Evaluation of micro-shear bond strength of self-adhesive giomer to bovine tooth

Eunjin Kim

The Graduate School

Yonsei University

Department of Dentistry

Evaluation of micro-shear bond strength of self-adhesive giomer to bovine tooth

A Master's Thesis

Submitted to the Department of Dentistry

and the Graduate School of Yonsei University

in partial fulfillment of the

requirements for the degree of

Master of Dental Science

Eunjin Kim

June 2024

This certifies that the Master's thesis

of Eunjin Kim is approved.

_____

Jeong-Won Park

_____

Yooseok Shin

_____

Dohyun Kim

The Graduate School

Yonsei University

June 2024

감사의 글

논문을 완성하기까지 옆에서 저를 많이 격려해주시고 아낌없이 지도해주신 박정원 교수님께 감사의 말씀을 올립니다. 교수님께서 차근차근히 알려주시고 배려해주셔서 부족한 제가 무사히 마무리할 수 있었습니다. 교수님 덕분에 책으로는 알 수 없는 지식을 배웠고 한층 넓어진 시야를 얻어 더욱 성장하는 경험을 할 수 있었습니다. 그리고 따뜻한 관심과 조언을 통해 논문의 완성도에 도움을 주신 신유석 교수님과 김도현 교수님께도 감사드립니다. 아울러 실험에 필요했던 장비를 편하게 사용하게 해주신 서덕규 교수님께도 감사드립니다. 많은분들의 도움으로 학위논문을 잘 마무리할 수 있었습니다.

수련기간 동안 많은 가르침을 주시고 부족한 저를 이끌어주신 김정희 부장님, 김선호 과장님, 김미연 과장님, 김난아 과장님, 김혜진 과장님, 장진영 과장님께도 감사드립니다. 그리고 행복한 의국생활을 할 수 있게 해주신 김성은 선생님, 박소현 선생님, 한벼리 선생님, 김대영 선생님께도 감사의 말씀 드리며 유라를 비롯한 우리 보존과 의국원들에게도 고맙습니다.

마지막으로 사랑하는 부모님께도 깊이 감사드립니다. 항상 저를 믿고 지켜봐주시며 응원해주신 덕분에 지금까지 올 수 있었습니다. 감사합니다.

2024년 6월 김은진 올림

Table of Contents

List of Figures

List of Tables

Abstract

Evaluation of micro-shear bond strength of self-adhesive giomer to bovine tooth

Eunjin Kim

Department of Dentistry, Graduate School, Yonsei University

(Directed by Professor Jeong-Won Park, D.D.S., M.S., Ph.D.)

Self-adhesive flowable giomer (SAG) has been used in dental practice recently to simplify clinical procedures and shorten chair times. However, there are only few studies evaluating its bond strength to enamel and dentin, resulting in a lack of evidence. This in vitro study aimed to evaluate the micro-shear bond strength (μ-SBS) of SAG bonded with or without a dental bonding system to enamel and dentin, both before and after thermocycling.

Sound bovine teeth were used as the tooth substrates. For μ-SBS tests, enamel and dentin specimens were prepared for SAG (Beautifil Kids SA - BK), a self-adhesive flowable composite (Vertise Flow - VF), and a nanohybrid flowable giomer (Beautifil Flow Plus F03 - BF). Two adhesive modes were tested for BK and VF (with self-etching adhesive and no adhesive), and one for BF (with self-etching adhesive). The μ-SBS test was conducted after 24 h and after

thermocycling for 10,000 cycles using a universal testing machine.

For all materials, when self-etching adhesive was used, the μ-SBS was significantly higher than that of no adhesive group ($p < 0.05$). No statistically significant difference was found between the restorative materials under any condition. Thermocycling had no significant effect on the μ-SBS of BK. In self-etching adhesive group, mixed failure was predominant for all materials. However, in no adhesive group, adhesive failure and mixed failure were observed at similar levels for all materials.

The bonding performance of self-adhesive material to the dental substrate was significantly weaker without adhesive compared to conventional adhesive. No statistically significant difference was found between the restorative materials under any condition. BK and BF were negligibly affected by thermocycling, but VF was affected. Therefore, this study showed that self-adhesive flowable composite has lower bond strength stability in enamel and dentin compared to SAG.

Keywords: Self-adhesive giomer; Surface pre-reacted glass-ionomer filler; Micro shear bond strength

I. Introduction

In the middle of the 1990s, flowable composites were released into dentistry. Due to their low viscosity, flowable composites can be shaped to fit cavity areas that are challenging to access (1, 2). Flowable composites have a broad range of uses, including use as pit and fissure sealants, conservative composite restorations, and cavity liners (3-5).

Restorative treatments using composite resins bonded to tooth structures became possible after Buonocore introduced resin adhesive techniques in 1955 (6). For these conventional composite resins to adhere to tooth structure, an additional adhesive is needed. One of the main goals in the development of dental adhesives has been to simplify the process. The extraordinary advancements in adhesives and techniques throughout the years have simplified and expedited clinical procedures using adhesive systems. The benefits of using simpler adhesive systems include chair time savings, less patient strain, and fewer procedural errors (7, 8). One of the main goals of dental manufacturers' current research and development is to make the clinical application of adhesive procedures simpler.

To decrease errors in clinical steps and shorten treatment times, self-adhesive flowable composites (SAC) have been developed (9). It simplifies the adhesive process by combining the benefits of flowable composites with self-etch adhesive technology and eliminates the requirement for pretreatment of the tooth (10). SAC's composition is similar to that of other flowable composites but includes acidic (functional) monomers, like 4- methacryloxyethyl trimellitic acid (4-MET), glycerol phosphate dimethacrylate (GPDM), and 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which are currently used in dental adhesives (9, 11). These acidic monomers can demineralize the tooth substrate and this provides a self-adhesion mechanism by promoting micromechanical and chemical interactions between hydroxyapatite and phosphate acidic groups

(12). This mechanism is based on what was formerly referred to as the “adhesive demineralization concept” (AD concept) (8, 13). According to manufacturers, these composites have adhesive qualities similar to self-etching bonding systems, making them appropriate for use as lining materials and as filling materials in small restorations (14, 15). Carla David et al. assessed bond strengths of SAC and conventional composite resins by systematically reviewing the literature and showed that regardless of the substrate assessed, the time of storage, and type of dentition, the bonding performance of SAC was significantly lower than that of traditional composite resins when combined with adhesive systems. (16).

By the acid-base reaction between polyalkenoic acids and fluoroaluminosilicate glasses, the surface-pre reacted glass ionomer (S-PRG) filler can be created in the presence of water (9). This uses the same mechanism as the glass ionomer and after the freeze gelatinization process, it is ground to form the fillers and silanized (17). One kind of composite resin that contains S-PRG filler is called giomer. Six different ions can be released and recharged in S-PRG fillers: fluoride, strontium, sodium, aluminum, silicate, and borate (18). Therefore, it has demonstrated a variety of benefits in treating dental caries by inhibiting the growth of oral bacteria and plaque formation (19-22).

SAG was recently developed by Shofu company (Kyoto, Japan). Like other SAC, SAG can be used without the need for a bonding procedure because its resin components contain phosphonic acid monomer, which can bond to the tooth structure by itself. (23). Because the product itself interacts with the tooth surface and create ion exchange, manufacturers recommended using SAG without the use of adhesives (24, 25). However, SAG has higher viscosity than conventional dental adhesives, its wetting to the dental hard tissue is difficult and this can deteriorate the demineralization and penetration of the tooth substrate. As a results, in the previous studies, SAC showed low bond strength to both enamel and dentin (16). The newly released SAG has the same mechanism with glass ionomer cement for bonding, it may have a higher bonding strength than SAC.

So it was determined to investigate this novel material to provide more data and evaluate its adhesive capability.

The aim of this in vitro study was to evaluate the micro-shear bond strength (μ-SBS) of SAG bonded with or without self-adhesive bonding system to enamel and dentin before and after thermocycling. The null hypotheses of the study were that materials, adhesive modes and thermocycling would not influence the μ-SBS to enamel and dentin.

II. Materials and methods

1. Materials

Three flowable composites were tested; a novel SAG (Beautifil Kids SA), a SAC (Vertise Flow), and a nano-hybrid giomer (Beautifil Flow Plus F03). The compositions of the tested materials of the study are listed in Table 1.

2. Preparation of the specimens

Tooth substrates were extracted intact bovine incisors without caries, discoloration, or structural defects. After removing the periodontal ligament and other surface contaminants using a scaler, the teeth were immersed in distilled water and stored at 4.0 °C until the study and used within 3 months after extraction. The distilled water solution was replaced every month.

Root portion of the tooth were trimmed off with a water-cooled model trimmer at the cemento-enamel junction and the pulp tissue was removed. The prepared tooth was embedded in a self-polymerizing acrylic resin (Ortho-Jet, Lang Dental, Wheeling, IL, USA) with a diameter of 30 mm and a height of 10 mm mold and buccal surface was exposed.

A water-cooled model trimmer was used to expose enamel or dentin surface and polished with #600 and #800 silicon carbide paper for 30 s each to get the homogeneously roughed surface. Figure 1 demonstrates the flow chart of this study.

Table 1. The compositions of tested materials.

Material (Abbreviation)	Type	Composition	Manufacturer	Lot no.
Beautifil Kids SA (BK)	Self-adhesive giomer	Matrix: UDMA, HEMA, phosphonic acid monomer Filler: S-PRG filler based on fluoroboroaluminosilicate glass Others: polymerization initiator, pigments	Shofu Dental Corporation, Kyoto, Japan	122209
Vertise Flow (VF)	Self-adhesive flowable composite	Matrix: GPDM and methacrylate co-monomers Filler: pre-polymerized filler, barium glass, nano-sized colloidal silica, nano-sized ytterbium fluoride	Kerr Corporation, Orange, CA, USA	9194881
Beautifil Flow Plus F03 (BF)	Nanohybrid flowable giomer	Matrix: Bis-GMA, TEGDMA Filler and others: Same with BK	Shofu Dental Corporation, Kyoto, Japan	112256
Singlebond universal (SBU)	Universal adhesive	MDP, bis-GMA HEMA, DMA, methacrylate functional copolymer, filler, ethanol, water, initiators, silane	3M ESPE, St. Paul, MN, USA	30327B

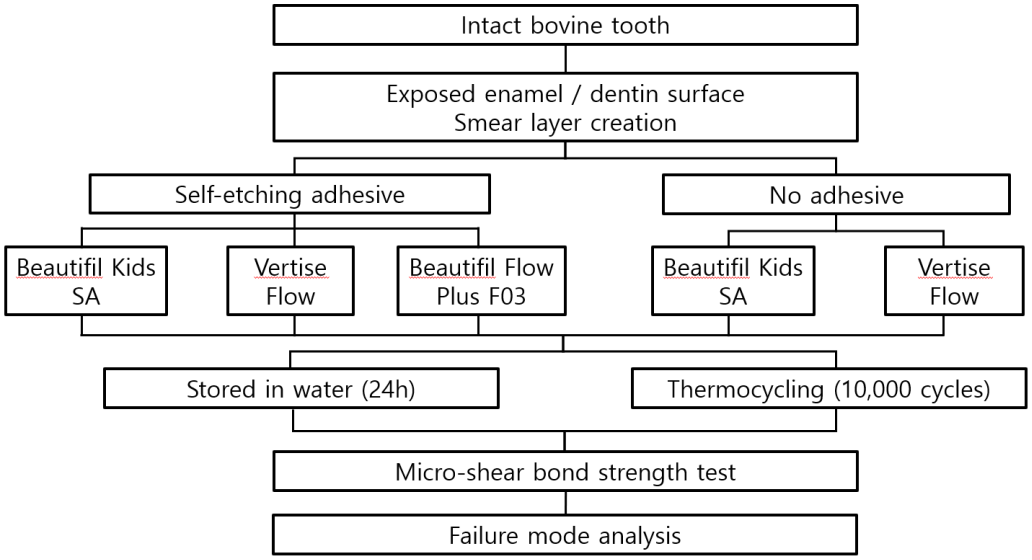

Fig. 1. Flowchart of the procedures of this experiment.

3. Experimental groups of the study

On the prepared enamel and dentin surfaces, flowable composites were bonded following the manufacturer's instructions (Table 2). Two adhesive modes were performed for BK, VF (with self-etching adhesive and no adhesive) and one for BF (with self-etching adhesive).

Table 2. Summary of the manufacturer's instructions.

Material (Abbreviation)	Application
Beautifil Kids SA (BK)	1. Apply BK in a thin layer (≤ 0.5 mm) with needle tip. Leave for 20 s. 2. Light cure for 5 s. 3. Apply additional increments (≤ 2 mm). 4. Light cure each increment for 10 s.
Vertise Flow (VF)	1. Dispense VF onto preparation in a thin layer (<0.5 mm) with provided dispensing tip. Leave for 15-20 s. 2. Light cure for 20 s. 3. After lining the cavity wall build the restoration with more VF in increments of 2 mm or less. 4. Light cure each increment for 20 s.
Beautifil Flow Plus F03 (BF)	1. Apply dentin adhesive. 2. Apply BF directly into the cavity. 3. Light cure for 10 s.

- Self-etching adhesive group

The SBU was applied to the tooth surface with an agitating motion for 20 s, air dried for 5 s and light cured for 10 s using a LED light curing unit (Elipar Deep Cure L, 3M ESPE, St. Paul, MN, USA) with an intensity of 1,400 mW/cm^2. The flowable composite filled a polyethylene Tygon tube with an internal diameter of 0.8 mm (0.5 mm$^{2)}$ and a height of 2 mm.

- No adhesive group

After using a water spray for 10 s to clean the tooth surface, the excess moisture was air dried. Self-adhesive composite was filled the Tygon tubes and left for 20 s for bonding interaction can occurred and light cured following the manufacturer's instructions.

To reduce the error of the experiment, one operator conducted the experiment.

4. Micro-shear bond strength (μ-SBS) test

After the preparation of the specimens, μ-SBS test was done at 24 hours after bonding stored in distilled water at 37.0 ℃ and after 10,000 thermocycles between 5 °C and 55 °C with a 30 s dwell time.

The μ-SBS test was performed using a universal testing machine (EZ-test, Shimadzu Corp., Kyoto, Japan) equipped with 500 N load cell and a crosshead speed of 1 mm/min. Shear force was applied to the bonded interface using an orthodontic wire with a diameter of 0.2 mm. The loop wire was placed as close to as possible to the bonded surface to minimize the torque force. Trapezium X software (version 1.5.1, Shimadzu Corp., Kyoto, Japan) were used to collect and analyze the bond strength data. The measured data (N) were calculated to MPa by dividing force by the bonded surface area (0.5 mm^2).

5. Failure mode analysis

After measuring the μ-SBS, the surfaces of the specimens were observed using an optical microscope (Zeiss Extaro 300, Carl Zeiss Meditec AG, Oberkochen, Germany) at 20 × and 31 x magnifications to evaluate the failure mode. Three categories were used to categorize the failure mode: cohesive failure; adhesive failure; and mixed failure.

After failure mode analysis using an optical microscope, representative specimens were photographed at up to 70 x magnifications using a scanning electron microscope (Hitachi S-3000N, Tokyo, Japan).

6. Statistical analysis

All statistical analyses were performed using SAS version 9.4 (SAS Institute, Cary, NC, USA). ANOVA and independent two-sample t-test were used to compare the mean μ-SBS values between the enamel and dentin surfaces. In self-etching adhesive condition, the three groups (BK, VF and BF) were compared using one-way ANOVA, while in no adhesive condition, the two groups (BK and VF) were compared using an independent two-sample t-test. Following the comparison of the three groups using one-way ANOVA, post-hoc tests (Bonferroni Correction) were used to determine significant differences between each pair of groups. Additionally, when all conditions were the same except for one, independent t-tests were used to compare the two groups. The interaction between restorative materials and conditions (tooth type, adhesive, thermocycling) on the mean μ-SBS values was evaluated using two-way ANOVA. Statistical significance was set at $p = 0.05$.

III. Results

1. Micro-shear bond strength

Means and standard deviations (MPa) of the μ-SBS of each group are presented in Table 2 and 3.

No statistically significant differences were observed between the restorative materials in any condition. Across all conditions, the μ-SBS of the groups that applied self-etching adhesive were significantly higher than no adhesive groups. The μ-SBS in enamel significantly decreased after thermocycling except for self-etching adhesive in BK. In dentin, μ-SBS significantly decreased after thermocycling in VF with self-etching adhesive group.

Table 3. The mean ± standard deviation of micro-SBS (MPa) of the tested materials in enamel.

Material		Self-etching adhesive	*p*-value	No adhesive	*p*-value
BK	24h	$19.51\pm4.48^{A++}$	0.2419	$15.78\pm4.44^{A+}$	<.0001*
	Thermocycling	$17.10\pm7.60^{a++}$		$9.62\pm4.89^{a+}$	
VF	24h	$20.63\pm4.42^{A++}$	0.0015*	$15.47\pm4.47^{A+}$	<.0001*
	Thermocycling	$15.48\pm4.49^{a++}$		$7.71\pm4.70^{a+}$	
BF	24h	21.78 ± 3.80^{A}	0.0298*	-	-
	Thermocycling	18.34 ± 5.18^{a}		-	

Same superscript uppercase letters vertically indicate that average μ-SBS was not significantly different among the materials after 24h.

Same superscript lowercase letters vertically indicate that average μ-SBS was not significantly different among the materials after thermocycling.

Statistical significance according to thermocycling is indicated by * (*p*-value).

Statistical significance according to use of adhesive system is indicated as +, ++

Table 4. The mean ± standard deviation of micro-SBS (MPa) of the tested materials in dentin.

Material		Self-etching adhesive	*p*-value	No adhesive	*p*-value
BK	24h	19.32±5.50^{A++}	0.5746	10.04±3.39^{A+}	0.7812
	Thermocycling	20.57±7.46^{a++}		9.67±4.37^{a+}	
VF	24h	22.40±3.29^{A++}	0.0081*	10.01±2.57^{A+}	0.2640
	Thermocycling	18.20±5.36^{a++}		8.65±4.21^{a+}	
BF	24h	21.38±4.90^{A}	0.5548	-	-
	Thermocycling	20.38±5.29^{a}		-	

Same superscript uppercase letters vertically indicate that average μ-SBS was not significantly different among the materials after 24h.

Same superscript lowercase letters vertically indicate that average μ-SBS was not significantly different among the materials after thermocycling.

Statistical significance according to thermocycling is indicated by * (*p*-value).

Statistical significance according to use of adhesive system is indicated as +, ++

2. Failure mode analysis

The distribution of failure modes for each group is shown in Fig. 2 and 3. In self-etching adhesive group, adhesive failure was absent, and mixed failure was predominant. However, in no adhesive group, adhesive failure and mixed failure were observed at similar level, whereas cohesive failure did not occur. Representative SEM images of enamel and dentin surfaces are shown in Fig. 4 and 5.

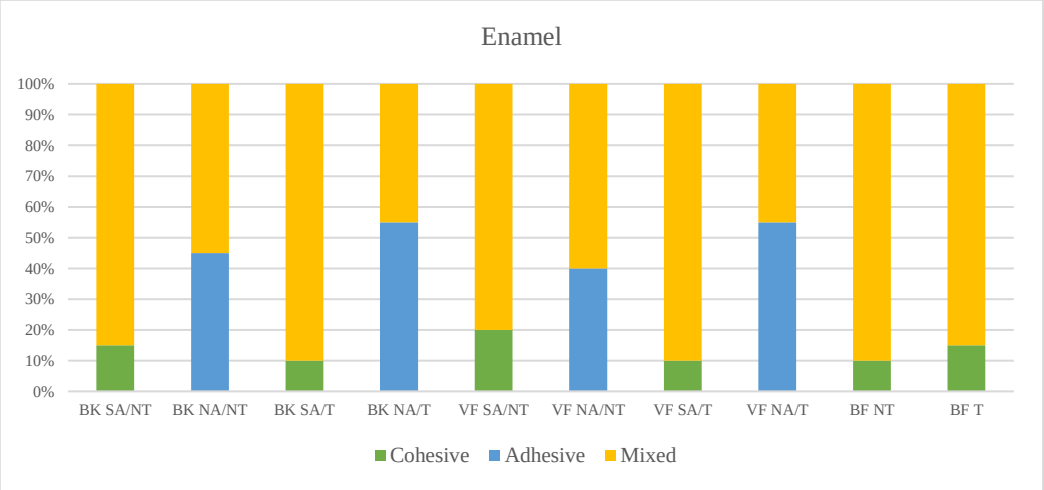

Fig. 2. Failure mode distribution (%) of the tested groups after micro-SBS test in enamel.

NT: Non thermocycling; T: Thermocycling; SA: Self-etching adhesive; NA: Non adhesive

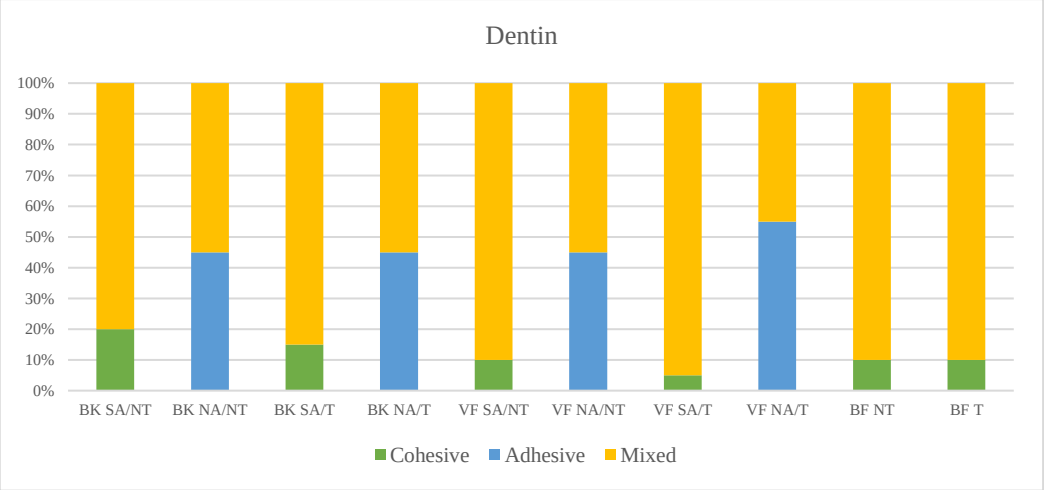

Fig. 3. Failure mode distribution (%) of the tested groups after micro-SBS test in dentin.

NT: Non thermocycling; T: Thermocycling; SA: Self-etching adhesive; NA: Non adhesive

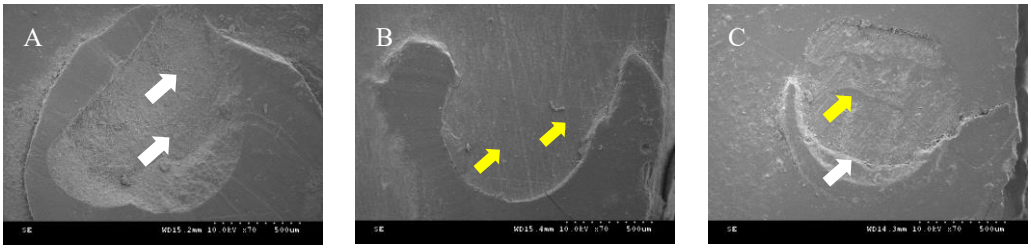

Fig. 4. Representative SEM images in enamel showing A) cohesive failure in the restorative material, B) adhesive failure at the tooth/material interface, and C) mixed failure in the restorative material and tooth surface. Yellow arrows indicate tooth substrate, whereas white arrows indicate restorative material.

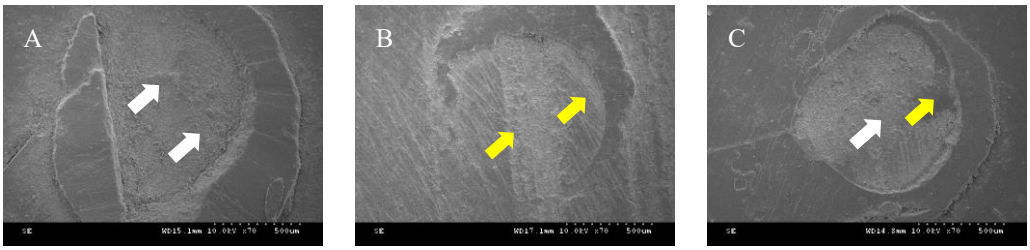

Fig. 5. Representative SEM images in dentin showing A) cohesive failure in the restorative material, B) adhesive failure at the tooth/material interface, and C) mixed failure in the restorative material and tooth surface. Yellow arrows indicate tooth substrate, whereas white arrows indicate restorative material.

IV. Discussion

Current SAC bond to tooth substrates less effectively than conventional flowable composites used with total-etching or self-etching adhesives, according to several studies (26-30). However currently, very few investigations regarding the bonding performance of SAG have been carried out. Following the results of this study, the μ-SBS of SAG was not affected by the type of restorative material, but there were significant differences depending on the use of adhesive and thermocycling, so the null hypothesis was partially rejected.

This study used the universal adhesive in self-etching mode since it is commonly used and has the simplest step for bonding.

In this study, adhesive systems and self-adhesive material were used together because previous studies showed that the poor bonding performance of self-adhesive materials to enamel and dentin and the combination of self-adhesive material and adhesive systems significantly improved bonding to tooth substrate (10, 24, 25, 31, 32).

VF is a novel and pioneering composite material that does not require traditional etching and bonding since it contains a functional GPDM monomer. VF was used to compare the bond strength of the new SAG with that of SAC. BF is a nanohybrid flowable giomer that combines the strength, durability, and aesthetic properties of hybrid composites with the delivery of flowable. BF was used in this study to compare the μ-SBS of SAG with a giomer without self-adhesive mode.

SAC and SAG do not require a separate clinical step of applying an adhesive system. Phosphonic acid monomers, found in BK, are hydrolytically stable functional monomers and are also utilized in self-etching primers and other materials (17, 33). The ionized phosphonic acid interacts with the calcium ions (Ca^{2+}) of hydroxyapatite in the tooth, creating a stable ionic link (34). During this

process, as substrate demineralization and resin penetration occur simultaneously, it is recommended to wait for 20 s before light-curing. When applied with self-adhesive materials without an adhesive system, as recommended by the manufacturer, it showed significantly low μ-SBS values with enamel and dentin. The findings of this study coincide with those of previous studies that assessed the bond strength between SAC and tooth substrate and reported low values (4, 16, 28).

In fact, compared to other self-adhesive materials such as resin cement or adhesive systems, SAC have a lower content of functional acidic monomers and that is the main reason for its poor bond strength. SAC only interact with the tooth structure superficially and do not sufficiently dissolve the smear layer and penetrate the tooth substrate (35). In addition, the hydrophilic monomer HEMA, one of the components of BK, improves the wettability of the dentin surface, but may reduce bond strength by increasing moisture absorption during photo-polymerization and after thermocycling (36).

Another possible reason for the low μ-SBS values observed in the self-adhesive materials could be their increased viscosity and decreased wettability compared to independent adhesive systems. Consequently, it is difficult to achieve adequate bonding effectiveness and micromechanical interlocking with the tooth structure because the self-adhesive materials cannot fully penetrate the space between collagen fibers or the dentinal tubules (10).

The results of this study suggest that using self-etching adhesive systems increases the bond strength of self-adhesive materials. The high wettability and low viscosity of the self-etching adhesive system may have enhanced the interaction between calcium ions and acidic monomers, resulting in improved bond strength (37). Although this study showed that the bonding strength significantly increased with the application of the adhesive, the bonding strength of the self-adhesive material can be affected by the application of phosphoric acid etching. Norah Sibai et al. (38) studied the bonding strength of SAC in enamel and dentin with or without phosphoric acid etching. The

results showed that the acid etching group had higher bonding strength than the without acid etching group, but significant differences were seen only in dentin. To further investigate whether the 3-step etch-and-rinse adhesive system with acid etching and the 2-step self-etch adhesive system without acid etching affect the bond strength of SAG, Polyxeni Papazekou et al. (9) compared the bond strength of SAG when applying a 3-step etch-and-rinse adhesive system, a 2-step self-etch adhesive system, and no adhesive. The results showed no significant difference in bond strength between the 3-step and 2-step adhesive systems. However, the group without adhesive exhibited a significantly reduced bond strength.

It is difficult for the tooth-restoration interface to survive in the oral cavity over time due to changes in temperature, chewing loads, and chemical attacks (39). Long-term clinical bonding performance is more likely to be represented by an artificial aging process. In this study, as recommended by the International Organization for Standardization (ISO), aging by thermocycling was used (40). Thermocycling was performed for 10,000 cycles, which corresponds to almost one year under oral conditions (41). In the current study, BK and BF were found to be negligibly affected by thermocycling, except for self-etching adhesive on enamel and no adhesive on enamel, respectively. In particular, thermocycling had no significant effect on the self-etching adhesive group of BK on both enamel and dentin. This suggests that applying adhesives can help improve the durability of restorations for a similar reason to the increase in bond strength. However, VF was affected by thermocycling except for no adhesive on dentin. Therefore, the hypothesis that thermocycling would not affect μ-SBS on enamel and dentin was rejected. Few studies have included data on both immediate and long-term bond strength tests of SAC (26, 27, 42), and this study showed that SAC has lower bond strength stability in enamel and dentin compared to SAG. It is possible that SAC and SAG have different interactions with dental hard tissue after thermocycling, probably due to their different compositions and functional monomers. VF contains GPDM

monomers and due to its high hydrophilicity and relatively short spacer chain, it provides improved dentin wettability and a strong etching effect. However, its chemical bonding potential with hydroxyapatite may be lower than with other self-adhesive monomers (43).

In self-etching adhesive group, mixed failure was the predominant failure mode, followed by cohesive failure within the restoration materials at both time points. However, in no adhesive group, adhesive failure and mixed failure showed similar failure rates at both time points. This suggests inadequate adhesive performance of the self-adhesive materials in no adhesive mode and adequate shear force distribution at the composite-tooth interface when using self-etching adhesive (44).

In this study, micro-shear bond strength was measured. Conventional (macro) shear bond strength tests have shown, through studies using finite element analysis, that they result in non-uniform and heterogeneous stress patterns (45). To overcome these limitations, a new method called the micro-shear bond strength test was introduced. The micro-shear bond strength test features a small bonding area specimen of less than 1 mm^2 (46). And in this study, wire loops were used for the micro-shear bond strength test. There are concerns about significant stress concentrations in the load application area when using knife-edge chisels. On the other hand, wire loops show better stress distribution at the edges of the joint area (47). This is supported by another study showing lower bond strength of knife-edge chisels compared to other shear loading methods, such as wire loops or flat rods (46). However, even when using wire loops, there is a limitation in achieving accurate loading from the interface (48).

There are some clinical indications for SAG in enamel and dentin, despite their low μ-SBS values. More precisely, it can be suggested for pit and fissure sealants, small cavities, and restorations of small, narrow cavities (25, 49). One major benefit of using SAG is that it can be in direct contact with the tooth structure without the requirement for an intermediate bonding layer. It is believed that the release of fluoride and other ions helps to prevent teeth from demineralization.

The limitation of this study is that it was carried out in a laboratory environment. Therefore, it is important to remember that the tooth substrate was ideally prepared for the formation of smear layers and adhesive procedures by using silicon carbide sandpapers. In addition, repeated thermocycling was used to artificially age the specimens, but no static or cyclic loads were applied to simulate the conditions of the oral cavity. Since the temperature, moisture, pH, and occlusal forces of the oral environment are dynamic, it is impossible to completely duplicate these conditions in a laboratory. Additional research is required to validate the clinical efficacy of SAG. Furthermore, other factors like wear rate, water sorption, and solubility must be considered as well as bonding effectiveness when determining the stability and success of a restoration. If better ion release capacity of SAG is validated by further research, it may help with resistance to demineralization and promotion of remineralization, which would be an advantage for restorative materials and promote clinical application of SAG. SAG is the future direction of dental adhesive, thus it is crucial to research the available materials and identify concerns that may be resolved to enhance the mechanism and effectiveness of bonding to dental tissues. The results of this study highlight the necessity of enhancing the bonding performance of SAG through compositional modifications or the use of adhesive systems to demonstrate long-term clinical survival.

V. Conclusion

According to the recommendation of manufacturer, the self-adhesive material should be used without adhesive system. However, the results of this study showed that the additional application of bonding to the dental substrate of self-adhesive material can improve bond strength compared to no adhesive groups. There were no statistically significant differences observed among the restorative materials in any condition. BK and BF were found to be negligibly affected by thermocycling, but VF was affected by thermocycling. This study showed that SAC has lower bond strength stability in enamel and dentin compared to SAG. Due to the lack of reliable clinical studies, careful selection of SAG is required until their bonding stability to tooth substrates and long-term clinical performance have been proven.

Reference

1. Bayne SC, Thompson JY, Swift Jr EJ, Stamatiades P, Wilkerson M. A characterization of first-generation flowable composites. The Journal of the American Dental Association. 1998;129(5):567-577.

2. Labella R, Lambrechts P, Van Meerbeek B, Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. Dental materials. 1999;15(2):128-137.

3. Baroudi K, Rodrigues JC. Flowable resin composites: a systematic review and clinical considerations. Journal of clinical and diagnostic research: JCDR. 2015;9(6):ZE18-ZE24.

4. Beauchamp J, Caufield PW, Crall JJ, Donly K, Feigal R, Gooch B, et al. Evidence-based clinical recommendations for the use of pit-and-fissure sealants: a report of the American Dental Association Council on Scientific Affairs. The Journal of the American Dental Association. 2008;139(3):257-268.

5. Kasraei S, Azarsina M, Majidi S. In vitro comparison of microleakage of posterior resin composites with and without liner using two-step etch-and-rinse and self-etch dentin adhesive systems. Operative dentistry. 2011;36(2):213-221.

6. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. Journal of dental research. 1955;34(6):849-853.

7. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, et al. Relationship between bond-strength tests and clinical outcomes. Dental materials. 2010;26(2):e100-e121.

8. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt K. State of the art of self-etch adhesives. Dental materials. 2011;27(1):17-28.

9. Papazekou P, Dionysopoulos D, Papadopoulos C, Mourouzis P, Tolidis K. Evaluation of micro-

shear bond strength of a self-adhesive flowable giomer to dentin in different adhesive modes. International Journal of Adhesion and Adhesives. 2022;118:1-9.

10. Kim G, Lee J, Kim H, Park H. Evaluation of Shear Bond Strength and Microleakage of Self-adhesive Giomer. The Journal of the Korean Academy of Pedtatric Dentistry. 2023;50(4):434-442.

11. Ferracane JL. Resin composite—state of the art. Dental materials. 2011;27(1):29-38.

12. Hanabusa M, Mine A, Kuboki T, Momoi Y, Van Landuyt KL, Van Meerbeek B, et al. TEM interfacial characterization of an experimental self-adhesive filling material bonded to enamel/dentin. Dental Materials. 2011;27(8):818-824.

13. Yoshioka M, Yoshida Y, Inoue S, Lambrechts P, Vanherle G, Nomura Y, et al. Adhesion/decalcification mechanisms of acid interactions with human hard tissues. Journal of Biomedical Materials Research: An Official Journal of The Society for Biomaterials and The Japanese Society for Biomaterials. 2002;59(1):56-62.

14. Kerr Dental Corporation. Vertise Flow: Product Brochure. Orange CA: Kerr Dental Corporation; 2010.

15. Pentron Clinical Technologies L. Fusio Liquid Dentin: Product Brochure. Wallingford CT: Pentron Clinical Technologies; 2009.

16. David C, Cardoso de Cardoso G, Isolan CP, Piva E, Moraes RR, Cuevas-Suarez CE. Bond strength of self-adhesive flowable composite resins to dental tissues: A systematic review and meta-analysis of in vitro studies. J Prosthet Dent. 2022;128(5):876-885.

17. Ikemura K, Tay F, Kouro Y, Endo T, Yoshiyama M, Miyai K, et al. Optimizing filler content in an adhesive system containing pre-reacted glass-ionomer fillers. Dental Materials. 2003;19(2):137-146.

18. Fujimoto Y, Iwasa M, Murayama R, Miyazaki M, Nagafuji A, Nakatsuka T. Detection of ions released from S-PRG fillers and their modulation effect. Dental materials journal. 2010;29(4):392-

397.

19. Hotta M, Morikawa T, Tamura D, Kusakabe S. Adherence of Streptococcus sanguinis and Streptococcus mutans to salivacoated S-PRG resin blocks. Dental Materials Journal. 2014;33(2):261-267.

20. Ma S, Imazato S, Chen J-H, Mayanagi G, Takahashi N, Ishimoto T, et al. Effects of a coating resin containing S-PRG filler to prevent demineralization of root surfaces. Dental materials journal. 2012;31(6):909-915.

21. Nomura R, Morita Y, Matayoshi S, Nakano K. Inhibitory effect of surface pre-reacted glass-ionomer (S-PRG) eluate against adhesion and colonization by Streptococcus mutans. Scientific reports. 2018;8(1):1-18.

22. Saku S, Kotake H, Scougall-Vilchis RJ, Ohashi S, Hotta M, Horiuchi S, et al. Antibacterial activity of composite resin with glass-ionomer filler particles. Dental materials journal. 2010;29(2):193-198.

23. Ikemura K, Tay FR, Nishiyama N, Pashley DH, Endo T. Design of new phosphonic acid monomers for dental adhesives-synthesis of (Meth) acryloxyalkyl 3-phosphonopropionates and evaluation of their adhesion-promoting functions. Dental materials journal. 2006;25(3):566-575.

24. Shimizu S, Kotake H, Takagaki T, Shinno K, Miyata S, Burrow MF, et al. Evaluation of bonding performance and multi-ion release of S-PRG fillercontaining self-adhesive resin composite. Dental Materials Journal. 2021;40(5):1257-1263.

25. Takamiya H, Tsujimoto A, Teixeira EC, Jurado CA, Takamizawa T, Barkmeier WW, et al. Bonding and wear properties of self-adhesive flowable restorative materials. European journal of oral sciences. 2021;129(5):1-12.

26. Brueckner C, Schneider H, Haak R. Shear bond strength and tooth-composite interaction with self-adhering flowable composites. Operative dentistry. 2017;42(1):90-100.

27. Bumrungruan C, Sakoolnamarka R. Microshear bond strength to dentin of self-adhesive flowable composite compared with total-etch and all-in-one adhesives. Journal of Dental Sciences. 2016;11(4):449-456.

28. Peterson J, Rizk M, Hoch M, Wiegand A. Bonding performance of self-adhesive flowable composites to enamel, dentin and a nano-hybrid composite. Odontology. 2018;106:171-180.

29. Poitevin A, De Munck J, Van Ende A, Suyama Y, Mine A, Peumans M, et al. Bonding effectiveness of self-adhesive composites to dentin and enamel. Dental Materials. 2013;29(2):221-230.

30. Yuan H, Li M, Guo B, Gao Y, Liu H, Li J. Evaluation of Microtensile Bond Strength and Microleakage of a Self-adhering Flowable Composite. Journal of Adhesive Dentistry. 2015;17(6):535-543.

31. Ozel Bektas O, Eren D, Akin EG, Akin H. Evaluation of a self-adhering flowable composite in terms of micro-shear bond strength and microleakage. Acta Odontologica Scandinavica. 2013;71(3-4):541-546.

32. Tuloglu N, Tunc ES, Ozer S, Bayrak S. Shear bond strength of self-adhering flowable composite on dentin with and without application of an adhesive system. Journal of applied biomaterials & functional materials. 2014;12(2):97-101.

33. Yao C, Ahmed MH, Zhang F, Mercelis B, Van Landuyt KL, Huang C, et al. Structural/chemical characterization and bond strength of a new self-adhesive bulk-fill restorative. Journal of Adhesive Dentistry. 2020;22(1):85-97.

34. Ikemura K, Endo T, Kadoma Y. A review of the developments of multi-purpose primers and adhesives comprising novel dithiooctanoate monomers and phosphonic acid monomers. Dental materials journal. 2012;31(1):1-25.

35. Goracci C, Cury AH, Cantoro A, Papacchini F, Tay FR, Ferrari M. Microtensile bond strength

and interfacial properties of self-etching and self-adhesive resin cements used to lute composite onlays under different seating forces. Journal of Adhesive Dentistry. 2006;8(5):327-335.

36. Wei Y-j, Silikas N, Zhang Z-t, Watts DC. Hygroscopic dimensional changes of self-adhering and new resin-matrix composites during water sorption/desorption cycles. Dental Materials. 2011;27(3):259-266.

37. Memarpour M, Shafiei F, Razmjoei F, Kianimanesh N. Effect of laser preparation on adhesion of a self-adhesive flowable composite resin to primary teeth. Microscopy research and technique. 2016;79(4):334-341.

38. Sibai N, El Mourad A, Al Suhaibani N, Al Ahmadi R, Al Dosary S. Shear bond strength of self-adhesive flowable resin composite. International Journal of Dentistry. 2022;1-8.

39. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Adhesion to enamel and dentin: current status and future challenges. Operative Dentistry. 2003;28(3):215-235.

40. International Organization for Standardization. TS 11405: Dental materials—testing of adhesion to tooth structure. Geneva, Switzerland: International Organization for Standardization ISO Central Secretariat. 2003.

41. Gale M, Darvell B. Thermal cycling procedures for laboratory testing of dental restorations. Journal of dentistry. 1999;27(2):89-99.

42. Goracci C, Margvelashvili M, Giovannetti A, Vichi A, Ferrari M. Shear bond strength of orthodontic brackets bonded with a new self-adhering flowable resin composite. Clinical Oral Investigations. 2013;17:609-617.

43. Wang R, Shi Y, Li T, Pan Y, Cui Y, Xia W. Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin. Journal of dentistry. 2017;62:72-80.

44. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different

test methods: a critical literature review. Dental materials. 2010;26(2):e78-e93.

45. Soares CJ, Santana FR, Castro CG, Santos-Filho PC, Soares PV, Qian F, et al. Finite element analysis and bond strength of a glass post to intraradicular dentin: comparison between microtensile and push-out tests. Dental Materials. 2008;24(10):1405-1411.

46. Braga RR, Meira JB, Boaro LC, Xavier TA. Adhesion to tooth structure: a critical review of "macro" test methods. Dental Materials. 2010;26(2):e38-e49.

47. DeHoff PH, Anusavice KJ, Wang Z. Three-dimensional finite element analysis of the shear bond test. Dental Materials. 1995;11(2):126-131.

48. Cheetham JJ, Palamara JE, Tyas MJ, Burrow MF. A comparison of the micro-shear bond strength and failure mode of non-enclosed and mold-enclosed luting cements bonded to metal. Dental materials journal. 2013;32(6):896-905.

49. David C, de Cardoso GC, Isolan CP, Piva E, Moraes RR, Cuevas-Suarez CE. Bond strength of self-adhesive flowable composite resins to dental tissues: A systematic review and meta-analysis of in vitro studies. The Journal of Prosthetic Dentistry. 2022;128(5):876-885.

Abstract (In Korean)

우치에 대한 자가 접착성 자이오머의 미세 전단 결합 강도 평가

김 은 진

연세대학교 대학원

치의학과

(지도교수 박 정 원)

자가 접착성 유동성 자이오머 (SAG)는 최근 진료 과정을 단순화하고 진료 시간을 단축하기 위해 도입되었지만, 법랑질과 상아질에 대한 결합 성능에 대한 정보는 적다. 이 연구의 목적은 열순환 전후에 법랑질과 상아질에 다양한 방식으로 결합된 SAG 의 미세전단결합강도를 평가하는 것이었다.

치아 시편을 위해 건전한 소 치아가 사용되었다. 미세전단결합강도 시험을 위해 법랑질 및 상아질 시편에 SAG (Beautifil Kids SA – BK), 자가 접착성 유동성 복합레진 (Vertise Flow – VF) 및 나노하이브리드 유동성 자이오머 (Beautifil FlowPlus F03 – BF)를 적용했다. BK, VF 에는 두 가지 (자가 부식 접착제 포함, 접착제 없음) 접착모드가, BF 에 대해서는 한 가지 (자가 부식 접착제 포함) 접착

모드가 수행되었다. 미세전단결합강도 시험은 24 시간 후, 열순환 후 만능 시험기를 사용하여 측정되었다.

자가 부식 접착제를 포함한 접착모드에서는 모든 재료에 대해 접착제가 없는 접착모드에 비해 상당히 높은 미세전단결합강도를 보였다 ($p < 0.05$). 열순환은 BK 의 미세전단결합강도에 큰 영향을 미치지 않았다. 자가 부식 접착제를 포함한 군에서는 모든 재료에서 혼합 실패가 우세했으나 접착제가 없는 군에서는 모든 재료에서 접착 실패와 혼합 실패가 유사한 수준으로 관찰되었다.

접착제가 없는 접착모드에서 자가접착 재료의 치아에 대한 접착 성능은 기존 접착제에 비해 상당히 약했다. 어떤 조건에서도 수복 재료 간에 통계적으로 유의미한 차이는 관찰되지 않았다. BK, BF 는 열순환의 영향을 무시할 수 있는 것으로 나타났으나, VF 는 열순환의 영향을 받았다. 따라서 본 연구에서는 자가 접착성 유동성 복합레진이 자가 접착성 유동성 자이오머에 비해 법랑질과 상아질의 결합 강도 안정성이 낮다는 것을 보여주었다.

핵심되는 단어: 미세 전단 결합 강도; 자가접착성 자이오머; 표면 사전 반응 글라스 아이오노머 필러 (S-PRG)