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EFFECT OF SIMULATED INTRAPULPAL PRESSURE ON MICROTENSILE BOND STRENGTH OF SELF-ADHESIVE RESIN COMPOSITE TO DENTIN

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ABSTRACT

Background/Aim: This in vitro study aimed to evaluate the microtensile bond strength (μTBS) of a self-adhesive flowable resin composite (Vertise Flow) under simulated intrapulpal pressure IPP) and compare it to a conventional adhesive and flowable composite combination.

Materials and Methods: Thirty human molars were used. IPP of 15 cm H_2O was applied. Group I (control): Adeper Easy One + Z 250 (composite), Group II: Etching + Adeper Easy One + Z 250, Group III: Adeper Easy One + flowable composite + Z 250, Group IV: Etching + Adeper Easy One + flowable composite + Z 250, Group V: Vertise Flow + Z 250, and Group VI: Etching + Vertise Flow + Z 250. Data analysis employed one-way ANOVA with Tukey's post-hoc test for inter-group comparisons (at $P < 0.05$).

Results: Etching the dentin prior to bonding with Adeper Easy One significantly increased μTBS to 25.12 MPa (S.D.: 5.78 MPa). Application of flowable composite resulted in a μTBS of 13.62 MPa (S.D. = 5.50 MPa), not statistically different. Group IV exhibited the highest mean μTBS (31.26 MPa, S.D. = 6.04 MPa). Group V showed the lowest mean μTBS (8.89 MPa, S.D. = 0.97 MPa). Etching before application of Vertise Flow in Group VI showed significant increased μTBS to 13.83 MPa (S.D. = 1.33 MPa)

Conclusion: Dentin conditioning before application of Vetise Flow is necessary to produce a μTBS comparable to that of conventional approach of self-etch adhesive.

KEYWORDS: Intrapulpal pressure, microtensile bond strength, self-etch, flowable composite, self-adhesive resin composite.

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INTRODUCTION

The application of resin composites in dentistry has witnessed a significant surge in recent years, driven by continuous advancements in their material properties and subsequent performance enhancements[1]. Notably, the development of flowable resin composites has introduced numerous advantages. Their reduced viscosity, stemming from a lower filler content, translates to improved handling characteristics. This improved handling allows for easier placement and adaptation of the material to the walls of the cavity [2].

Flowable composites have been proposed for various dental applications beyond simple restorations. They can be employed as liners beneath the more viscous hybrid resin composites. In this role, they function as a stress-absorbing layer, their low moduli of elasticity and increased flow capacity might provide more contraction stress relaxation [2,3]. Additionally, their suitability extends to use as fissure sealants and for the restoration of small cavities. It was proposed that the consistency and hydrophobic properties of flowable composites contribute to preventing the outward passage of fluids from self-etch adhesives. [3] These adhesives can exhibit a permeable membrane-like behavior [4,5].

Vertise Flow represents a novel approach to restorative dentistry by incorporating self-etching adhesive monomers like phosphoric acid ester methacrylate and glycerol phosphate dimethacrylate (GPDM) within its composition. [6] The low pH of GPDM (around 1.9) offers a potential two-fold benefit, Self-Etching Effect through the acidic GPDM may eliminate the need for a separate etching step, simplifying the bonding procedure. This can be advantageous for dentists by reducing treatment time and potentially improving patient comfort. Also, the functional phosphate groups in GPDM can potentially interact chemically with calcium ions in the tooth dentin, promoting the formation of a hybrid layer between the composite and the tooth structure. [7] However, despite these potential advantages, several studies have raised concerns regarding the bond strength of Vertise Flow compared to conventional composites that rely on separate etching and bonding agents [8].

In addition, vital dentin exhibits inherent hydration due to the outward flow of dentinal fluid driven by a positive intratubular pulpal pressure of approximately 15 cm H_2O (equivalent to 1.5 kPa or 11.1 mm Hg) [9]. This fluid originates from the exposed dentin surface of the prepared cavity. Consequently, this dentinal fluid flow directly influences the bonding efficacy between dentin and restorative materials [10,11].

So that, dentin surface phosphoric acid etching was tried before the application of Verise Flow although it can deplete the hydroxyapatite content of dentin, potentially compromising the chemical bonding efficacy of GPDM, the functional monomer in Vertise Flow [12]. But it is important for dentists to have adequate comparative information to allow them to select the most appropriate material for any particular use. Furthermore, understanding the optimal application procedures is essential to maximize the material's performance.

MATERIALS AND METHODS

Sample Selection and Criteria:

This study utilized thirty extracted human molar teeth, free from caries and other defects. Each tooth underwent magnified examination to ensure the absence of cracks or other structural defects. Extracted teeth were stored in a 0.5% chloramine solution at 4°C and employed within one month. Molar teeth were chosen due to previous research by Pashley et al. 1981[13] demonstrating significantly higher hydraulic conductance of dentin in molars compared to other teeth, even with similar dentin thickness.

Tooth Preparation:

Root Sectioning:

Each tooth root undergoes sectioning approximately 3-4 millimeters apical to the cementoenamel junction (CEJ). Following sectioning, access to the pulp chamber is established at the furcation level. The pulp chamber contents were meticulously removed using a combination of endodontic barbed nerve broaches and cotton pliers. This approach aimed to minimizing disruption to the odontoblastic layer surrounding dentin by minimizing contact with the pulp chamber walls and the predentin surface. [10,14]

Dentin Surface Preparation:

A flat occlusal surface was created on each tooth to expose the dentin. This was achieved using a flat-end cylindrical diamond bur (ISO #111/014, Mani Inc., Utsunomiya, Tochigi, Japan) mounted in a high-speed handpiece with a copious air-water spray. The protocol specifies the preference for a flat occlusal table over a cavity preparation to minimize the influence of cavity configuration (C-factor) on the resultant bond strength. [15,16]

Dentin Thickness Standardization:

Using a pincer Iwanson thickness caliper (Renfert GmbH, Hilzingen, Germany), dentin thickness was carefully measured after enamel was removed and during the grinding operation. Measuring from the outer occlusal dentin surface to the pulp chamber roof's middle region (inner surface), 3 mm was the target thickness. This standardization made sure that all specimens had the same dentin thickness and, consequently, permeability. In comparison to thicker dentin, thinner dentin has shorter, hyper-conductive tubules, which increases permeability.[17, 18]

TABLE (1) Materials Compositions and Application Protocols

Material	Composition	Supplier	Application Protocols
Scotchbond Etching Gel	Amorphous silica-thickened 35% phosphoric acid gel	3M ESPE, St Paul, MN, USA	for 15 s before rinsing with water for 20 s
Adper Easy One (all-in-one adhesive)	2 HEMA, Bis GMA, methacrylated phosphoric esters, 1,6 hexanidol dimethacrylate, methacrylate functionalized polyalkenoic acid, camphorquinone, ethanol, water, silica filler, and stabilizer	3M ESPE, St Paul, MN, USA	5 s air dry. Apply adhesive to tooth surface for 20 s. Dry the adhesive for 5 s. Two consecutive layers of adhesive were applied and each layer was light cured for 10 s.
Filtek Flow Composite (A2)	Bis-GMA, TEGDMA, dimethacrylate polymer, zirconia–silica fillers	3M ESPE, St Paul, MN, USA	5 s air dry. Apply the material to adhesive layer with a dispensing tip. Brush the layer with moderate pressure for 15–20 seconds to obtain a thin layer (<0,5 mm). 20 s light-curing
Filtek Z250 Hybrid Resin Composite (A2)	Bis-GMA, UEDMA, Bis-EMA, zirconia–silica fillers	3M ESPE, St Paul, MN, USA	Two consecutive increments, of two mm thickness, were applied and light-cured separately for 40 seconds.
Vertise Flow	Matrix: GPDM adhesive monomer, UDMA, BisGMA, and other methacrylate comonomers, photoinitiators Fillers: 70% by weight. Ytterbium Fluoride, barium aluminosilicate glass, prepolymerized fillers, and colloidal silica	Kerr, Orange, CA, USA	5 s air dry. Apply the material to dentin with a dispensing tip. Brush the layer with moderate pressure for 20 seconds to obtain a thin layer (<0,5 mm). 20 s light-curing

To establish a consistent, uniform smear layer, the exposed dentin surface was polished for 30 seconds using 600-grit silicon carbide paper under running water. [19]

Grouping:

Teeth were randomly assigned to six equal groups (five teeth per group) based on the restoration protocol employed (detailed in Table 2).

TABLE (2) Groups with dentin surface treatment and restorative system buildup

Group	Restorative Protocol
G I	Adeper Easy One + Z 250
G II	Etching + Adeper Easy One + Z 250
G III	Adeper Easy One + Flowable + Z 250
G IV	Etching + Adeper Easy One + Flowable + Z 250
G V	Vetise Flow + Z 250
G VI	Etching + Vetise Flow + Z 250

Intrapulpal Pressure Simulation:

To reduce evaporation and heat loss, a special lidded plastic water bath container was constructed. There were apertures on the lid to place an embedded fan for water circulation and a digital temperature control unit that maintained a constant 37°C ± 0.1. A perfusion system filled with distilled water was attached to every tooth in order to replicate the physiological pulpal pressure of 15 cm H_2O (1.5 kPa or 11.1 mm Hg). Distilled water was chosen for dentin perfusion based on previous research indicating its superiority over other fluids (plasma serum, saline) in promoting higher bond strength values for adhesives. [20,21] Teeth were perfused for 24 hours before bonding and composite resin procedures to ensure complete dentin hydration. During bonding and restorative procedures, the specific row of teeth being treated was lifted from the water bath and positioned on a benchtop level with the container's bottom. This allowed procedures to be performed under dry conditions while maintaining perfusion and pulpal pressure. The burette position and pressure gradient were thus maintained throughout the adhesive and restorative material/materials application.

Specimen Preparation for Microtensile Bond Strength Testing:

The teeth were sectioned into several slabs with a low-speed diamond disc that was continuously cooled by water spray. In relation to the crown, sections were cut in an axial direction. After that, the slabs were sectioned once more in a perpendicular manner after being turned 90 degrees around the tooth's long axis. Specimens in the shape of beams and with a cross-sectional area of approximately one mm^2 were produced by this method. Four core beams, or a total of 20 beams per group, were chosen from each tooth's peripheral beams, which were not tested.

Microtensile Bond Strength Testing:

Universal testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with a 5 kN load cell at a crosshead speed of 0.5 mm/min was applied.

The exact dimensions (width and breadth) of each beam specimen were input into the machine computer. The load required for each beam debonding or failure was recorded in megapascals.

Statistical Analysis:

The collected data were analyzed statistically to assess significance between groups. This analysis was performed using one-way analysis of variance (ANOVA) and Tukey's post-hoc test ($P < 0.05$) within SPSS for Windows (version 22, IBM Corp., Chicago, IL, USA).

RESULTS

One-way analysis of variance (ANOVA) revealed a statistically significant effect of the examined variables on the microtensile bond strength of resin composite to dentin ($F(5, 114) = 419.45$, $p = .000$). This indicates that the different bonding protocols employed across the six groups significantly influenced the measured bond strength values.

Detailed Group Analysis:

Table 3 summarizes the mean microtensile bond strength (μTBS) and standard deviation (S.D.) values for each group. **Group I (Adeper Easy One + Z 250):** This group served as the control, with a mean μTBS of 15.38 MPa and a standard deviation of 5.29 MPa.

- **Group II (Etching + Adeper Easy One + Z 250):** Etching the dentin prior to bonding with Adeper Easy One and Z 250 significantly increased the mean μTBS to 25.12 MPa (S.D.: 5.78 MPa) ($p = 0.000$) compared to the control group.
- **Group III (Adeper Easy One + Flowable + Z 250):** The application of Adeper Easy One followed by a layer of flowable composite before Z 250 resulted in a mean μTBS of 13.62 MPa (S.D. = 5.50 MPa), which was not statistically different from the control group ($p = 0.462$).
- **Group IV (Etching + Adeper Easy One + Flowable + Z 250):** Similar to Group II, etching followed by Adeper Easy One, flowable composite, and Z 250 yielded a significantly higher mean μTBS (31.26 MPa, S.D. = 6.04 MPa) compared to the control group ($p = 0.000$). This group also exhibited the highest overall mean μTBS among all groups.
- **Group V (Vertise Flow + Z 250):** Utilizing Vertise Flow, a self-adhesive flowable composite, without prior etching resulted in the lowest mean μTBS (8.89 MPa, S.D. = 0.97 MPa) among all groups. This value was statistically different from the control group ($p = 0.000$).
- **Group VI (Etching + Vertise Flow + Z 250):** Etching before application of Vertise Flow and Z 250 increased the mean μTBS to 13.83 MPa (S.D. = 1.33 MPa) compared to Group V (without etching). However, this increase was not statistically significant from the control group ($p = 0.245$).

TABLE (3) Dentin microtensile bond strength mean readings and standard deviation.

Group	Restorative Protocol	Mean μTBS	S.D.	P-Value	Significant Difference
Group I	Adeper Easy One + Z 250	15.38	5.29	Control	A
Group II	Etching + Adeper Easy One + Z 250	25.12	5.78	.000*	B
Group III	Adeper Easy One + Flowable + Z 250	13.62	5.50	.462	A
Group IV	Etching + Adeper Easy One + Flowable + Z 250	31.26	6.04	.000*	C
Group V	Vetise Flow + Z 250	8.89	0.97	.000*	D
Group VI	Etching + Vetise Flow + Z 250	13.83	1.33	.245	A

: There was significant difference. Different letters mean statistically significant difference ($p < 0.05$) among study groups. A p-value of 0.05 was used to assess statistical significance between groups. Different letters within the "Significant Difference" column indicate statistically significant differences ($p < 0.05$) between groups.

Highest and lowest microtensile results

- Among all groups tested under simulated intrapulpal pressure, Group IV exhibited the highest microtensile bond strength to dentin. This group employed Adper Easy One following a preliminary acid conditioning step and a layer of Filtek Flow flowable composite before final restoration with Filtek Z250 hybrid resin composite.
- Conversely, Group V demonstrated the lowest microtensile bond strength to dentin. This group utilized Vertise Flow without any prior acid etching.

Effect of Dentin Acid Conditioning on Adper Easy One:

- A significant increase in Adper Easy One's microtensile bond strength (μTBS) was observed following dentin acid conditioning, reaching 25.12 MPa ± 5.78 MPa.
- In contrast, applying a layer of flowable composite over Adper Easy One without preliminary acid etching resulted in a non-significant decrease in bond strength, yielding the lowest value among all tested Adper Easy One groups (13.62 MPa ± 5.50 MPa).
- Notably, Adper Easy One achieved its highest μTBS value (31.26 MPa ± 6.04 MPa) when both phosphoric acid conditioning and flowable composite application were incorporated into the bonding protocol.

Effect of Acid Etching on Vertise Flow:

- Group V, utilizing Vertise Flow without any preparatory steps, exhibited a mean microtensile bond strength of 8.89 MPa ± 0.97 MPa.
- In Group IV, where an additional acid etching step was implemented before Vertise Flow application, a significantly higher bond strength value of 13.83 MPa ± 1.33 MPa was recorded.

Vertise Flow vs. Conventional Flowable Composite

Within the limitations of this study, Vertise Flow, the self-adhering flowable resin composite, demonstrated lower microtensile bond strength to dentin compared to the conventional flowable resin composite used in the control group(s).

DISCUSSION

The study acknowledges limitations such as the use of in vitro testing, which may not fully replicate the complexities of the oral environment. The additional factor of simulated pulpal pressure and its impact on bond strength is highlighted.

The study employed microtensile bond strength testing as the primary evaluation method. This choice is justified because bond strength tests are widely used for initial screening and comparison of adhesives. [22,23] The underlying principle is that a stronger adhesive bond between the restorative material and tooth structure translates to better resistance against stresses arising from resin polymerization and routine oral function.

The results show a significant improvement in microtensile bond strength when dentin is etched prior to bonding with a self-etch adhesive (Adeper Easy One in this study). This aligns with findings of Osorio et al. (2010) [24] who observed higher μTBS with preliminary etching. In agreement too, Taschner et al. (2010) [25] reported a significant increase in shear bond strength when a preliminary phosphoric acid etching of enamel and dentine before the application of two adhesive systems, one-step self-etch adhesive systems.

However, De Munck et al. (2003) [26] reported a decrease in μTBS for self-etch luting cements with acid etching. This discrepancy could be attributed to the etching leaving a demineralized, poorly infiltrated dentin surface. Van Landuyt et al. (2006) [27] also found a decrease in μTBS with acid etching for Clearfil SE Bond (a two-step self-etch adhesive). This difference might be due to the adhesive's specific chemical composition

(containing MDP monomer) and application protocol (converted to a three-step process in their study).

The study also suggests a potential negative influence of the flowable composite layer on the subsequent bond strength of Adeper Easy One (although not statistically significant). This could be attributed to factors such as incomplete removal of the oxygen-inhibited layer on the flowable composite surface. Further investigation is needed to elucidate this observation. Abdalla (2010) [28] reported no significant increase in microtensile bond strength (μTBS) when a flowable composite layer was added, regardless of the adhesive system employed. This included a total-etch adhesive and two two-step self-etch adhesives. In disagreement, De Goes et al. (2008) [29] found that placement of a low-viscosity flowable resin after adhesive application increased the microtensile bond strength for all the four tested adhesive systems (two etch-and-rinse, one two-step self-etch and one all-in-one). However, they also noted that this increase was statistically significant only for Clearfil SE Bond, a two-step self-etch adhesive containing the MDP monomer. They attributed this material-specific effect to potential interactions between the flowable resin and the adhesive components.

Vertise Flow employs acidic monomers functioning similarly to the contained acid etchant found in self-etch adhesives. [6] The self-adhesive composite, exhibited considerably lower bond strength compared to the conventional approach involving the self-etch adhesive, Adeper Easy One. This disparity could be attributed to several factors.

Self-adhesive composites aim to achieve simultaneous etching and resin infiltration, eliminating the need for separate etching, rinsing, priming, and bonding steps. However, Fu et al. (2013) [8] used transmission electron microscopy (TEM) and scanning electron microscopy (SEM) to detect dentin-resin gaps and vacant dentinal tubules. The weak adherence of Vertise Flow was connected with these observations. Interestingly, Vertise Flow non-rinse technique produced dentinal tubules that were opened and exposed a microporous fibrillar collagen network, which was similar to the outcome of phosphoric acid etching [8]. However, Vertise Flow's non-rinse method leaves calcium phosphates lodged in the dentin, in contrast to etch-and-rinse systems where rinsing eliminates these phosphates. It is postulated that this process has a role in compromising interfacial integrity and adhesion process [30].

Several investigations have revealed that Vertise Flow exhibits significantly lower bond strengths to both primary and permanent enamel and dentin compared to other flowable composite materials [6,31,32]. Notably, Vichi et al. (2013) [6] attributed this reduced bond strength to the material's inadequate wetting capability. Bektas et al. (2013) (33) attributed the low bond strength of Vertise Flow to permanent dentin to the inclusion of fillers, which might compromise wettability by increasing viscosity. In support of this, Eliades et al. (2013) [34] reported that Vertise Flow possesses a high filler content by weight (70%), potentially contributing to the observed decrease in both wettability and flow characteristics.

Previous research has attributed deficiencies associated with self-adhesive composites to three primary factors: (1) incomplete removal of the smear layer, (2) inadequate development of micromechanical retention due to the diminished etching capability of self-adhesive composites compared to conventional dentin bonding agents, and (3) potentially lower flowability of them. While manufacturer recommendations for Vertise Flow advocate brushing the initial layer onto the entire cavity surface for 20 seconds to enhance the efficacy of the acidic composite matrix through active application, Vichi et al. (2013) [6] reported that this technique did not improve the bond strength of Vertise Flow sufficiently.

Poitevin et al. (2013) [31] identified glycerol phosphate dimethacrylate (GPDM) as the functional

monomer responsible for Vertise Flow's self-etching properties. However, it was reported that they examined a two-step adhesive containing GPDM that revealed a shallow hybrid layer (2 μm) devoid of hydroxyapatite, suggesting that GPDM may etch, rather than chemically bond, to hydroxyapatite [6].

According to Wei et al. (2013) [35], self-adhesive flowable composites inherent hydrophilicity is implicated in their decreased bond strength. Because they contain functional acidic monomers, the resin's hydrophilicity is greatly increased, which may eventually cause plasticization and water absorption. The likelihood of filler separation and filler-matrix interface degradation may be further increased by this increased hydrophilicity [36]. Eliades et al. (2013) [34] provided evidence in support of this theory when they spotted that Vertise Flow showed excessive water uptake and increased hardness even after a week of water storage.

Finaly, self-adhesive flowable composite although having a low performance in bonding to dentin, as indicated by its low microtensile bond strength, but it might have a roll in some arising developments in application techniques that are employing a liner of flowable composite under the conventional composite, as in snow-plow technique in which a thin layer of flowable composite is placed on the pulpal floor of the cavity and the gingival margins. This layer is not cured, then a denser composite is placed on top of the uncured flowable composite, so that it pushes the flowable composite ahead of it, like a snow-plow pushing snow. The excess flowable composite is squeezed out of the cavity. [37,38]

CONCLUSION

Dentin acid conditioning significantly improved the bond strength of self-etch composite to dentin under simulated pulpal pressure. The findings of this study have significant implications for clinical practice. While self-adhesive composites like Vertise Flow offer the advantage of a simplified bonding procedure, their lower bond strength compared to conventional techniques raises concerns about their long-term durability, particularly under conditions of stress or high pulpal pressure. Careful consideration of these trade-offs is crucial when selecting restorative materials for different clinical scenarios. However, it is important to acknowledge that this study was conducted in vitro, and further research, including in vivo studies, is necessary to fully evaluate the clinical effectiveness of this approach.

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