Comparative analysis of bond strength and microleakage of newer generation bonding agents to enamel and dentin: An in vitro study

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Abstract

Aims: This study aims to evaluate the bond strength (BS) and microleakage (ML) of the newer bonding agents to enamel and dentin.

Objective: (1) To analyze the BS between self-etch and total-etch adhesives. (2) To analyze the depth of ML between self-etch and total-etch adhesives.

Materials and Methods: Sixty mandibular premolars were fabricated and randomly divided into three groups: Group I (n = 20)-bonded with self-etch adhesive + nanohybrid, Group II (n = 20) with total-etch adhesive + micro hybrid and Group III (n = 20) with total-etch adhesive + nanocomposite. Teflon ring molds were used to make composite resin cylinders bonded to the buccal surface. Class II box cavity was prepared on the samples’ proximal surface and condensed with composite resin with each group’s specific bonding protocol. Shear BS and ML testing were conducted, and data analyzed.

Statistical Analysis: Kruskal–Wallis analysis was done to statistically differentiate the BS and ML between the three experimental groups; the P < 0.05, it showed a statistically significant difference. Intergroup comparison was made using the Mann–Whitney U test.

Conclusions: Within this study’s limitation, resin bonded with self-etch G-Premio Bond used in selective etch technique showed the highest BS and resistance to ML.

Keywords: Bond strength; bonding agents; microleakage; self-etch adhesive; total etch adhesive

INTRODUCTION

A rapidly developing specialty of dentistry is adhesive dentistry. The dental profession has been working to achieve strong adhesion of composite to tooth substrates for many years. Buonocore created Enamel acid etching in 1955, which gave way to total-etch methods.[1] The evaluation of bonding durability is essential as the long-term clinical performance of tooth-colored restorations depends on the consistency of the bond between restoration and tooth substrate.[2]

Resin-based composites obtain their retention, most notably by micromechanical adhesion to the structure of the tooth. The implementation of the etch and rinse method was the most productive technique to achieve sufficient resin composite bonding to the enamel. However, with additional rinsing and drying of the tooth, it requires a two-step or three-step procedure, making it tedious and technically sensitive.

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In the form of self-etch adhesives, a simpler procedure, a nonrinse approach, was introduced. By combining hydrophilic and hydrophobic acid functional monomers, organic solvents, and water into a single-bottle solution, this method incorporates all the etching steps, priming, and bonding into one step, making it more user-friendly and less technique sensitive.\[^{3}\]

Successful dentin bonding techniques have been extensively studied. The dentin substrate, however, differs from enamel, as it has more organic material, an increased presence of fluid within the dentin tubules, a smear layer, and surface wetness that is inherent. The resin infiltration into the acid-etched dentin to form a new structure composed of a resin matrix reinforced by collagen fibrils was demonstrated by Nakabayashi et al. This bio composite was named hybrid layer. After which, the concept of complete etching of the tooth was revolutionized by Fusayama.

Two patterns are involved in the latest adhesion strategies: the total-etch bonding technique distinguished by its components and bonding protocol, and following a simpler process, the self-etching systems. Etch and rinse adhesives, which dissolve the smear layer and superficial hydroxyapatite through etching with a separate acid gel, are the adhesion mechanisms currently followed; self-etch adhesive renders the smear layer permeable without obliterating it.\[^{4}\]

Self-etch adhesives were developed to prevent discrepancies between the depth of dentin demineralized by the acid and the ability of primer to penetrate this demineralized layer as it uses a weaker acid to partially remove the smear layer. Hence, in this study, self-etch adhesive is used in selective etch technique to correlate its bond strength (BS) with the total etch adhesive system.\[^{5}\]

G-Premio Bond (GC Asia Dental Pte. Ltd.), a universal bonding agent, 8th generation provides outstanding durability, compatible with total-etch self-etch, and selective etch techniques. A unusual combination of three functional monomers (4methacryloyloxyethyltrimeletic acids [4-MET], MDP and MDTP), notably excluding HEMA, ensures outstanding stability and exceptional BS not only for dental tissue but also for all indirect substrates, including precious and non-precious metals, composites, alumina, and zirconia for repair cases. This adhesive system was bonded with G-âenial Sculpt, a nanohybrid composite.

Adper™ Single Bond 2 Adhesive (3M ESPE) is a fast, simple, and convenient single-component total-etch bonding agent that offers outstanding BS. It includes BisGMA, HEMA, dimethacrylates, water, ethanol, a novel photo-initiator system, and a functional copolypemopolyacrylic and polyaconic acid methacrylate. The adhesive system contains five nanometer-diameter spherical silica particles, 10% by weight. They prevent accumulation, as the silane-treated particles are incorporated into the adhesive. Their small size keeps them in colloidal suspension as distinct particles. Filtek™ P60 Posterior Restorative System (3M ESPE), micro-hybrid composite, and Filtek™ Bulk Fill Posterior Restorative (3M), nanocomposite was bonded with this adhesive system.\[^{6}\]

**MATERIALS AND METHODS**

**Division of sample and materials**
- Group 1: G-Premio Bond (self-etch adhesive) + nanohybrid (G-âenial Sculpt) (n = 20)
- Group 2: Adper™ Single Bond 2 Adhesive (total-etch) + micro hybrid (Filtek™ P60 Posterior Restorative System Single Fill) (n = 20)
- Group 3: Adper™ Single Bond 2 Adhesive (total-etch) + nanocomposite (Filtek™ Bulk Fill) (n = 20).

**Methodology**

The ethical committee cert. no: ABSM/EC9/2017.

Sixty human mandibular premolar teeth with intact buccal/lingual surfaces removed for orthodontic or periodontal reasons were used. Debris was removed, the teeth were cleaned, autoclaved, and placed in distilled water at room temperature using an ultrasonic scaler. The teeth were used within 3 months of extraction, as per recommendations from the Occupational Safety and Health Administration guidelines for infection-control health-care settings.

**Preparation of samples**

A total of 60 class II (box only) cavities for microleakage (ML) evaluation were prepared. The buccolingual width of 4 mm and the depth of 1.5 mm, on mesial and distal surfaces using water cooled high-speed hand-piece with a diamond bur, to which composite resin was incrementally condensed. For testing the shear BS, the composite pellets were fabricated using a Teflon mold of 2 mm × 2 mm diameter bonded on the buccal surface and light cured, mounted on an acrylic block 2.5 cm in height and 1.5 cm thickness.

The specific bonding protocol was followed for all the groups (according to the manufacturer’s instruction)
1. The application of self-etch adhesive system in selective etch technique (G-Premio Bond)
   - The enamel surface in group 1 was etched using 37% phosphoric acid for 3 sec and applied with G-Premio Bond adhesive system, cured for 30 s.
2. Application of total-etch adhesive (Adper™ Single Bond 2 Adhesive)
   - The total-etch system was used in Group II, and Group III; the tooth surface was preetched with 37% phosphoric acid for 10 s and rinsed, to which two coats of Adper™ Single Bond 2 Adhesive was applied and cured for 15 s.
The specimens for BS and ML analysis were subjected to 300 cycles of thermocycling at 50°C–55°C ± 2°C, dwell time of 25 s.

BS analysis of samples (n = 30).

The prepared samples were then transferred to the universal Instron (3366) testing machine at a crosshead speed of 1 mm/min to check the shear BS. The shear BS values were calculated as the fracture load and bonding area ratio and expressed in megapascals.

ML strength analysis of samples (n = 30).

The surfaces of teeth were painted, two coats of nail varnish, leaving only the restoration and the surrounding 1 mm area of the tooth unpainted. They were immersed in freshly prepared, 2% solution of basic fuchsin dye, an interval of 48 h in separate containers. The samples were removed and washed under running water. The teeth were sectioned mesiodistally with a diamond disc, held in a straight hand-piece, and observed under a stereomicroscope at 40X magnification for the depth of ML. According to the depth of dye penetration, it was scored as follows:[5] 0-Penetration up to coronal one-third, 1-Penetration up to middle one-third, and 2-Penetration up to apical one-third.

**Statistical analysis**

The statistical difference among the three experimental groups was evaluated using Kruskal–Wallis analysis, and Mann–Whitney U tests made their intergroup comparison.

**RESULTS**

Kruskal–Wallis test was done to compare the statistically significant differences between BS of the bonding agent [Table 1]. The P value was 0.001, P < 0.05, indicating a considerable difference in BS among all groups. Mann–Whitney U test done to analyze the intergroup comparison, the P values were 0.001, which was P < 0.05, which indicates a significant difference between Group I versus Group II, Group II versus Group III, and Group III versus Group I.

Kruskal–Wallis test done to analyses the ML showed significant statistical difference between the three experimental group with P = 0.02, which is <0.05 [Table 2]. Mann–Whitney U test was done to analyze the intergroup comparison, which showed a statistically significant difference between Group I versus Group II (P = 0.04) and the Group I versus Group III (P = 0.007). No significant difference was seen between Group II versus Group III with P = 0.37, P > 0.05.

**DISCUSSION**

As the evaluation of BS and ML of bonding agents is essential for long-term clinical success.[7] The two primary purposes of the enamel and dentin adhesives are, the first being to minimize the marginal gap at the tooth-restoration interface, as ML is one of the essential parameters used to study the formation of the gap. The second purpose is to preserve the tooth restoration and resist any failure, as the strength of this bond is analyzed for shear BS of the bonding agent.[8]

It has been postulated that minimum BS of 17–20 MPa is needed to resist contraction forces of resin composite materials for enamel and dentin.[9] As shown in Table 1, the mean BS showed by the nanohybrid composite is higher (513.43 MPa) followed by the micro-hybrid system (313.18 MPa) and least demonstrated by the nanocomposite resin (243.54 MPa); this might be because of the bonding agent used, as micro-hybrid and nanocomposite is bonded with Adper™ Single Bond 2 Adhesive which works on the total-etch concept, etching both the enamel and dentin simultaneously. Whereas nanohybrid resin was bonded with G-Premio Bond, applied using the selective etch technique, which removes the smear layer leaving the smear plugs intact in the dentinal tubules and forms a hybrid layer hence shows the maximum adaptation of the restorative material.

In the total-etch concept of the Adper™ Single Bond 2 adhesive system, the hybrid layer is bisected by an adhesive resin tag. As the adhesive layer above indicates a continuous nanofiller concentration, the resin tag has a similar electron density. The particulate nanofiller inside the resin tag is higher (513.43 MPa) followed by the micro-hybrid and nanocomposite resin (243.54 MPa); this might be because of the bonding agent used, as micro-hybrid and nanocomposite is bonded with Adper™ Single Bond 2 Adhesive which works on the total-etch concept, etching both the enamel and dentin simultaneously. Whereas nanohybrid resin was bonded with G-Premio Bond, applied using the selective etch technique, which removes the smear layer leaving the smear plugs intact in the dentinal tubules and forms a hybrid layer hence shows the maximum adaptation of the restorative material.

![Table 1: Comparison of bond strength between the study groups](image)

| Group    | n  | Mean (SD)     | Range        | Median (Q1–Q3) | Kruskal-Wallis test | Mann-Whitney U-test |
|----------|----|--------------|--------------|---------------|--------------------|---------------------|
|          |    |              |              |               | \( \chi^2 \)         | Group I versus Group II | Group I versus Group III | Group II versus Group III |
| Group I  | 10 | 513.43 (4.27) | 505.9–520.3  | 513.95 (510.2–516.7) | 25.81 \*            | <0.001*            | <0.001*             | <0.001*             |
| Group II | 10 | 313.18 (5.28) | 305.9–325.9  | 312.1 (310.4–314.93) |                    | <0.001*            | <0.001*             | <0.001*             |
| Group III| 10 | 243.54 (22.90)| 200.3–260.9  | 251.4 (237.9–256.38) |                    |                    |                    |                    |

*P<0.05 statistically significant, P>0.05 NS. NS: Nonsignificant, SD: Standard deviation
achieved between the tooth and the restorative material. This outcome forms the long-lasting restoration foundation.

Adhesive systems, using material-specific formulations and simplified application techniques, have also been significantly improved.[10] In this study, single-step self-etch adhesives were chosen because they do not remove the smear layer and the smear plugs but only modify these structures, so the plugs remain sealed by the tubular orifices. This adhesion mechanism, which allows bonding without exposing the dentinal tubules, is believed to eliminate ML and hence postoperative sensitivity.[11] Scanning electronic microscope observation at dentin–adhesive interfaces reported an acid-base resistant zone beneath the hybrid layer after acid-base challenge in all-in-one adhesive was better compared to total-etch adhesives.[12]

The ML is higher in the nanocomposite [Table 2], followed by micro-hybrid resin, and the least is seen in nanohybrid resin. G-Premio Bond used in this study contained the functional monomers of 4MET, which have been reported to contribute to Resin-dentin bonding. The potential interaction of calcium in the tooth and 4MET results in additional chemical bonds. Although the single-step self-etch adhesives are easy to use in the present day dental practice, there are shortcomings and could not eliminate ML.[13] It showed that, on using all-in-one adhesive, the margins of restorations in enamel were more compromised than the margins of restorations, where phosphoric acid etchant was used.[14]

Pre-etching of enamel with phosphoric acid, partially erodes and produces a porous and retentive structure on the surface of hydroxyapatite crystals.[15] Acidic functional monomers could even bind effectively to preetched enamel chemically as dentin. Therefore, these results indicate that a simplified all-in-one adhesive system (G-Premio Bond) need pre-etching of the margins with phosphoric acid for an effective seal.[14] Hence, an attempt was made on preetching of enamel for 3 s as the previous studies indicate that decreased phosphoric acid preetching times do not affect the fatigue BS of universal adhesives.[16,17]

**Strength and limitation**

The current research was carried out under in vitro conditions and used to restore natural extracted teeth, and thermocycling was used as part of the test procedure. For the early evaluation of dental products, in vitro studies are very relevant. However, all the possible factors that differ from patient to patient are only considered in a clinical trial. Masticatory forces, food types, oral temperature, moisture variability, and the presence of salivary enzymes and bacterial by-products are some of the variables. Therefore, to confirm their in vitro findings, further studies are needed to assess these products’ substantial clinical value through clinical long-term follow-up.

**CONCLUSIONS**

Within the limitation of this study, it concludes that the self-etch G-Premio Bond system used in selective etch technique showed the highest shear BS and maximum resistance to ML compared to the resin bonded with total-etch Adper™ Single Bond 2 adhesive system.

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**Conflicts of interest**

There are no conflicts of interest.

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