single-bottle self-etching adhesives, make bonding

easier, as all the ingredients required for bonding are placed and delivered in a single bottle (15). Universal adhesives, known as the 8<sup>th</sup>-generation, are the latest generation of the available adhesives on the market. They can be used in both etch-and-rinse and self-etch modes, as the name “all-in-one” implies (20).

Various methods are available for measuring microleakage, such as scanning electron microscopy, bacterial assessment or the dye penetration technique (26, 27). In previous studies, it has been shown that the results of microleakage assessment techniques do not differ significantly (28, 29). We used the dye penetration technique with silver nitrate in our study. This semiquantitative technique is easy to use and there are enough studies whose results can be used for comparison (23, 30).

The dye penetration technique uses silver nitrate for penetration. Silver nitrate particles are around 0.06 nm in size and therefore smaller than the smallest bacteria (0.5–1.0 µm) in the mouth. Therefore, if the adhesive succeeds in preventing the penetration of silver nitrate, it is therefore a very promising candidate for preventing bacterial microleakage (23). The simulation of clinical conditions is an important part of *in vitro* research in order to imitate thermal stresses in the oral cavity. Thermal changes can impair the bonding effectiveness and cause the formation of gaps, which leads to microleakage and impairs the long-term durability of the restoration (31).

In our study, thermal cycling was used for the simulation of oral environment and thermal stress. In a research by Gupta *et al.* (11), the microleakage for several adhesive generations was evaluated. Their results indicated that 6<sup>th</sup>-generation adhesives had the highest microleakage. However, they found no significant differences between total-etch, 7<sup>th</sup>-generation, and universal adhesives, which is consistent with the results of our study. According to our results, the microleakage of Iperbond Ultra was lower than that of Quickbond, which is a 6<sup>th</sup>-generation adhesive (P=0.077) (Table II).

These two adhesives have different bonding mechanisms and are different in the way they form a hybrid layer. The better performance of Iperbond Ultra compared to Quickbond could be related to the different organic solutions. Iperbond Ultra contains ethanol, while the organic solvent in Quickbond is acetone. Some studies have reported that acetone-based adhesives have a lower bond strength and retention rate compared to ethanol-based adhesives, which leads to higher microleakage. However, this also depends on the acetone content (32). In Deliperi *et al.*, the microleakage comparison between total-etch and self-etch adhesives, the acetone-based

adhesive showed the highest microleakage, which agrees with the results of our study (33).

Crystals that are persistent around collagen fibrils play an important role in preventing microleakage from bacterial penetration as they chemically react with functional monomers in adhesives (34, 35). In the one-step self-etching adhesive system, hydroxyapatite crystals are exposed to acidic monomers, but not completely broken down. Even with all-in-one adhesives, which combine etching, priming and gluing in a single bottle, the application is less time-consuming and has fewer steps and is easier compared to the other generations (30).

The statistical analysis showed that G-Premio Bond, a universal adhesive, had the lowest microleakage between all three groups, which means that it has a lower marginal permeability. It is also a single-bottle adhesive system and contains 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer, which has the ability to bind to calcium. The chemical bonding of MDP monomers to the tooth can improve long-term bonding, so that the likelihood of microleakage is lower (36).

Another functional monomer in G-Premio Bond is 4-MET, which also forms a chemical bond with the cavity surface, as it can form a strong bond with the calcium-containing substrates. This adhesive is also HEMA-free, as previous research has shown that HEMA can absorb surrounding water and leads to a decrease in bond strength (37). As a result, the bond will deteriorate over time due to residual, unpolymerized monomer in the adhesive layer (38). Based on the results of our study, the null hypothesis that there was no significant difference between microleakages in the adhesive groups was rejected.

## Conclusion

Within the limitation of this study, it can be concluded that Quickbond, a 6<sup>th</sup>-generation adhesive, has a higher level of microleakage, which can compromise the durability of the restoration. In addition, G-Premio Bond, a universal adhesive, has less microleakage than the other two adhesives. Functional monomers in adhesives play an important role in restorative microleakage that leads to their longevity. Long term clinical research is recommended to compare other features such as bond strength and reliability of these adhesives.

## Conflict of interest

The authors declare that there is no conflict of interest.

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