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Micro-Tensile Bond Strength of Conventional and Self-adhering Flowable Composites to Intact and Incipient Caries-Affected Enamel

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ABSTRACT

Introduction: The clinical success of resin composite restorations largely depends on their bond strength to the dental substrate. This study aimed to compare the microtensile bond strength (µTBS) of conventional and self-adhering flowable composites to intact and incipient caries-affected enamel.

Materials & Methods: This *in vitro* study was conducted on 40 freshly extracted human third molars with sound enamel, free of caries and cracks. Standardized flat enamel surfaces (5 × 6 mm) were prepared on the buccal surfaces using silicon carbide abrasive papers. Artificial incipient caries lesions were induced in 20 specimens, while the remaining teeth served as the sound enamel group. Each group was subdivided into two subgroups (n = 10) according to the type of flowable resin composite used: a self-adhering flowable composite (Vertise Flow; Kerr, USA) and a conventional flowable composite (Filtek Z350; 3M ESPE, USA). Composite buildups with a height of 5 mm were placed on the prepared enamel surfaces following the manufacturers' instructions. The specimens were sectioned to obtain beam-shaped samples with a cross-sectional area of approximately 1 × 1 mm², and the microtensile bond strength (µTBS) was measured using a universal testing machine. Data were analyzed by two-way ANOVA (alpha=0.05).

Results: The highest µTBS was found in the Z350 Flow composite with intact enamel, while the lowest was observed in Vertise Flow with incipient caries enamel ($P<0.001$). In addition, a significant difference in µTBS was found between the two composite groups in both intact and incipient caries enamels ($P<0.001$).

Conclusion: These findings suggest that conventional flowable composites can provide more reliable bonding performance than self-adhering flowable composites, particularly when bonding to caries-affected enamel.

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Introduction

Dental caries is the most common chronic disease of childhood. One major reason for its high prevalence is the morphology of pits and fissures, which creates favorable sites for

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plaque accumulation and bacterial growth while being difficult to clean. In addition, enamel thickness is reduced in pit and fissure areas, which accelerates the demineralization process [1-3]. Dental caries is a dynamic process involving alternating phases of demineralization (acid attack) and remineralization of tooth structure at the surface and subsurface levels. Primary caries develops when the balance shifts toward demineralization [4,5].

One effective method for caries prevention is the application of a physical barrier over caries-prone pits and fissures, known as fissure sealants [1]. However, the low utilization of fissure sealants has been attributed to concerns regarding (a) inadequate bonding to enamel, (b) sealing over undetected carious lesions, and (c) difficulties in achieving proper isolation during placement [6]. Despite these concerns, several studies have demonstrated that residual caries beneath well-sealed fissure sealants remains arrested and does not progress [7,8].

Currently, the most commonly used fissure sealants are resin-based and glass ionomer-based materials. Resin-based fissure sealants show good adaptation to fissure walls, sufficient resistance to occlusal forces, and effective sealing due to their adhesive properties [9]. Flowable composites are widely used in clinical practice because of their ease of handling and injectable delivery systems. Their low viscosity and good wetting ability are expected to enhance adaptation to the internal walls of pits and fissures compared with conventional hybrid composites [10,11].

For optimal bonding, enamel surfaces must be etched with acids such as phosphoric acid to create a strong and durable resin–enamel interface. Despite their advantages, conventional resin-based materials are time-consuming and highly technique-sensitive [9]. Over the past decade, efforts have been made to reduce this technical sensitivity, leading to the development of self-adhering composites. These materials combine an all-in-one adhesive system with a flowable composite, eliminating the need for separate etching and bonding steps. Self-adhering flowable composites are considered more economical and clinically convenient, particularly for uncooperative patients such as children [10,12-14].

Durmuşlar et al. reported that the micro-tensile bond strength (μTBS) of conventional flowable composites to dentin was higher than that of self-adhering flowable composites [10]. Other studies have also indicated that the bond strength of conventional flowable composites to dental substrates is greater than that of self-adhesive flowable composites [12,13].

However, few studies have evaluated the μTBS of conventional and self-adhering flowable composites to enamel surfaces, particularly incipient caries-affected enamel. Therefore, the aim of this study was to evaluate and compare the μTBS of conventional and self-adhering flowable composites bonded to intact and incipient caries enamel when used as fissure sealants and preventive resin restorations (PRR).

Materials & Methods

Ethical Approval

This experimental study was approved by the Ethics Committee of Babol University of Medical Sciences (IR.MUBABOL.REC.1399.358).

Specimen Preparation

Forty freshly extracted human third molars without caries, restorations, or visible enamel cracks were selected. The teeth were disinfected by immersion in 0.5% chlorine solution for 24 hours [15].Then, a flat area measuring 5 × 6 mm was created on the buccal surfaces using 180 and 600 grit sandpaper [15-17].

Artificial Caries Induction

The specimens were randomly divided into two main groups (n = 20). In the first group, enamel surfaces were kept intact. In the second group, artificial incipient caries lesions were induced using a pH-cycling method. Each tooth was immersed at room temperature for 14 cycles consisting of two steps:

(1) immersion in 10 mL of demineralization solution (2.2 mM $CaCl_2$, 2.2 mM NaH_2PO_4, 0.05 M acetic acid) adjusted to pH 4.8 with 1 M KOH for 8 hours, and (2) immersion in 10 mL of remineralization solution (1.5 mM $CaCl_2$, 0.9 mM NaH_2PO_4, 0.15 mM KCl) adjusted to pH 7.0 for 16 hours. The depth of the induced carious lesions was evaluated using a polarized light microscope at ×50 magnification. Only specimens with a lesion depth of approximately 100 μm were included in the study [15, 18].

Restorative Procedures

Each main group was further divided into two subgroups (n = 10) based on the type of flowable composite used: a self-adhering flowable composite (Vertise Flow, Kerr Corp., Italy) and a conventional flowable composite (Filtek Z350, 3M ESPE, St. Paul, MN, USA). Table 1 lists all materials used in this study. Composite buildup to a height of 5 mm was performed on the prepared buccal surfaces using a mold according to the manufacturers' instructions.

For Vertise Flow, an initial layer was applied to a forcefully dried enamel surface, brushed for 15–20 seconds with moderate pressure, and light-cured for 20 seconds. Additional composite was then applied in increments of less than 2 mm, with each increment light-cured for 20 seconds. A light-curing unit with an intensity of 1000 mW/cm², verified using a radiometer, was used for polymerization [19]. The specimens were subsequently stored in distilled water for 24 hours.

For the conventional flowable composite, enamel etchant was applied using a syringe and left for 30 seconds, followed by rinsing with water for 20 seconds. The surface was air-dried, and an adhesive was applied using a microbrush, followed by gentle air dispersion. Light curing was then performed for 10 seconds. Finally, the mold was filled incrementally with Filtek Flow.

Micro-Tensile Bond Strength Testing

After storage in distilled water at 37°C for 24 hours, the restored teeth were vertically sectioned in both the mesiodistal and buccolingual directions along their long axis using a slow-speed diamond saw (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) under water cooling. This procedure produced rod-shaped micro-tensile specimens with a cross-sectional area of approximately 1 × 1 mm², yielding four specimens per tooth [17]. Each specimen was examined under a stereomicroscope to ensure structural integrity and homogeneity. The thickness of each specimen was measured using a digital caliper (Mitutoyo, Tokyo, Japan). A total of 40 specimens were obtained for each experimental group.

Then, the rod-shaped specimens were attached to a micro-tensile testing jig using cyanoacrylate glue and subjected to micro-tensile bond strength testing using a universal testing machine (Shimadzu AGS-X 5kN, Shimadzu Corporation, Kyoto, Japan). Tensile load was applied at a crosshead speed of 1 mm/min until failure occurred. The micro-tensile bond strength (µTBS) was calculated in megapascals (MPa) by dividing the maximum load at failure (N) by the bonded surface area (mm²).

Table 1. Materials used in the study

Material	Manufacturer	Composition	LOT number
Filtek Z350 (Nanohybrid flowable composite)	3M ESPE, St. Paul, USA	Bis GMA, TEGDMA, Bis-EMA 6 Zirconia/silicacluster, UDMA, silica nanoparticle	NA63866
Vertise flow	Kerr Corp, Italy	GPDM, HEMA, prepolymerized filler, nanosized colloidal silica, nano-sized ytterbium fluoride, 1-lm barium glass filler	G74G257
Adper single bond 2	3M, ESPE, USA	Ethanol. Water. 5nm silane treated Colloidal silica .2-hydroxyethylmethacrylate. Glycerol 1,3dimethacrylate.methacrylate functional copolymer of polyacrylic and poly itaconic acids and diurethane dimethacrylate. Bis-GMA	NA62587

Statistical analysis

The data were analyzed using SPSS 18 (IBM Corp., Armonk, NY, USA). The mean and standard deviation of µTBS were calculated for all groups, followed by data analysis using two-way ANOVA. A P value<0.05 was considered statistically significant.

Results

According to the two-way ANOVA results, there were significant differences in µTBS between intact and incipient caries enamels ($p < 0.001$) and between the types of composite ($p < 0.001$) (Table 2). Mean micro-tensile bond strength values and standard deviations for the experimental groups are shown in Table 2. Figure 1 illustrates the results obtained for micro-tensile bond strength.

According to the two-way ANOVA results, µTBS was affected by the type of enamel, with higher µTBS was higher in intact enamel than in incipient caries enamel for both composite groups, and this difference was significant. The highest µTBS was found in the Z350 Flow composite with intact enamel, while the lowest was observed in Vertise Flow with incipient caries enamel ($P<0.001$). In addition, a significant difference in µTBS was found between the two composite groups (in both intact and incipient caries enamels) ($P <0.001$).

In other words, the µTBS of the Z350 composite (in both intact and incipient caries enamels) was the highest, and this difference was significant compared to Vertise Flow composite (P <0.001).

Table 2. Micro-tensile bond strength values (µTBS*) (Mean±SD)

Type of enamel	Restorative material		P-value**
	Filtek Z350	Vertise Flow	
Intact enamel	29.89±2.32	13.61±3.10	<0.001
Incipient caries enamel	21.85±2.93	5.51±2.91	<0.001
P-value*	<0.001	<0.001	—

*Values are expressed in MPa

**Two-way ANOVA

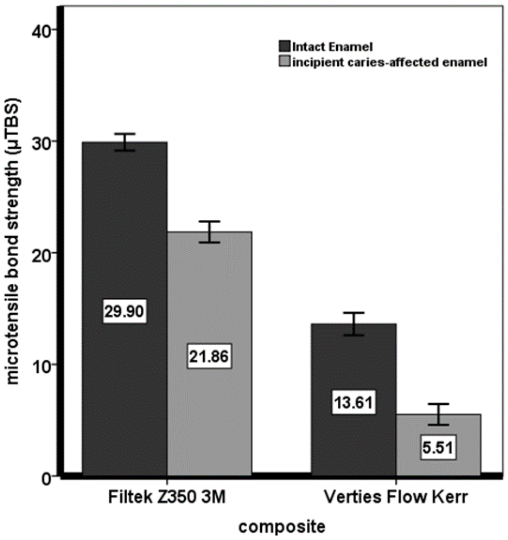

Figure 1. The bond strengths (MPa) (Mean±SD) for the different types of enamel and composite

Discussion

The results of this *in vitro* study showed that the µTBS was higher in intact enamel compared to incipient caries enamel in both composite groups. Furthermore, the µTBS of the conventional flowable composite was superior to that of the self-adhering composite in both intact and incipient caries enamels. These differences were statistically significant.

In this study, artificial enamel caries were created instead of natural enamel caries. The formation of artificial enamel caries through acidic demineralization produces lesions comparable to white spot lesions. This method is advantageous because it provides a smooth bonding surface and ensures a standardized degree of enamel demineralization [19].

The results of the present study demonstrated that the µTBS of the tested composites was significantly influenced by the enamel substrate. Specifically, both composites exhibited significantly higher µTBS values when bonded to intact enamel compared to incipient caries enamel. This finding is consistent with the study by Tedesco TK et al. and may be explained by the reduced mineral content, increased surface porosity, and enlargement of interprismatic spaces in carious enamel. These structural alterations can compromise the effectiveness of the acid-etching process, resulting in an irregular etching pattern, limited resin monomer penetration, and incomplete formation of resin tags, ultimately leading to reduced bond strength [15, 20].

On the other hand, Filtek Z350 flowable composite exhibited the highest µTBS values, showing a statistically significant difference compared with the self-adhering flowable composite. This finding is generally consistent with previous studies that have reported higher bond strength values for conventional flowable composites compared with self-adhering systems, although some variability has been reported in the literature depending on substrate conditions and experimental protocols [9, 10, 21].

The bonding mechanism of self-adhering composites such as Vertise™ Flow involves two main processes. The first is a chemical interaction between calcium ions in the tooth structure and the functional phosphate groups of the glycerophosphate dimethacrylate (GPDM) monomers within the resin. The second is micromechanical bonding, resulting from the etching of the tooth surface by the low pH of the resin material (pH ≈ 1.9), similar to the action of many self-etch adhesives [14,22,23].

On the other hand, the self-adhering composite Vertise™ Flow demonstrates limited wettability due to its relatively high filler loading (~70 wt%) and increased viscosity [24]. Moreover, the penetration of the GPDM monomer into enamel is restricted by its short molecular chain and pronounced hydrophilicity, which favors surface demineralization over chemical adhesion. As a result, the formation of stable chemical bonds with hydroxyapatite is compromised, leading to a lower overall bond strength of the composite [10,24-27].

Additionally, the absence of separate etching and bonding steps during the application of self-adhering composites results in weak adhesion to the enamel surface. Several studies have shown that using a separate adhesive significantly increases the bond strength of Vertise™ Flow, compared to conventional flowable composites [22].

According to the manufacturer, the pH of Vertise™ Flow (pH=1.9) is higher than that of phosphoric acid, indicating a lower etching potential. Although ionization occurs in the presence of water, the etching ability remains limited, resulting in insufficient resin penetration into the enamel surface. Additionally, all-in-one self-etch adhesive systems are susceptible to hydrolytic degradation, which compromises the adhesive interface and ultimately leads to reduced bond strength [9, 28, 29].

This study is limited by its in vitro design, which does not fully replicate the complex clinical oral environment, including thermal and mechanical stresses, salivary contamination, and long-term aging. Therefore, future clinical and long-term aging studies are recommended to better evaluate the bonding performance of these materials under conditions that more closely simulate the oral environment. Additionally, the effects of different surface pretreatments, adhesive strategies, and aging protocols on bond strength should be further investigated.

Conclusion

These findings suggest that conventional flowable composites can provide more reliable bonding performance than self-adhering flowable composites, particularly when bonding to caries-affected enamel.

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Conflict of interest

There was no conflict of interest.

Author’s Contribution

Maryam Salmani jelodar developed the original idea and protocol, summarized the data, drafted the manuscript. Asieh Khalilpour edited the article. Hemmat Gholinia analyzed the data. The study was supervised by Fariba Ezoji and Mahdieh Aziznejad.

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