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Interfacial Bond Strength and Morphology of Sound and Caries-affected Dentin Surfaces Bonded to Two Resin-modified Glass Ionomer Cements

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Clinical Relevance

Some resin-modified glass ionomers may enhance outcomes when used with resin composite in stress bearing areas.

SUMMARY

Objective: To evaluate the shear bond strength and interfacial morphology of sound and caries-affected dentin (CAD) bonded to two resin-modified glass ionomer cements (RMGICs) after 24 hours and two months of storage in simulated body fluid at 37°C.

Methods and Materials: Sixty-four permanent human mandibular first molars (32 sound and 32 with occlusal caries, following the International Caries Detection and Assessment System) were selected. Each prepared substrate (sound and CAD) was conditioned (10% polyacrylic acid) and bonded to Activa BioACTIVE Restorative (Activa) and Fuji II LC (F2LC) as per the manufacturers' instructions. Shear bond strength (SBS) was performed after 24 hours and two months of storage. The interfacial surfaces were examined using a digital microscope and scanning electron microscope (SEM). Three-way ANOVA, Bonferroni post-hoc tests (α=0.05), and independent T-tests were used for multifactorial analysis.

Results: Activa exhibited reduced bond strength values to sound and CAD in comparison to F2LC after two time periods (*p*=0.01). There is a pronounced enhancement in SBS of F2LC when bonded to CAD (*p*=0.01) after storage, with no statistically significant effect on sound dentin (*p*=0.309). Activa showed stable SBS to sound and CAD immediately and post-aging (*p*>0.05).

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However, the evidence of mineral-like deposits under an SEM attached to the aged, debonded dentin surfaces, thereby obliterating the exposed dentinal tubules, might support the tissue repair potential of Activa.

Conclusions: The SBS of Activa was lower than F2LC when bonded to sound and CAD, but the bonding stability and sealing ability is advantageous in minimally invasive therapy, suggesting use with a resin composite coverage when used in stress-bearing areas.

INTRODUCTION

Dental caries is the most predominant bacterially mediated disease worldwide, affecting many people globally and causing considerable health care costs.[1] There are significant challenges in managing deep carious lesions since the traditional complete removal of dental caries compromises pulp health and affects the structural integrity of the tooth. In contrast, incomplete removal of the carious lesion, leaving the deeper layers (caries-affected dentin) that exhibit dentin demineralization but whose collagen framework remains intact without bacterial penetration,[2] will help in arresting caries progression, thereby allowing the remineralization of the residual dentin while maintaining dental pulp vitality.[3] There is a recent focus on using self-adhesive materials with bio-reactive properties that can improve the mineral recovery of tissue. This technique demonstrated satisfactory attributes at the tooth-material interface, producing a high-quality seal that is resistant to contamination from oral fluids, as well as interaction with the microbiological species in addition to restoring the diseased tooth tissues.[4,5]

Glass ionomer cements (GICs) have been used for the Atraumatic Restorative Treatment (ART) techniques as a therapeutic alternative to adhesives/composite restorations. These materials contain a polysalt matrix that exhibits a smart behavior[6] with an ability to release biologically active ions such as fluoride, calcium/strontium, and silicate into the surrounding tissue,[7] thus making them capable of supporting tissue repair and remineralization of the residual caries-affected dentin (CAD) with anti-cariogenic properties.[8] However, the inherent brittleness and solubility of GICs[9] may affect their durability and longevity.[10]

The combination of resin chemistry with the coexistent salt matrix is responsible for the smart interactions of resin-modified glass ionomer cements (RMGICs) with enhanced longevity.[6] They are hybrid materials composed of fluoroaluminosilicate glasses, polyacrylic acid, and resinous materials. The interaction between resin and acid-base reactions may affect the structure and properties of RMGICs, even though they exhibited more enhanced flexural and tensile strengths, elastic modulus, and wear resistance than GICs.[11,12] RMGICs showed an ability to release fluoride and other ions including Na, Ca, Sr, Al, P, and Si as conventional GIC but at different rates.[13] Additionally, RMGICs exhibited higher bonding strength to tooth tissues than conventional GIC, since it is based on both chemical interactions and micromechanical interlocking of the polymer and polyacrylic acid-conditioned tooth surface.[14] The dual setting mechanisms of RMGIC are supposed to encourage relief from polymerization stresses leading to an enhanced marginal seal, despite their lower bond strength compared to resin composites.[15] However, the functionalization of an RMGIC via incorporating a phosphate-based monomer into the liquid phase showed an enhancement in its mechanical and adhesive properties.[16]

ACTIVA BioACTIVE Restorative (Activa) is a functionalized self-adhesive RMGIC with bioactive properties due to the ability to release calcium, phosphate, and fluoride ions.[17] However, Porenczuk and others[18] reported a lower fluoride release profile of Activa compared to Ketac Molar Quick Aplicap but higher than the nanohybrid composite resin Tetric EvoCeram. Previous studies[19,20] reported higher flexural strength, flexural fatigue, and wear resistance of Activa compared to RMGICs and GICs, but they were comparable to that of resin composites. The presence of the hydrophilic ionic resin matrix facilitates the diffusion of ions added to the ability to interact with pH changes, as claimed by the manufacturers, which is supposed to enhance the marginal seal with antimicrobial qualities.[21] However, a one-year randomized clinical study[22] showed a high initial failure rate mainly due to restoration loss and post-operative sensitivity with secondary caries. It is therefore important to investigate the materials' bonding ability and the structural changes of the CAD interface in relevance to the minimal invasive concepts. Therefore, this *in vitro* study compared bonding ability and assessed the interfacial surface changes of sound and CAD surfaces bonded to Activa in comparison to Fuji II LC (F2LC) after 1 day and 60 days of storage in simulated body fluid (SBF) at 37°C. The null hypotheses proposed in this study were: (1) there are no significant differences in the shear bond strength (SBS) of both materials when bonded to sound vs CAD surfaces immediately and post-storage; and (2) there is no significant effect of aging on the SBS of each material per substrate.

METHODS AND MATERIALS

Sixty-four human permanent mandibular first molars (32 sound and 32 with occlusal caries) were collected and stored in distilled water in a cold cabinet (4°C). Samples were hemi-sectioned longitudinally (Isomet 1000, Buehler, Lake Bluff, IL, USA) using a water-cooled diamond blade (330-CA/RS-70300, Struers, LLC, Westlake Cleveland, USA) and embedded in epoxy resin molds. Thirty-two teeth with carious dentin lesions were selected having lesion score 4 following the International Caries Detection and Assessment System (ICDAS),[23] in which the lesion extended through dentin without pulp exposure. The CAD area was inspected visually via detecting the color change[24,25] and by tactile sensation using a dental explorer to examine the consistency and moisture of the different carious zones.[26] Further characterization of the CAD area was carried out using a digital Vickers microhardness machine (TH714, China) by applying 200 gf load for 15 seconds under 400× microscopic magnification. Six measurements (n=2 per zone) were taken in a sequential pattern within each carious lesion, starting from the dentin-enamel junction, by which three zones were identified: caries-infected dentin, CAD, and sound dentin with the Vickers hardness number (VHN) values range for the three zones—17.7-22.7, 27.9-39.0, 43.7-54.5, respectively. All substrates were polished with 600-grit Al_2O_3 abrasive paper under running water using a polishing machine (Laryee Technology CO LTD, China) for 60 seconds, in order to gain flat and smooth surfaces with a standardized smear layer, followed by 3 minutes of ultrasonic cleaning. Then, all surfaces were conditioned using a Dentin Conditioner (10% PAA, GC Corp) for 20 seconds to remove the smear layer, then washed away using air/water spray for 15 seconds and dried for 15 seconds. The experimental groups following the experimental procedures are illustrated in Figure 1.

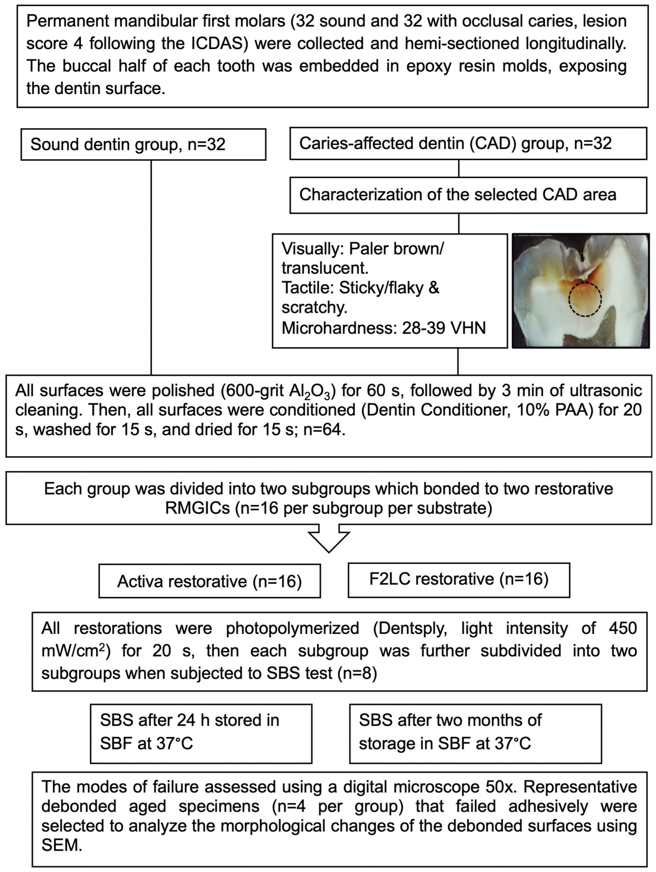

Figure 1. *A scheme representing the experimental groups following the experimental procedures.*

Shear Bond Strength

Each substrate (sound and CAD, n=32 per group) was subdivided randomly into two groups (n=16), one bonded to Activa and the other to F2LC. Following the manufacturers' instructions (Table 1), the materials were dispensed into cylindrical silicone molds (1.75 mm diameter x 3 mm height) that covered the selected areas completely (Tygon tubing, Saint-Gobain, USA), then photopolymerized using a light-curing device (Model 503, Dentsply, Germany; light intensity of 450 mW/cm^2) for 20 seconds.

A SBS test was performed after 1 day and following 60 days of storage in SBF at 37°C (n=8 per subgroup). The solution was prepared following Kokubo and Takadamas' formula[27] to examine the reactivity of Activa for apatite induction and was changed on a weekly basis. The specimens were attached to a universal testing machine (Wdw-50, Laryee Technology Co., China), in which the shear force is directed as close as possible to the tooth/restoration interface. It was applied at a crosshead speed of 0.5 mm/min until failure. The SBS (t) was calculated in MPa using the equation $t=F/(\pi R^2)$ where F was the applied load at failure and R was the radius of the material cylinder.

Mode of Failures and Morphological Assessment of the Debonded Surfaces

The modes of failure were assessed using a digital microscope 50× (Dino-Lite Basic-AM-2011, Taiwan). Then, representative debonded aged specimens (n=4 per group) that failed adhesively were selected to analyze the morphological changes of the debonded surfaces using a scanning electron microscope (SEM; TESCAN, Vega III, Czech Republic) at an accelerating voltage of 10 kV and magnification powers (30×, 500×, and 2500×) and working distances (2 mm, 100 μm, and 20 μm, respectively).

Table 1: *Materials Used and Their Chemical Composition*

Materials and Manufacturers	Composition	Method of Application	Lot No.	Code
Fuji II LC Capsule, Shade A2 GC Corp, Japan	Powder: FAS-glass Liquid: PAA 5%-10%, HEMA 25%-50%, UDMA 1%-5%, Camphorquinone, water.	The capsules were tapped, activated for 10 s and mixed for 10 s (Ultramat 2, 4600 oscillations/min, SDI, Germany). The material was photo-polymerized using a light-curing device (Model 503, Dentsply, Germany), with a light intensity of 450 mW/cm^2 for 20 s.	1909061	F2LC
ACTIVA BioACTIVE Restorative, Shade A2 Pulpdent Corp. USA	Matrix: Diurethane modified by the insertion of a hydrogenated poly-butadiene and other meth-acrylate monomers, modified PAA, sodium, fluoride (0.75%) Filler: 57 wt% (50% bioactive glass & calcium, and 7% silica)	The mixing tip was placed on the Automix syringe. The material was dispensed directly onto the mold and photopolymerized (Model 503, Dentsply, Germany) with a light intensity of 450 mW/cm^2 for 20 s.	190801	Activa
Dentin Conditioner GC Corp, Tokyo, Japan	90% distilled water and 10% polyacrylic acid	Applied for 20 s, washed with air/water spray for 15 s, and dried for 15 s without desiccation	1907101	

Abbreviations: FAS-glass, fluoro-alumino-silicate glass; PAA, polyacrylic acid; HEMA, 2-hydroxyethylmethacrylate; UDMA, urethane dimethacrylate.

Statistical Analysis

The normality tests were performed using Q-Q plots and Shapiro-Wilk. Since the data were normally distributed, further analyses were carried out using three-way analysis-of-variance (ANOVA) and Bonferroni post-hoc tests at the alpha level of significance of 0.05 at each time interval. Then independent t-tests were used to determine the effect of aging on the SBS of each material per substrate, and the differences between substrates ($p<0.05$). The analyses were conducted using SPSS statistical package (version 24, SPSS Inc, IBM, Chicago, IL, USA).

RESULTS

Shear Bond Strength to Sound Dentin

Activa showed statistically significantly lower early and delayed SBS values (3.0±0.5, 2.9±0.4 MPa, respectively) in comparison to F2LC (10.7±2.1, 8.7±2.6 MPa, respectively) ($p<0.01$) when bonded to sound dentin, as shown in Table 2. Aging did not improve the SBS of either material to sound dentin. F2LC showed a lower SBS than its early value, but the difference was statistically insignificant ($p=0.309$), while Activa maintained its bond strength over time ($p=0.943$). The early failures in Activa were predominantly adhesive (87.5%), while in F2LC they were predominantly mixed (50%), combined with 37.5% adhesive and 12.5% cohesive within the cement (Figure 2). However, mixed failures were increased post-aging in F2LC to 75%, and Activa to 50%, combined with less adhesive failures (25% and 50%, respectively).

Shear Bond Strength to Caries-Affected Dentin

Similar to sound substrate, Activa showed reduced SBS values to CAD immediately and post-aging (2.2±0.3, 2.1±0.4 MPa, respectively) compared to F2LC (5.5±1.1, 10.8±2.2 MPa, respectively) ($p=0.01$), as shown in Table 2. The bonding strength of F2LC to CAD was doubled after two months of aging as compared to its early value ($p=0.01$), while it remained constant in Activa post-aging ($p=0.863$). After 24 hours, failures in Activa were entirely adhesive which changed to 75% mixed mode after storage. The same pattern of failures was noticed in F2LC, as they were predominantly adhesive (75%) after 24 hours, but shifted to 25% after storage, as shown in Figure 2.

The difference between substrates (sound vs CAD) had no influence on the bond strength of Activa, immediately and post-storage ($p=0.389$ and 0.337, respectively). In contrast, F2LC showed higher SBS to sound dentin after 24 hours ($p=0.01$) and higher to CAD

Table 2: *Shear Bond Strength (mean MPA±SD) to Sound Dentin and Caries-Affected Dentin After 1 Day and 60 Days Aging in Simulated Body Fluid at 37°C (n=8)*

Materials	Sound Dentin		Caries-Affected Dentin	
	1 d	60 d	1 d	60 d
F2LC	10.7 ± 2.1 ab	8.7 ± 2.6 a	5.5 ± 1.1 a	10.8 ± 2.2 abc
Activa	3.0 ± 0.5	2.9 ± 0.4	2.2 ± 0.3	2.1 ± 0.4

[a] *A statistically significant difference between subgroups in each column (Bonferroni post-hoc tests (α=0.05).*

[b] *A statistically significant difference in values of each material per substrate.*

[c] *A statistically significant effect of aging for each subgroup from day 1 value with in each row (Independent t-test).*

post-aging (p=0.020). The analyzed morphological changes through an SEM of the debonded surfaces are described in Figure 3.

DISCUSSION

The incorporation of energy-absorbing resilient resin matrix in Activa, which is a blend of diurethane and methacrylates with modified polyacrylic acid and polybutadiene modified diurethane dimethacrylate,[28] is supposed to enhance the resilience against impact forces as manifested by high flexural strength and flexural fatigue, which might make it suitable to be used in stress-bearing areas.[19] Additionally, the presence of micro- and nano-hydroxyapatite with a nano-filled RMGIC was reported to reduce the surface roughness changes upon mechanical brushing.[29] On the other hand, the presence of Bioglass in pursuit of enhanced physical properties, combined with the ability to release calcium, phosphate, and fluoride ions, rendered Activa a bioactive restorative material.[17,30] However, the RMGIs already contain reactive, ion-releasing glasses, which in the broadest sense make them bioactive materials. Accordingly, this study was conducted to evaluate whether this class of material is suitable for the Atraumatic Restorative Treatment techniques regarding the bonding efficiency to healthy and diseased tooth tissues, and if there is a potential for tissue repair compared to the widely used RMGIC (F2LC).

Concerning the bonding strength to sound dentin, Activa showed reduced values (3 MPa) immediately and post-aging in comparison to F2LC (<11 MPa), which was even lower than the reported values for RMGICs in the literature (≃7 MPa).[31] The reduced adhesive strength of Activa was also reported in a previous study from Benetti and others[32] who reported a complete loss of all restorations before shear testing when bonded directly to dentin without surface pretreatment. This contradicted the self-adhesion capability that was claimed by the manufacturer, who promised the formation of a resin-hydroxyapatite complex that enhanced the marginal seal against microleakage.[33]

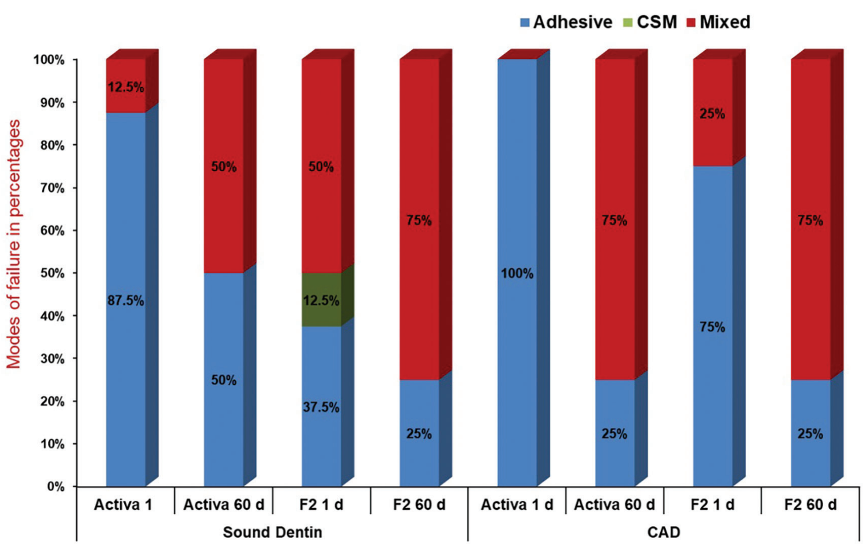

Figure 2. *Modes of failures of Activa and F2LC bonded to sound and caries-affected dentin after 1 day and 60 days of storage in simulated body fluid at 37°C. CSM: cohesive strength within restorative materials.*

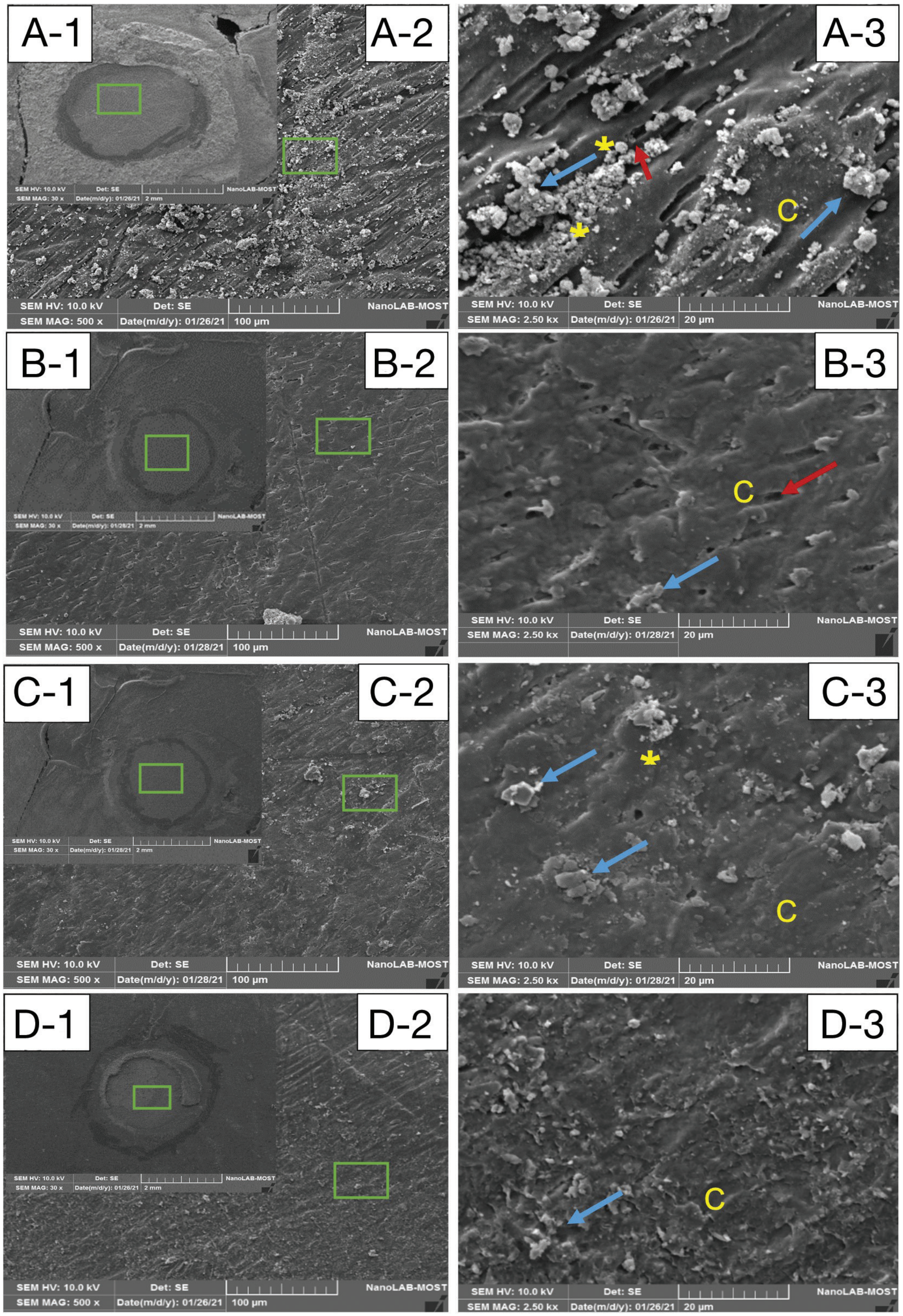

Figure 3. *Representative SEM observations of fractured sound dentin surfaces bonded to Activa (A) and F2LC (B), following two months of storage in simulated body fluid. In A-3 and B-3, failures are cohesive within the hybrid complex showing numerous obliterated dentinal tubules by resinous tags (blue arrows), while some tubules are still open (red arrows), with the remaining cements (C) well integrated to debonded surfaces. In A-3, there is evidence of irregularly shaped granular patches (yellow asterisk) that are attached to the surface and obliterated the dentinal tubules which might indicate the mineral-forming potential of Activa. The SEM observations of the adhesive failures in CAD substrates bonded to Activa and F2LC are shown in C, and D, respectively. The failures occurred within the hybrid layer (C-3, D-3) where the lesions are well sealed by both materials (C), with numerous resinous tags occluding the dentinal tubules (blue arrows).*

Clinical and laboratory studies[21,22,32] reported an enhancement in the bond strength of Activa if they combined with self-etch adhesives, and hence the manufacturers advocated their use recently. However, the use of an adhesive with Activa may interfere with the ion exchange concept, rendering any interfacial bioactive activity very doubtful.[34]

Nevertheless, the bond strength of Activa to dentin was steady after aging accompanied with a shift in the mode of failure from predominantly adhesive (87.5%) to 50% adhesive and mixed patterns, Figure 2, which might indicate the potential chemical integration to sound dentin substrate. This might be related to the reactivity and hydrophilicity of matrix in both cements, which contribute to the acid-base reaction and the formation of stable ionic interactions that endure dissolution and resist the plasticizing effect of water over time[31] and lead to the formation of a homogenous matrix, as evidenced by the SEM images that also showed a dispersion of an irregularly shaped granular mineral kind of deposit covering the aged interface that debonded from Activa associated with completely closed dentinal tubules, as shown in Figure 3 (A-3). This might support the mineral-forming potential of the cement, as claimed by the manufacturer, which would help remineralize the residual carious lesions which requires further investigation. Accordingly, the first hypothesis was rejected, but the second one was accepted as aging does not affect the bond strength of both materials to sound dentin.

The bonding to natural caries dentin is challenging and somewhat unpredictable. This is attributed to the mineral depletion, as presented by a lower Vickers hardness number than sound dentin, the poor quality of hybrid layer, and the extra moisture that might induce hydrolysis of resin and collagen fibrils.[35] These factors might jeopardize the micromechanical interlocking of resin-based polymers producing a lower immediate bond strength in comparison to sound dentin.[36,37] This fact coincides with the finding of this study regarding the reduced initial bond strength of F2LC to CAD than to sound dentin with a higher percentage of adhesive failures (75%) (Figure 2). Although the thick hybrid layer might jeopardize the early micromechanical interlocking into CAD, more ions will be available at the interface for more ionic bonding which might reinforce the carious substrate as presented by a two-fold increase in bond strength after aging, which was higher than that to sound dentin, combined with predominant mixed failures (75%), Figure 2, which might be correlated to the chemical integration of F2LC to CAD surface as shown under an SEM, as the surface appeared well sealed by F2LC with numerous resin tags occluding the exposed tubules (Figure 3, D-3).

The bond strength of Activa to CAD was low (2 MPa), immediately and after storage, compared to F2LC (5.5-10.8 MPa). However, the expected smart behavior of Activa, coupled with the potential chemical bonding due to the ion exchange phenomena,[38] offered a stable bond strength over time without further deterioration. It could be supposed that this ion exchange would be favorable within the residual carious lesion to contribute to the remineralization process, since the lost mineral and apatite crystals can be reintegrated when bonded to bioactive materials,[39] and would potentially protect the restorative-dentin interface from the degradation effect. This was evident through the shift from totally adhesive failures to 75% mixed failures after storage, associated with sealing the carious interface with numerous resinous tags occluding the exposed dentinal tubules under an SEM (Figure 3, C-3). The bond stability of RMGICs to CAD was also revealed in previous studies.[8,40] Accordingly, the first hypothesis was rejected, but the second one was accepted in Activa only, as aging did not affect the bond strength to CAD. Nevertheless, randomized clinical trials are required to justify the use of this promising material regarding tissue repairability and clinical longevity.

CONCLUSIONS

Activa showed reduced bond strength to sound and CAD when compared to F2LC. There was a great enhancement in shear bond strength of F2LC to CAD post storage with no effect of aging noticed in Activa when bonded to sound and CAD. However, the evidence of mineral-like deposits under an SEM attached to the aged debonded dentin surfaces, obliterating the exposed dentinal tubules, might support the tissue repair potential of Activa which might necessitate further investigation.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the human subjects oversight committee guidelines and policies of Baghdad College of Dentistry. The approval code was Reference Number 226.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article

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