The use of Self-Adhering Flowable Composite as Immediate Dentine Sealing Material: In Vitro Study

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Abstract

To investigate the usage of self-adhering flowable composite and universal adhesive for the immediate dentine sealing (IDS) technique under simulated pulpal pressure.

Thirty permanent third molars were divided into three subgroups according to IDS technique: universal adhesive (U), self-adhering flowable composite (F) and universal adhesive with self-adhering flowable composite (UF). They were tested under two conditions: simulated and non-simulated pulpal pressure (PP, NP). The pulpal chamber in the PP group was subjected to a hydrostatic pressure of 20 cm H_2O throughout the experiment. After IDS application, self-adhesive resin cement was employed to attach a composite rod to the treated dentine. Ten small beams from each group were randomly selected to investigate μTBS, mode of failure, and examine the ultrastructure of the bonding interface.

The μTBS of NP group was significantly greater than that of the PP group. The F-NP group yielded the highest μTBS, followed by the UF-NP and U-NP groups. While the F-PP and UF-PP groups provided comparable μTBS and significantly higher than the U-PP group.

One-layer IDS using self-adhering flowable composite showed higher μTBS than one-layer IDS using universal adhesive and two-layer IDS using universal adhesive and self-adhering flowable composite in both pulpal conditions.

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Introduction

Tooth preparation procedures for restorative treatments, especially fixed prostheses, involve cutting part of the dentine. The exposed dentine could be contaminated with bacteria and their toxins, which might invade the dentinal tubules and cause an inflammatory response of the dental pulp and postoperative sensitivity in some cases.[1,2] The residuals of dental impression materials and the remnants of provisional cement can also get dirt on the dentine surface, which affects the polymerization and bonding processes of permanent resin cement.[3-7]

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To prevent the problems mentioned above, the immediate dentine sealing technique (IDS) using dentine bonding agents applied after tooth preparation was introduced.[3,8,9] The advantages of this technique were supported by many studies[3,4,9-12], including the higher bond strength between dentine and resin cement, the reduction of marginal leakage, and the increase in the long-term survival rate of restorations. Later, a second layer of dental adhesives or flowable composite was suggested to completely seal the exposed dentinal tubules and increase bond strength even more.[11] However, a thin film of unfilled or lightly filled dental adhesives was suggested to be used for the IDS technique rather than a flowable composite.[13]

A recently developed self-adhering flowable composite was introduced to be used as a lining material since it had the same bonding quality as the self-etching bonding systems[14-16] and had the advantages of easy handling, time savings, low microleakage, and favorable bond to dentine. The hybrid layer of dental adhesives that

might strengthen the IDS approach has not yet been reported in any studies.[13] Testing the dentine adhesives under conditions of simulated pulpal pressure could also provide a close representation of the physiological conditions of a vital tooth, where a continuous outflow of dentinal fluid interferes with the formation of a complete hybrid layer[17,18] and weakens the bond strength of permanent cement. This study aims to investigate the use of self-adhering flowable composite and universal adhesive for the IDS technique under simulated pulpal pressure.

Materials and methods

Thirty sound human third molars were included in this study under the approval of the Human Experimentation Committee, Faculty of Dentistry, Chiang Mai University No. 28/2022. After being extracted, the surrounding soft tissue was removed, and the teeth were disinfected according to ISO/TS 11405/2015 specifications.[17] The tooth specimen was stored in a 1% chloramine T trihydrate solution for one week and transferred to grade 3 distilled water for up to six months.

The tooth, except 1/4 part of the buccal surface, was embedded in epoxy resin. A low-speed cutting machine (Isomet®1000, Buehler, USA) was used to section the root 1 mm below the cementoenamel junction (CEJ). The coronal pulpal tissue was removed through the cut end using small forceps. The tooth was cut parallel to the long axis using a diamond disc on a low-speed cutting machine under water coolant to expose the buccal dentine surface, which was approximately 5 mm in diameter. The remaining dentine thickness was measured and controlled at an average of 1 mm with a crown gauge caliper. The prepared surface was polished with 400-grit silicon carbide paper for 30 seconds with water irrigation.

The specimens were randomly divided into 2 major groups (simulated pulpal pressure; PP and non-simulated pulpal pressure; NP) for testing the effects of simulated vital and non-vital pulp conditions. Under simulated pulpal pressure condition, the Plexiglas plate (20 x 20 x 5 mm) with 18-gauge stainless steel tube insertion was attached to the cut end of the specimen using cyanoacrylate glue. The 20 cmH_2O hydrostatic pressure was applied to the system to create the outward flow through dentine, whereas no device was connected to the non-simulated pulpal pressure condition.

The teeth in both groups were randomly divided into 3 subgroups for 3 different IDS techniques: one-layer IDS using universal adhesive (3M™ Single bond universal adhesive; 3M ESPE, Seefeld, Germany) (U group), one-layer IDS using self-adhering flowable composite (Vertise™ flow; Kerr Corporation, USA) (F group), and two-layer IDS with universal adhesive and self-adhering flowable composite (UF group). An aluminum tape (25 μm thick) with 5 mm diameter hole was attached to the prepared dentine surface for controlling the bond area and the thickness of permanent cement.

For IDS application, in the U group, a universal adhesive was applied on the dentine surface using a microbrush with a light brushing motion for 20 seconds, followed by gentle air-blowing and light curing for 20 seconds. For the F group, a self-adhering flowable composite was applied according to the manufacturer's instructions with a scrubbing technique. For the UF group, the universal adhesive was applied for the first layer and light cured, and then the self-adhering flowable composite was applied for the second layer using the same technique as in the F group.

The composite rod (6 mm in diameter and 4 mm in height) was cemented to the treated dentine surface with resin cement (RelyX™ U200, 3M ESPE, USA) under a constant load of 10 N for 60 seconds. After resin polymerization and removal of excess cement, the specimens were stored in distilled water at 37 ◦C for 24 hours before testing for microtensile bond strength[18-20] (Figure 1).

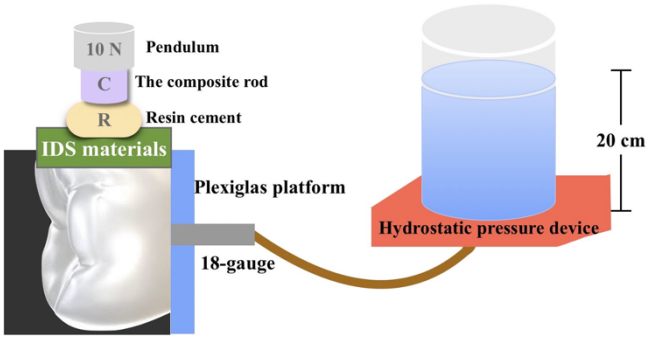

Figure 1. Preparation of specimens. IDS materials (green color) are the different factor from each group. Hydrostatic pressure device is only involved to the simulated pulpal pressure group.

The specimen was cut perpendicular to the bond surface into small beams of approximately 1 x 1 mm^2 using a low-speed cutting machine under water cooling. Ten beams were randomly selected from each subgroup for testing microtensile bond strength using the Universal Testing Machine (Instron Cop., Canton, MA, USA). Cyanoacrylate glue (Model repair II blue, Dentsply, Japan) was used to attach the specimen to the gripping device. The specimen was pulled at 1 mm/ min crosshead speed until fracture. The microtensile bond strength (MPa) was calculated from the maximum force (N) divided by the bonding area (mm^2).

The ultrastructure of the bonding interface was examined under the scanning electron microscope (SEM) (JSM-5910LV, Jeol, Massachusetts, USA). A low-speed cutting machine was used to cut a small beam at the bonding interface. The cut surface was decalcified by immersing it in 6 mol/L of HCl solution for 25 seconds, followed by deproteinization with a 6% NaOCl solution for 3 minutes. The ultrasonic cleaner was used to remove all dissolved debris from the decalcified and deproteinized surface. The specimens were air dried and desiccated in a dehumidifier chamber for 50 minutes, then gold sputter coated before being examined in the scanning electron microscope at 1,000x magnification.

The failure modes were examined using a stereomicroscope and recorded with a digital camera (SZX7 & SZ-ILST LED illuminator stand & E-330 & Olympus, Tokyo, Japan) at 50x magnification. The failure mode was categorized into adhesive, cohesive, and mixed failures. Adhesive failure is the breakdown of the interfacial bond between the adherend and the adhesive. When a fracture permits an adhesive layer to stay on both surfaces, it leads to a cohesive failure of the adhesive. A cohesive failure of the substrate occurs when the adherend fails before the adhesive. The preferred type of failure is cohesive failure within the adhesive or one of the adherends since it occurs when the joint's components have reached their maximum strength. The mixed or partial adhesive failure left some resin cement remaining on the dentine. Due to the mixed or partial adhesive failure, there was still some resin cement on the dentine.[21]

The data of μTBS was compared statistically with two-way ANOVA and Tukey's multiple comparison test using a statistical analysis program (SPSS for windows, version 24, SPSS Inc., USA). A *p*-value less than 0.05 was considered significantly different.

Results

The two-way ANOVA statistical analysis suggested that there were significant interactions ($p<0.05$) between the IDS techniques and pulpal pressure conditions. The non-simulated pulpal pressure group (NP) showed a higher μTBS (29. 84±13. 58 MPa) than the simulated pulpal pressure group (PP) (16.36±6.41 MPa) ($p<0.05$). Without considering pulpal pressure, one-layer IDS using self-adhering flowable composite (F) showed the highest μTBS (32. 34±11. 21 MPa) followed by two-layer IDS using both universal adhesive and self-adhering flowable composite (UF) (27. 33±7. 59 MPa) . While one-layer IDS using universal adhesive (U) showed the lowest μTBS (9.64±2.23 MPa).

With non-simulated pulpal pressure, the self-adhering flowable composite (F-NP) group showed the highest μTBS (43. 15±2. 08 MPa) followed by the combination group using universal adhesive and self-adhering flowable composite (UF-NP) (34. 68±0. 91 MPa) and universal adhesive (U-NP) groups (11. 70±0. 87 MPa) respectively. While under simulated pulpal pressure condition, the comparable μTBS results of the F-PP and UF-PP groups (21. 52±1. 04, 19.99±0.98 MPa, respectively) were significantly higher than those of the U-PP group (7. 58±0. 55 MPa) . Interestingly, one-layer IDS using self-adhering flowable composite showed the highest bond strength for both pulpal conditions (Table 1).

Bonding technique	Mean ± SD (MPa)	
	Non-simulated pulpal pressure (NP)	Simulation of pulpal pressure (PP)
One-layer IDS using universal adhesive (U)	11.70±0.87*	7.58±0.55*
One-layer IDS using self-adhering flowable composite (F)	43.15±2.08*	21.52±1.04
Two-layer IDS using universal adhesive and self-adhering flowable composite (UF)	34.68±0.91*	19.99±0.98

Table 1. Mean ± SD of microtensile bond strength of 3 bonding techniques under non-simulated pulpal pressure and simulated pulpal pressure conditions.

* indicated significant difference ($p<0.05$).

Adhesive failure was presented mostly in the U and UF groups in both simulated pulpal and non-simulated pulpal conditions (mean 65 and 70%, respectively). While the highest cohesive failure was found in the F-NP group (50%) and the mixed failure was shown the most in the F-PP group (40%) (Figure 2) (Table 2).

The SEM images showed that there were resin tags extended into some dentinal tubules apart from the adhesive layer in all groups (Figure 3) . The non-simulated pulpal pressure group showed longer resin tags and more complete adhesive layers than the simulated pulpal pressure group. In particular, the F-NP group showed the largest number and the longest resin tags.

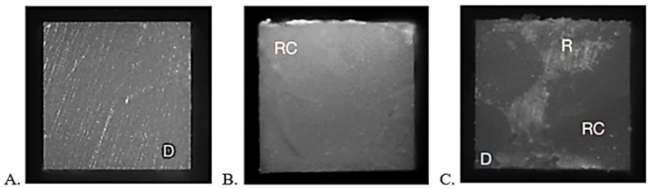

Figure 2. Stereomicroscope micrograph of failure mode; A. Adhesive failure at interface, B. Cohesive failure in resin composite, C. Mixed failure Abbreviation: R: Resin cement, D: Dentine, RC: Resin composite.

Group		% Failure Mode		
		Adhesive (A)	Cohesive (B)	Mixed (C)
U	U-NP	**70**	10	20
	U-PP	**60**	10	30
F	F-NP	20	**50**	30
	F-PP	30	30	**40**
UF	UF-NP	**60**	20	20
	UF-PP	**80**	10	10

Table 2. Percentage of failure mode.

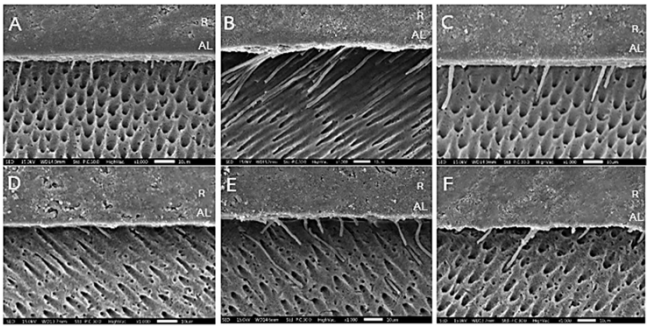

Figure 3. Bonding interface between resin cement and dentine under SEM at 1,000x magnification. A. U-NP group, B. F-NP group, C. UF-NP group, D. U-PP group, E. F-PP group, F. UF-PP group Abbreviation: R: Resin cement, AL: Adhesive layer.

Discussion

A single-layer immediate dentine sealing using self-adhering flowable composite yielded significantly higher bond strength of permanent resin cement than a single-layer immediate dentine sealing with universal adhesive and double-layer immediate dentine with both universal adhesive and self-adhering flowable composite in both simulated pulpal and non-simulated pulpal conditions.

In 2014, Tuloglu and his colleagues[22] found the mean shear bond strength (SBS) of self-adhering flowable composite was lower than the universal adhesive. On the contrary, this study showed that the F-NP group had a significantly higher μTBS (p<0.05) than the U-NP group in the non-simulated pulpal pressure condition. Furthermore, the result of the IDS technique using universal adhesive and a self-adhering flowable composite showed a significant improvement in bond strength.

An excessive thickness of a lightly filled adhesive resin layer could affect the cohesive strength of the adhesives at the bonding interface.[23] The result of this study suggested that the one-layer IDS using self-adhering flowable composite (containing fillers up to 70% by weight)[24] has greater μTBS than the universal adhesive, which contains approximately 0.5-40% fillers by weight.

According to the study of Stavridakis and his colleagues in 2005[25], the self-adhering flowable composite had a bonding interface thickness of about 70-180 μm. During our pilot study, we found that self-adhering flowable composite had low tensile bond strength, which might be caused by its thickness, an incomplete infiltration of the adhesives into demineralized dentine, or ineffective sealing of dentine tubules.[26] In order to increase the bond strength, we improved the technique of the self-adhering flowable composite application. The monomer was led onto the etched dentine by scrubbing with a microbrush, followed by air-blowing to create a thin film of adhesives. This procedure facilitated the bonding of the adhesives to the composite rod and the dentine surface. This technique was slightly modified from the

manufactory instruction.

This scrubbing technique had also been used in other studies and received a good result[27] as it accelerated the evaporation of solvent contained in dental adhesives and increased the diffusion potential of monomer into decalcified dental tissue.[28] As a result, the μTBS of self-adhering flowable composite in this study (43. 15±2. 08 MPa) was higher than the value (32.66±8.2 MPa) in the other study.[29] The same technique was also recommended for the application of universal adhesive, as it involved the evaporation of the solvent and water to form a uniform adhesive layer, that remained on the surface.[30]

Even though the thickness of the self-adhering flowable composite was controlled to a minimum, it was still thick enough to form a hybrid layer with decalcified dentine and leave an oxygen-inhibiting layer to bond with resin cement. From the studies of Bektas[15], Tuloglu[22], and Garcia[31], they found that the shear bond strength of self-adhering flowable composite obtained by using the scrubbing technique (23.7 ± 5.28 MPa, 193 ± 23 MPa, and 20.8 ± 3.2 MPa, respectively) was higher than obtained by using the cylindrical casting technique in the studies of Vichi[16] (3.4 ± 1.6 MPa) and Peterson[32] (6.5 ± 6.2 MPa).

Under the simulated pulpal pressure condition, μTBS values significantly decreased in every adhesive group, which corresponded to many studies.[12,33,34] The continuous outward flow of dentinal fluid in vital tooth[35] and in simulated pulpal pressure conditions interfered with the penetration of dental adhesives into the demineralized dentine. The flow also diluted the concentration of dental adhesives, resulting in impeding the formation of a hybrid layer, reducing bond strength, and substantially increasing hydrolytic degradation of the bonded interface. [36] The double application of dental adhesives, especially for thinner filler loading, was recommended to enhance the mechanical properties of the adhesive layer by bringing the adhesive layer much closer to the optimum thickness.[37] It also granted higher bond strength values than a single application significantly.[38,39] Moreover, it provided a larger plastic zone, which can disperse stress concentration, and produced a more homogeneous adhesive layer. Lastly, it compensated for possible application defects that were left over from a single application mode.

The result indicated that double application of dental adhesives using universal adhesive and self-adhering flowable composite (UF group) under simulated pulpal pressure showed no advantage over a single application of self-adhering flowable composite (F group) . In the same way, the study by Tuloglu and colleagues[22] stated that using the self-adhering flowable composite in conjunction with a self-etch adhesive provided significantly greater SBS values than using only universal adhesive.

The two-layer IDS using universal adhesive and self-adhering flowable composite (UF group) showed a higher μTBS than the one-layer IDS using universal adhesive (U group), but a lower result than the one-layer self-adhering flowable composite (F group). The SEM images showed that the U group presented the shortest and thinnest resin tags, while the F group had the longest, thickest, and largest number of resin tags. In UF groups, SEM images showed microcracks between the U and F layers, which may have occurred from the cutting procedure, but specimens were still attached. Consistent with our study, the study by Carvalho presented that failures were originally occurred at the hybrid layer interface in the IDS group.[13]

The adhesive failure, which occurred at the dentine-resin interface, was mainly found in the U and UF groups. This failure might be caused by incomplete polymerization of the hybrid layer, residual free radicals from the preparation procedure, and environmental conditions including carbon dioxide, pH level, high humidity, and oxygen.[21] A process of pretreatment with the acidic monomer of the universal adhesive might not provide a completely etched surface. The smear layer and smear plug might partially remain in the dentinal tubules, resulting in interference with the bonding ability and an incomplete hybrid layer. These findings were similar to those of Brueckner et al.[40], who found that the self-adhering flowable with Vertise Flow showed significantly more cohesive defects than other self-adhering flowable composites. Cohesive failure within a composite layer was found to be prominent in the F-NP group. This result suggested that the bond of dental adhesives to dentine and to composite resin was stronger than the covalent bond in the composite resin itself.

Under non-simulated pulpal pressure, the F group showed cohesive failure, which indicated

a higher bond strength at the bonding interface than the cohesive bond in composite resin material. Under simulated pulpal pressure condition, the mixed failure occurred more due to the moisture absorption into the dental adhesives. This caused plasticization or swelling, which subsequently facilitated interfacial crack growth, resulting in decreased joining strength.[41]

Conclusions

One-layer IDS with self-adhering flowable composite showed a higher μTBS than both one-layer IDS with universal adhesive and two-layer IDS with universal adhesive and self-adhering flowable composite in both pulpal pressure conditions. While under a simulated pulpal pressure condition, the μTBS of one-layer IDS with self-adhering flowable composite and two-layer IDS using universal adhesive and self-adhering flowable composite showed no significant difference.

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Declaration of Interest

The authors report no conflict of interest.

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