# Bioactive Restorative Systems and Self-Adhesive Bonding: Assessing Long-Term Durability and Effectiveness in Dentin Adhesion

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#### Abstract

Background: Bioactive, ion-releasing restorative materials have emerged as a promising solution for improving dentin adhesion and longterm restoration durability. Self-adhesive materials simplify bonding procedures by eliminating the need for pre-treatment adhesives, potentially enhancing clinical outcomes. This study aimed to evaluate the microshear bond strength (uSBS) and interfacial micromorphology of contemporary bioactive restorative systems bonded to dentin. Methods: Eighty freshly extracted human molars were prepared and divided into four groups based on restorative material: Surefil One (self-adhesive bulk-fill composite), Cention Forte (alkasite-based material) with and without primer, and Fuji II LC (resin-modified glass ionomer, control). Specimens underwent immediate (24-hour) and delayed (6-month artificial saliva storage)  $\mu SBS$  testing using a universal testing machine. Failure modes were analyzed with stereomicroscopy, and scanning electron microscopy (SEM) was used to assess the restoration-dentin interface. Results: Cention Forte with primer exhibited the highest immediate and delayed  $\mu SBS$  values, significantly outperforming all other materials (p < 0.05). Surefil One and Cention Forte without primer showed the weakest bond strength, with a high incidence of adhesive failure and pre-test failures after aging. SEM analysis revealed superior interfacial adaptation in Cention Forte with primer and Fuji II LC, while Surefil One and Cention Forte without primer demonstrated poor dentin infiltration, interfacial gaps, and lack of resin tag formation. Conclusion: The results indicate that the use of a primer significantly enhances the bonding performance of alkasite-based materials, making them a viable alternative to resin-modified glass ionomers. However, self-adhesive bulk-fill composites and alkasite-based materials without primer exhibit weak adhesion to dentin, limiting their clinical applicability. Further research is needed to optimize self-adhesive restorative materials for improved long-term bonding efficacy.

#### Introduction

The use of bioactive, ion-releasing restorative materials offers potential solutions to many challenges related to bonding to caries-affected dentin (CAD). These materials can establish a durable bond between restorative substances and CAD tooth structures, which is vital for the long-term success of dental restorations (1). Traditionally, bonding restorations has relied on dental adhesives, but self-adhesive restorative materials have emerged as a significant advancement in direct restorations (1). These materials provide simpler application techniques and may overcome issues associated with complicated adhesive procedures (2). Additionally, they reduce chair-side time and minimize iatrogenic errors by eliminating the need for pre-treatment adhesive steps (3,4).

The current philosophy of minimally invasive dentistry emphasizes the use of bioactive, ion-releasing restorative materials. These materials are capable of promoting biomineralization by releasing essential ions in a strategic manner (5). Bioactive materials offer significant advantages in restorative dentistry, including improved restoration longevity, stimulation of dentin repair mechanisms, and enhanced interfacial adhesion. Consequently, the occurrence of recurrent caries and marginal leakage is greatly minimized (6).

Among dental restorative materials, glass ionomer cements (GICs) hold a prominent position due to their inherent bioactivity (7). This distinctive characteristic enables direct interaction with hard tooth tissues through a self-adhesive mechanism (8). GICs were once considered the gold standard for self-adhesive direct restorations (9). Developed in the late 1960s, conventional glass ionomer cements were based on silicon and polycarboxylic cements (5). To address the shortcomings of conventional GICs, such as prolonged setting times, weak mechanical properties, and moisture sensitivity, resin-modified GICs were introduced. These incorporated 2-hydroxyethylmethacrylate (HEMA) into the original formula to improve color stability and adhesion to tooth structures (9). Both conventional and resin-modified GICs bond to

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tooth structures through shallow hybridization (micromechanical interlocking) and ionic bonding between the carboxyl groups of polyalkenoic acid and calcium ions from hydroxyapatites (10).

Composite resins, due to their esthetic and mechanical properties, are in high demand for routine dental practice. Composite resins have been in use for about 50 years (11) but come with certain limitations, such as plaque accumulation and bacterial growth, which can increase the likelihood of recurrent caries (12).

The development of innovative hybrid materials, such as 'self-adhesive', 'bulk-fill', or 'ion-releasing' restorative materials, marks a significant step forward in minimally invasive restorative dentistry. These materials eliminate the need for separate adhesives, reducing contamination risks from blood or saliva. They also address potential problems with adhesives, like post-operative sensitivity. The bulk-fill concept is another simplification, and many of these materials exhibit ion-releasing properties. Several studies (13,14) have demonstrated the benefits of ion-release capabilities in remineralizing and preventing dental caries.

A recent breakthrough in dental restorative materials is the introduction of self-adhesive resinous composites that offer fluoride-releasing and "bulk-fill" properties. These materials, classified as "bioactive" or "smart", have a distinct chemical composition compared to traditional glass ionomer cements (GICs). Early investigations suggest that these materials may surpass GICs in performance (9).

Alkasite-based materials represent a new category of restorative materials and aim to create potentially bioactive materials (5). These materials are described as hybrid tooth-colored restoratives that release calcium, fluoride, and hydroxyl ions, demonstrating strong anti-cariogenic properties. Alkasite-based materials combine favorable features of GICs and resin-based composites. They also offer dual-cure functionality, allowing for bulk placement with or without an adhesive layer (15). The similarity to conventional resin-based composites lies in the monomer matrix and certain inorganic fillers (16). Alkasite materials contain alkaline fillers (SiO2-CaO-CaF2-Na2O glass; 24.6 wt%) that release acid-neutralizing ions to prevent tooth demineralization (16). These fillers are the source of the name "alkasite," as designated by the manufacturer (5).

Furthermore, self-adhesive hybrid composites were introduced in 2019 as advanced self-adhesive restorative materials (ASAR). These materials combine ion-releasing (18) and self-adhesive properties of GICs within a resinous structure. The unique MOPOS (Modified Polyacid System) monomer promotes adhesion and strengthens the material, while BADEP (bifunctional acrylates) facilitates cross-linking and covalent bond formation. These self-adhesive hybrid composites have mechanical properties similar to established posterior restorative materials, such as flexural strength, fatigue strength, flexural modulus, and fracture toughness. They also show comparable wear resistance to resin-based composites (17). The manufacturer claims that these composites release calcium, aluminum, and fluoride ions, exhibiting long-term fluoride release comparable to that of GICs and resin-modified GICs (even after 450 days) (5).

Numerous studies have shown that alkasite-based materials exhibit superior compressive, tensile, and shear bond strengths compared to GICs (19,20,21,22). Manufacturers position these materials as comparable to amalgam in terms of compressive strength and durability, while offering ion-releasing properties similar to those of GICs (21). The enhanced translucency of alkasite materials is also noted as an aesthetic advantage over GICs (23). However, there remains a lack of direct comparisons between alkasite restorations and other modern esthetic restorative materials.

Recent investigations into a novel self-adhesive hybrid composite material for posterior restorations have demonstrated mechanical properties comparable to clinically established materials, including flexural strength, fatigue strength, flexural modulus, and fracture toughness. These materials also exhibit wear resistance on par with resin-based composites (17). The self-adhesive properties of these materials to enamel and dentin are comparable to those of GICs and modern adhesives (24,25). However, further research is required to definitively assess the bond strength of this new bulk-fill restorative material (Supplementary Table S1).

The rapid development of novel dental materials presents a challenge for clinicians aiming to optimize patient outcomes (26). A notable gap exists in the current research regarding the bond strength of bioactive restorative materials. To address this knowledge gap and support clinical decision-making, a carefully designed laboratory study was conducted to evaluate the performance of contemporary materials (alkasite and self-adhesive bulk-fill hybrid composite) in comparison with resin-modified GICs.

A laboratory study found no significant difference in the dentin shear bond strength between self-adhesive bulk-fill composites and resin-modified GICs (1). However, other studies (9,10) have reported differences in clinical and laboratory performance between self-adhesive restorative materials. One laboratory study (9) revealed differences in shear bond strength and interfacial surface formation between self-adhesive bulk-fill composites, alkasite, and HV-GICs.

A key question this study aimed to answer was whether these self-adhesive restorative materials are sufficiently effective for bonding to dentin. To evaluate the effectiveness of self-adhering restorative materials, both immediate and long-term bonding efficacy to dentin were assessed. Scanning electron microscopy (SEM) is commonly used for morphological analysis of adhesive-dentin interfaces. Several studies have explored the correlation between bonding performance and interfacial characteristics, including hybrid layer formation, thickness, integrity, and resin tag morphology (27,28). SEM is thus employed in this study to provide insights into the adhesive interface and ensure optimal bonding for durable dental restorations (29).

This study aimed to compare the bond durability, microshear bond strength, and interfacial micromorphology of various self-adhesive bioactive restorative systems bonded to dentin. The null hypothesis was that there would be no significant difference in these properties across the tested materials.

#### Materials and Methods

The materials used in the current study are detailed in Table 1.

Materials	Specification	Manufacturer	Composition	Application	Code batch
					no. (lot)
Surefil one	Self-adhesive bulk-fill resinous restorative material with ionic release after polymerization No bonding agent required regardless of cavity	Dentsply Sirona, Konstanz, Germany	Aluminum-phosphor- strontium-sodium-fluoro- silicate glass, highly dispersed silicon dioxide, ytterbium fluoride, iron oxide pigments, titanium dioxide pigments ,polycarboxylic acid, acrylic acid, bifunctional acrylate, water, self-cure initiator, camphorquinone, stabilizer	1. Activate the capsule 2. Mix it with an amalgamator for 10s 3. Injected directly by capsule applier 4. Light cure for 20s with an output of 1200 mW/cm2 $\rightarrow$ Self-cure for 6 min (prior to further specimen processing)	('SU-O') 2,201,000,713
Cention forte	"Alkasite" bulk-fill resinous restorative material with ionic release after polymerization under acidic challenge	Ivoclar Vivadent; Schaan, Liechtenstein	Barium aluminum silicate glass, ytterbium trifluoride, pre-polymerized filler, calcium barium aluminum fluorosilicate glass, and calcium fluoro-silicate glass,UDMA, tricyclodecan-dimethanol dimethacrylate, tetramethyl-xylylene diurethane dimethacrylate,	<ol> <li>Actively scrub and agitate the primer for 10s</li> <li>Dry with compressed air until a glossy thin immobile</li> </ol>	('CNF') ZL08SV

Table 1 Materials used in this study

			DO	1: <u>https://doi.org/10</u>	<u>).62/54/j0e.v318.6338</u>
	Bonding agent		polyethylene glycol, 400	layer	
	(Cention		dimethacrylate, and	remains	
	primer) not		Ivocerin	3. Activate	
	required for			the cansule	
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	required for a			amalgamator	
	non-retentive			5. Extrude	
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				capsule	
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				Cliffe	
				cure,	
				optionally	
				speed up the	
				process by	
				light cure for	
				15s	
Fuji II LC	Resin-	GC;	Fluoro-alumino-silicate	1.Apply	('FJI')
,	modified olass-	Tokvo	glass.	Dentin	2.302.132
	ionomer	Ianan	Polybasic carboyylic acid	Conditioner	_,~~_, ~~
	Solf adhesing	Japan	LIDMA	for 20 a	
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	material with			3. Mix it with	
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	cavity			ancedy by	
				capsule	
				applier	
				5. Light cure	
				for layers of	
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Cention	л two-	ivociar	Liquid: Disphenol A	1.Applied to	$(\mathbf{p})$
primer	component	Vivadent,	glycerolate dimethacrylate,	the dentin	Z031Z2
	self etching	Schaan,	2-hydroxyethyl	surface with	
	and self-curing	Liechtenstein	methacrylate,	a single-use	
	primer		methacrylated phosphoric	applicator.	
	-		acid, 1,10-decandiol	2.Coating	
			dimethacrylate	and	
			methacrylate modified	scrubbing	
			polyacrylic acid 2	for 10 c	
			poryacryne aciu, 2-	2 TL -	
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			methacrylate, ethanol,	primer was	
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				with	
				compressed	
				air until a	
				thin and	

	DOI: https://doi.org/10.02/04/j0c.voi0.000					
				shiny film		
				had formed		
Dentin	Polyacrylic	GC;	(GC; 20% polyacrylic acid,	1.Spplied for	('DC')	
conditioner	acid-etch	Tokyo,	3% aluminum chloride,	20 s to the	5,040,518	
		Japan	distilled water)	dentin		
				surface of		
				the		
				specimen.		
				2.Rinse		
				thoroughly		
				with water		
				and dry		
				gently		

Eighty freshly extracted human permanent molars, free from caries and restorations, were obtained from patients undergoing extractions due to periodontal disease. The teeth were thoroughly cleaned with a hand scaler (Nordent, Ivory #2e3, USA) to remove calculus and soft tissue deposits, followed by polishing with a rubber cup (prophy rubber polishing cup, China) and fine pumice water slurry (PSP, Dylan Rd, Belvedere, England). The teeth were then stored in 0.5% chloramine T solution at 4°C for 24 hours. After the extraction, the teeth were stored in distilled water until use, adhering to international and institutional infection control guidelines.

Each tooth root was embedded in self-cure acrylic resin (Acrostone) within cylindrical polyvinyl chloride (PVC) tubes ( $2 \times 2.7$  cm) to a depth of 2 mm below the cementoenamel junction (CEJ). A standardized mid-coronal dentin exposure was created by removing the occlusal enamel and superficial dentin. The mid-coronal location was identified using preoperative radiographic measurements and marked on the tooth surface.

Using a low-speed diamond saw (PICO 155, Pace Technologies, USA) with water cooling (Diacut Waterbased Cutting Fluid, Pace Technologies, USA), the cutting was performed perpendicular to the tooth's longitudinal axis. After cutting, the exposed dentin surfaces were finished with 600-grit silicon carbide paper (Microcut, Buehler, Lake Bluff, IL, USA) for 30 seconds under running water, creating a standardized smear layer.

A total of forty molar teeth were used for the microshear bond strength ( $\mu$ SBS) test. The teeth were randomly assigned to four groups based on the type of restorative material and pretreatment agent used. Each group contained 10 teeth, with each tooth receiving three cylinders of restorative material (n = 30):

- Group A: Surefil One (self-adhesive bulk-fill composite, Dentsply Sirona, USA)
- Group B: Cention Fort without primer (Alkasite-based material, Ivoclar Vivadent, Schaan, Liechtenstein)
- Group C: Cention Fort with primer (Alkasite-based material)
- Group D: Fuji II LC (resin-modified glass ionomer, GC, Tokyo, Japan, control group).

Each group was divided into two subgroups (5 teeth per subgroup), based on storage time: immediate specimens (evaluated after 24 hours) and delayed specimens (evaluated after 6 months of storage in artificial saliva) to assess bond durability. Another forty molars were assigned to the micromorphological analysis group under SEM to evaluate the restoration/dentin interface, divided into four groups (n = 10) according to the restorative material tested. Each subgroup was again divided based on storage time: immediate (n = 5) and delayed specimens (n = 5) after 6 months of artificial saliva storage.

The self-adhesive restorative materials were applied to each specimen using a mold created from addition silicon (polyvinylsiloxane) impression material (Ghenesyl, Super Light Body, Lascod, Florence, Italy). The mold was designed with a 2 mm thickness at the edges and 1 mm thickness at the prepared dentin surface. The molds were used to form restorative material micro-tubes, and three marks were made at the depression

points of the rubber base cylinders. A 1 mm diameter cylindrical end-cutting diamond bur (ISO #150, Osung Dental, USA) was used to drill holes at these marks. The space between adjacent holes was 2 mm. A digital caliper was used to measure the hole diameter and spacing.

For the Cention Fort group with primer, the dentin surface was treated with Cention Primer using a singleuse applicator. After a 10-second scrubbing, the primer was dried with compressed air to form a thin, shiny film. For the Fuji II LC group, dentin conditioner was applied for 20 seconds, followed by thorough rinsing and gentle drying.

Once the pretreatment steps were complete, the rubber base molds were realigned, and the restorative materials were condensed into the holes. The materials were adapted using a rounded blunt-end periodontal probe (1 mm diameter) and compacted with a small ball burnisher. The excess material was removed with a glass slide before light curing, following the manufacturer's instructions with an LED curing unit (Elipar TM Deep Cure-S, 1200 W/cm<sup>2</sup>, 350-520 nm wavelength range).

After setting, the mold was removed using a scalpel, and any excess material around the base was trimmed. Three tubes were obtained, each with a length and diameter of 1 mm, and their cross-sectional area was measured using a digital caliper.

The immediate specimens (n = 15) were stored in water at 37°C for 24 hours before evaluation. The delayed specimens (n = 15) were stored in artificial saliva for 6 months at 37°C in an incubator. The artificial saliva was changed weekly during the storage period.

The specimens were mounted on a universal testing machine (Model 3345, Instron, USA) for the  $\mu$ SBS test. The specimens were fixed to the lower compartment using tightening screws, and a thin orthodontic wire loop (0.2 mm diameter) was attached to the material cylinder/dentin interface. The loop was connected to the upper arm of the testing machine. Load was applied at a crosshead speed of 1 mm/min until fracture occurred. The software of the testing machine was used to analyze the stress-strain curve. The  $\mu$ SBS was calculated by dividing the load at failure (in Newtons) by the cross-sectional area of the material cylinder ( $\pi$ r<sup>2</sup> in mm<sup>2</sup>).

The failure modes of the specimens were examined using a stereomicroscope (SZX10; Olympus, Japan) at ×40 magnification and classified as follows:

- Cohesive failure within dentin (CF-D)
- Adhesive failure at the material/dentin interface (AF)
- Mixed failure (MF)
- Cohesive failure within the restorative material (CF-M)
- Failure during artificial aging period (PTF).

Representative samples of each failure mode were gold-coated and examined using scanning electron microscopy (SEM, JSM-6510LV SEM, JEOL Ltd., Tokyo, Japan) at 50× or 55× magnification for further analysis.

An additional five molars from each subgroup were prepared, totaling 40 teeth across all groups, in line with the study design outlined for the microshear bond strength test. The immediate subgroup (I) was stored in distilled water at 37°C for 24 hours, while the delayed subgroup (D) was immersed in artificial saliva for six months.

The enamel and superficial dentin layers were removed to expose the mid-dentin using a low-speed diamond saw (PICO 155 precision saw). Water coolant was applied throughout the procedure. The cut surfaces were then polished with 600-grit silicon carbide paper to standardize the smear layer, which is required for the microshear bond strength assessment. Following this, the assigned self-adhesive restorative materials were applied to the prepared surfaces. All materials were prepared and applied in accordance with the manufacturers' instructions. The material height was standardized to 2mm using a Tofflemire matrix system, measured with a periodontal probe. A glass slide was used to compress the restorative material, reducing surface voids and extruding excess material. After complete curing of the material, the metal band

was removed, and the specimens were kept in distilled water at room temperature for 24 hours before sectioning.

After the 24-hour period, the molars were vertically sectioned bucco-lingually into two halves along the long axis of the teeth, perpendicularly to the restoration-dentin interface. Horizontal sectioning at the level of the cementoenamel junction (CEJ) followed, separating the specimens from the acrylic resin using a diamond disc under low-speed conditions. Water-based coolant (Diacut Water-based Cutting Fluid) was used during the cutting process at a ratio of 1:33 (lubricant: water).

Resin-dentin slabs from each half of the tooth were polished sequentially with silicon carbide papers of increasing grits (600, 1000, 1200, 2000, and 4000 grit), followed by diamond pastes of progressively smaller sizes (6, 4, and 1 microns). A polishing cloth was used for this step. To remove any remaining debris, the samples were placed in an ultrasonic cleaner for 10 minutes.

Prior to the acid-base challenge, specimens were stored at room temperature in a saline solution for 10 minutes. They were then exposed to 10% orthophosphoric acid for 5 seconds, followed by immersion in a 5% sodium hypochlorite solution for 5 minutes. This process demineralized any dentin areas not infiltrated by resin, allowing for dehydration of the dentin. The specimens were dried and kept dry for 24 hours before gold plating. The samples were gold sputtered and observed using scanning electron microscopy (SEM) at an accelerating voltage of 30 kV and a working distance of 10–15 mm, with secondary electron detection mode. A series of SEM images were taken at various magnifications, and the clearest image at ×1000 magnification was selected for analysis. This procedure follows the methodology established by Hamama et al. (32).

## Statistical Analysis

The data were organized and coded using Microsoft Excel 2016. Statistical analysis was performed with SPSS (Version 22).

# Results

The Shapiro-Wilk test confirmed that the  $\mu$ SBS data in all groups adhered to a normal distribution pattern (p > 0.05). Analysis via two-way ANOVA demonstrated that both the "restorative material type" and "storage time" had significant effects on the  $\mu$ SBS (p < 0.05), with a notable interaction between the two factors (material type \* storage time) (p < 0.05)

For the immediate (un-stored) groups, the post-hoc Tukey's HSD multiple comparison test revealed that the CNF + P (Cention Forte with Cention primer) group exhibited a significantly higher  $\mu$ SBS mean (26.0360 ± 5.14593 MPa) than all other groups (p < 0.05). This was followed by the FJI (Fuji II LC) group, which had a mean of 21.7860 ± 3.30991 MPa (control group). On the other hand, the CNF (Cention Forte without Cention primer) group had the lowest  $\mu$ SBS mean (1.5540 ± 1.09871 MPa), significantly lower than all other groups (p < 0.05). The  $\mu$ SBS mean for the SU-O (Surefil One) group (2.1587 ± 1.24608 MPa) was not statistically different from that of the CNF group (p > 0.05).

For the delayed groups (after 6 months in artificial saliva), the CNF + P group still had a significantly higher  $\mu$ SBS mean (21.3773 ± 2.77064 MPa) compared to the other groups (p < 0.05). However, no statistically significant differences (p > 0.05) were observed between the  $\mu$ SBS values for CNF (0.3333 ± 0.86874 MPa), SU-O (0.2060 ± 0.37127 MPa), and FJI (2.2880 ± 1.17500 MPa) groups.

The results indicated that aging in artificial saliva notably affected the  $\mu$ SBS of certain restorative materials (FJI and CNF + P). Specifically, the delayed group exhibited lower  $\mu$ SBS compared to the immediate group of the same material. A significant difference was found between stored and un-stored groups for some restorative materials (p < 0.05).

es of all specimens were analyzed using a stereomicroscope (SZX10, Olympus, Japan) at 40× magnification. Representative samples from each failure mode were further gold-coated and examined under scanning electron microscopy (SEM, JSM-6510LV SEM, JEOL Ltd., Japan) at 50× to 55× magnification. The predominant failure mode across all groups was adhesive failure. The cohesive failure mode was the least common among all groups. The immediate CNF + P group, which exhibited the highest  $\mu$ SBS, showed fewer adhesive failures. Cohesive failures were recorded only in the CNF + P I and FJI I groups.

SEM micrographs at a magnification of  $\times 1000$  of the resin-dentin interface across all groups revealed that both the immediate and delayed groups of Cention Forte with Cention primer and Fuji II LC demonstrated good, uniform adaptation. In contrast, Cention Forte without Cention primer and Surefil One groups showed poor adaptation to the dentin substrate.

For both the SU-O and CNF groups, immediate and delayed, the material-dentin interface appeared disjointed. There was no evidence of material infiltration into dentin tubules, and resin tags did not penetrate the dentin surface. Additionally, the interfacial gaps, absence of a hybrid layer, and incomplete removal of the smear layer were observed, with dentinal tubules blocked by smear plugs (Figures 5 and 6).

The SEM images of CNF + P (immediate and delayed groups) showed a continuous material/dentin interface with long resin tags extending into the dentinal tubules. Minor interfacial gaps corresponding to the residual smear layer and a hybrid-like layer were present.

The FJI/pre-conditioned dentin interface analysis revealed that both immediate and delayed groups demonstrated complete smear layer removal and open dentinal tubules, thanks to the dentin conditioner. There was close contact at the FJI/dentin interface, with resin tags exhibiting a short, thick, funnel-shaped pattern that penetrated the dentin surface, without any sign of separation or interfacial gaps. A hybrid-like layer was also clearly visible.

Conversely, the material/dentin interface for SU-O and CNF showed a discontinuous appearance in the delayed specimens, with evidence of a thick smear layer, blocked dentinal tubules, and a lack of resin tag penetration. The separation at the material/dentin interface was more pronounced in the delayed groups.

## Discussion

The adhesive interfaces in dental restorations are a critical point that can be prone to failure. Insufficient adhesion to the tooth structure or marginal leakage at this interface can lead to a series of adverse outcomes, such as discoloration, bacterial penetration, and eventual restoration failure (33). This in-vitro study was designed to assess the bonding performance of two newly developed self-adhesive bulk-fill hybrid restorative materials in comparison with a well-established control material, RMCIC restoration.

One of the self-adhesive bulk-fill hybrid composite materials, a recent innovation, was introduced by the manufacturer (34) as a "forgiving material" combining the ease of application found in glass-ionomer cement (GIC) with the enduring stability typical of conventional resin-based composites (RBCs), while still delivering aesthetically pleasing results. To verify these claims, the study compared the bonding effectiveness of the self-adhesive bulk-fill hybrid composite to two established materials: Alkasite-based restorative material and resin-modified GIC (RMGIC), with RMGIC acting as the control group.

This study also explored the performance of the Alkasite-based restorative material, a novel hybrid, in two application methods: self-adhesive and non-self-adhesive. The manufacturer's protocol was followed, utilizing Alkasite's Primer, a dedicated bonding system for self-adhesive application, on untreated dentin surfaces, specifically in un-retained cavities. However, Alkasite-based restorative material is also designed to function as a self-adhesive material without the need for a primer in retentive cavities.

The choice of RMGIC as the control material was based on its strong chemical bond with the tooth substrate, making it a widely used self-adhesive restorative material. The material was applied following the manufacturer's instructions, which included pre-conditioning with polyalkenoic acid (PAA) (35).

The current study aimed to evaluate bonding effectiveness and durability after simulated aging (6 months in artificial saliva) by assessing: 1) the microshear bond strength of the tested materials, along with identification of failure modes, and 2) the micromorphological characteristics of the materials at the restoration/dentin interface.

Simulated aging is crucial in assessing bond durability (36). In this experiment, teeth were stored in artificial saliva in an incubator at 37 °C  $\pm$  1 for six months (37) to replicate the oral environment, providing a more accurate assessment of the restorative materials' behavior (38).

A microshear bond strength ( $\mu$ SBS) test was selected to evaluate the bonding strength of the three restorative materials applied to flat (mid-coronal) dentin. This test is particularly relevant in clinical settings, as shearing forces predominantly act at the tooth-restoration interface (39). Microshear bond strength testing offers certain advantages over traditional SBS testing, as smaller specimens tend to exhibit stronger performance. This is due to a reduced likelihood of critical defects, though any minor defects present may cause misalignment with the applied force (40).

The study compared bond strength under ideal conditions with bonding in high C-factor class-I cavities, which represent a more challenging scenario where shrinkage stress would significantly affect the bond to the cavity-bottom dentin, especially for restorative materials applied in a full self-adhesive and bulk-fill manner (self-adhesive bulk-fill composite, Alkasite-based restorative material) (41).

Rubber base impression material was identified as the best choice for creating molds for restorative material micro 'tygon tubes,' as this method prevents the tested material from adhering to the testing tube. This was believed to preserve the integrity of the microtube structures during the mold removal process (42,43).

The results showed significant differences in  $\mu$ SBS between immediate groups, with self-adhesive bulk-fill composite and Alkasite-based restorative material without primer exhibiting significantly lower bond strength. After six months of aging in artificial saliva, the aged  $\mu$ SBS values of all three restorative materials showed a significant decrease compared to the immediate  $\mu$ SBS values at 24 hours, particularly for resinmodified GIC and Alkasite-based restorative material with primer. In contrast, the  $\mu$ SBS of self-adhesive bulk-fill composite and Alkasite-based restorative material without primer did not show a significant decline after aging.

The aging process had a notable impact, as solvent absorption and solubility during storage are common phenomena that cause chemical changes and adversely affect the mechanical properties of polymeric materials. The absorption of aqueous solvents leads to swelling, which results from the separation of polymeric chains. This swelling is accompanied by the loss of non-reacted components, erosion of the fillermatrix interface, and plasticization, ultimately reducing the material's stiffness, hardness, wear resistance, and flexural strength (36).

The self-adhesive bulk-fill composite demonstrated lower microshear bond strength (µSBS) results compared to a pre-treated Alkasite-based restorative material with primer, as well as the immediate group of pre-conditioned resin-modified glass-ionomer cement (RMGIC) (control group). Interestingly, no significant difference was observed between the self-adhesive bulk-fill composite samples (both immediate and aged). These results highlight the limitations in the self-adhesive properties of the bulk-fill composite material, despite its inclusion of components designed to promote adhesion. The composite contains high molecular weight polyacrylic acid, which is meant to aid in smear layer hybridization and facilitate covalent bond formation. Additionally, the material incorporates a hydrolytically stable MOPOS monomer, which is intended to enhance adhesion to dentin via ionic interactions between the dentin's calcium and MOPOS' carboxyl groups. Despite these features, the composite exhibited weak self-adhesion to dentin, yielding weak bond strength similar to the untreated Alkasite-based restorative material (34,44). Both materials primarily showed adhesive failure at the dentin interface, with a significant rate of pre-test failures (PTFs) in the delayed aging groups. Microscopic examination of the interfaces revealed a lack of resin-dentin interdiffusion and signs of separation, attributed to the smear layer blocking dentinal tubules and a potential absence of a resin adhesive agent. The absence of strong bonding could be due to the smear layer preventing the materials from properly penetrating into the dentin. These observations further suggest that both the self-adhesive bulk-fill composite and the Alkasite-based material's bonding abilities are compromised due to insufficient penetration of the smear layer, reinforcing the conclusion that the self-adhesive properties of both materials are inadequate (45).

The study also confirms that the Alkasite-based restorative material, without a primer, exhibited the weakest bond strength, with significantly lower µSBS values for both immediate and aged samples, and a high rate of PTFs after aging. The results for this material can be explained by the absence of polyacrylic acid or acidic monomers, which are typically used to improve bonding strength. These findings advocate for the systematic application of a primer with Alkasite-based restorative material. Previous studies also support this, showing that bond strengths were lower without pre-treatment compared to when a pre-treatment agent was applied (46). The primer used in this study, a self-etching and self-curing system, resulted in the highest bond strength among all tested materials, with no PTFs observed during either preparation or storage. Scanning electron microscope (SEM) analysis showed a hybrid layer formation and resin tags, indicating that the primer effectively demineralized the dentin surface and facilitated the diffusion of resin into exposed collagen fibrils.

In contrast, the RMGIC group exhibited a higher bond strength in the immediate subgroup, primarily due to micro-mechanical interlocking and chemical bonding with dentin. However, aging led to a significant reduction in bond strength, especially in the aged RMGIC samples. While no PTFs were recorded, adhesive failure was the predominant failure mode in the aged subgroup, while mixed failure was more common in the immediate subgroup. The failure mode was correlated with the  $\mu$ SBS results, as higher bond strengths were associated with mixed failure, and lower strengths were linked to adhesive failure (48).

In SEM imaging, RMGICs showed intimate contact with the dentin surface, where resin tags and an acidbase resistant layer were visible. These features were indicative of the material's ability to resist demineralization. Both Alkasite-based restorative material with primer and RMGICs demonstrated this ionreleasing property, which supported the formation of a hybrid layer and improved bond strength. The bioactivity of Alkasite-based restorative material and self-adhesive bulk-fill composite has been studied in several papers, which highlighted their superior fluoride and calcium ion-release rates compared to traditional glass ionomer restorations. This mineralization capability has been associated with enhanced bond strength (49,50,51), as confirmed in other studies that observed a positive correlation between the hybrid layer and bond strength in RMGICs compared to high-viscosity glass ionomer cements (HV-GICs) (52,53).

Similar research has shown that materials requiring pre-treatment, such as Alkasite-based material with primer, achieved much higher bond strength than self-adhesive materials like self-adhesive bulk-fill composite or RMGIC, which are less dependent on pre-treatment. Among the materials without pre-treatment, the self-adhesive bulk-fill composite exhibited the highest shear bond strength (30). The superior bond strength of Alkasite-based restorative material was also attributed to the use of a primer, which promotes better bonding (54,55).

Self-adhesive bulk-fill composite and resin-modified glass-ionomer cements have similar self-adhesiveness to both enamel and dentin, but there is limited information on the bond strength of self-adhesive bulk-fill composites compared to other materials (24,25). The findings of this study reject the null hypothesis, which suggested no significant difference in bond strength and interface morphology among the tested materials. Moreover, the hypothesis that the bond strength would not decrease significantly with aging was also rejected for all materials except for the self-adhesive bulk-fill composite and Alkasite-based material groups.

The clinical significance of this in-vitro study suggests that using Alkasite-based material with primer may be a viable alternative to preconditioned Fuji II LC in terms of bond durability. However, using Alkasite-based material without its corresponding primer is not recommended. This study does not provide sufficient evidence to support SU-O as a permanent restorative alternative.

This study does have limitations, such as the use of extracted teeth, which lack blood flow and may not fully replicate the dynamic oral environment with its pulpal pressure, fluid flow, and varying pH (56). Further clinical trials are necessary to assess the long-term performance of these bioactive restorative materials in patients and to explore how they interact with decayed dentin.

# Conclusion

In conclusion, this study emphasizes the importance of using primers with Alkasite-based restorative material for optimal bonding to dentin. This outcome highlights the value of applying primer to enhance adhesion, which slightly contrasts with the initial manufacturer's recommendations, categorizing the material as self-adhesive.

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