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LONG-TERM DURABILITY OF COMPOSITE BONDING TO SILVER DIAMINE FLUORIDE–TREATED ENAMEL: A COMPARATIVE EVALUATION OF ETCH-AND-RINSE AND SELF-ETCH UNIVERSAL ADHESIVE STRATEGIES UNDER THERMOCYCLING AND EXTENDED WATER STORAGE

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ABSTRACT

Background: Silver diamine fluoride (SDF) is widely used for caries arrest; however, its influence on the long-term durability of composite bonding to enamel remains a clinical concern. This study evaluated the effect of etch-and-rinse and self-etch universal adhesive strategies on the bond durability of composite restorations to SDF-treated enamel following artificial aging.

Materials and Methods: One hundred extracted human premolars were randomly allocated into four groups ($n = 25$): Control Etch-and-Rinse, Control Self-Etch, SDF + Etch-and-Rinse, and SDF + Self-Etch. Specimens received 38% SDF application where applicable, followed by composite restoration using a universal adhesive in the designated bonding mode. All samples underwent 10,000 thermocycles and six months of water storage before shear bond strength testing and failure mode analysis.

Results: The Control Etch-and-Rinse group demonstrated the highest mean bond strength (24.82 ± 2.94 MPa), while the SDF + Self-Etch group exhibited the lowest values (17.85 ± 2.48 MPa). One-way ANOVA revealed a statistically significant difference among groups ($p < 0.001$). Etch-and-rinse protocols showed significantly greater bond durability than self-etch approaches, and mixed failures predominated in etch-and-rinse groups, whereas adhesive failures were more frequent in self-etch groups.

Conclusion: SDF pretreatment reduced the long-term bond strength of composite restorations to enamel; however, the etch-and-rinse universal adhesive strategy effectively mitigated this effect and demonstrated superior durability after aging. Phosphoric acid etching appears beneficial for optimizing composite bonding to SDF-treated enamel and may enhance the longevity of restorative outcomes.

KEYWORDS: Silver diamine fluoride; Universal adhesive; Etch-and-rinse; Self-etch; Shear bond strength; Composite resin; Thermocycling; Water storage; Enamel bonding; Bond durability.

INTRODUCTION

Silver diamine fluoride (SDF) has emerged as a highly effective, minimally invasive agent for the prevention and arrest of dental caries. Owing to its dual action of antimicrobial activity and remineralization potential, SDF has gained widespread acceptance in pediatric dentistry, geriatric dentistry, and community oral health programs [1]. The material contains silver ions that exert potent antibacterial effects and fluoride ions that promote remineralization of demineralized tooth structure. Numerous clinical studies have demonstrated the efficacy of 38% SDF in arresting active carious lesions and reducing the progression of dental decay, making it a valuable tool in contemporary caries management strategies [2]. Despite its clinical benefits, concerns remain regarding the subsequent adhesive performance of restorative materials bonded to SDF-treated tooth surfaces [3].

The growing popularity of the Silver Modified Atraumatic Restorative Treatment (SMART) technique has intensified interest in understanding the interaction between SDF-treated enamel and adhesive restorative systems [4]. In this approach, SDF is applied to arrest caries, followed by restoration using resin-based materials. The long-term success of such restorations depends largely on the establishment of a durable adhesive interface between the restorative material and the treated tooth substrate [5]. However, the chemical and structural alterations induced by SDF may influence adhesive bonding mechanisms and compromise restoration longevity if not adequately addressed [6].

The application of SDF results in the formation of silver phosphate, calcium fluoride, and other reaction products that precipitate on the enamel surface [7]. These deposits can alter surface morphology, reduce enamel surface energy, and obstruct microporosities necessary for resin infiltration [8]. Furthermore, the formation of a highly mineralized superficial layer may modify the etching pattern achieved by phosphoric acid or acidic adhesive monomers [9]. Such alterations can affect micromechanical retention and potentially reduce bond strength. Consequently, understanding the influence of SDF pretreatment on adhesive performance has become an important area of restorative research [10].

Universal adhesive systems have transformed adhesive dentistry by providing flexibility in application through either etch-and-rinse or self-etch strategies [11]. Etch-and-rinse adhesives involve phosphoric acid conditioning prior to adhesive application, creating a pronounced enamel etching pattern and enhancing micromechanical retention [12]. In contrast, self-etch adhesives utilize acidic functional monomers that simultaneously condition and infiltrate the tooth surface, simplifying the clinical procedure while minimizing technique sensitivity [13]. Universal adhesives contain functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which can chemically interact with hydroxyapatite and contribute to durable bonding [14]. However, the effectiveness of these adhesive approaches on SDF-treated enamel remains controversial.

Several in vitro investigations have evaluated the immediate bond strength of composite restorations to SDF-treated enamel and dentin [15]. While some studies have reported a reduction in bond strength following SDF application due to interference with resin penetration, others have demonstrated that appropriate surface conditioning can mitigate these adverse effects [16]. Particularly, phosphoric acid etching has been suggested to remove superficial precipitates and enhance resin infiltration, potentially restoring bond strength values comparable to untreated enamel [17]. Nevertheless, the available literature remains inconsistent, partly due to variations in SDF concentration, application protocols, adhesive systems, and testing methodologies[18].

An important limitation of many previous studies is their focus on immediate bond strength evaluation [19]. Although initial adhesion is critical, the long-term clinical success of adhesive restorations depends on the durability of the bonded interface under conditions that simulate the oral environment [20]. Factors such as thermal fluctuations, water sorption, hydrolytic degradation, and mechanical stresses can progressively weaken adhesive bonds over time. Therefore, aging protocols are essential for assessing the longevity and stability of adhesive interfaces [21]. Thermocycling and extended water storage are widely accepted artificial aging methods that mimic thermal and hydrolytic challenges encountered intraorally [22].

Thermocycling subjects restorative materials and tooth structures to repeated temperature changes, generating contraction and expansion stresses at the adhesive interface [23]. These stresses may promote microcrack formation, interfacial debonding, and degradation of resin components [24]. Similarly, prolonged water storage can result in hydrolysis of resin polymers, plasticization of adhesive layers, and deterioration of resin-enamel interactions. The combined use of thermocycling and extended water storage provides a comprehensive assessment of adhesive durability and may better predict long-term clinical performance than immediate bond strength testing alone [25].

Despite the increasing clinical use of SDF and universal adhesives, limited evidence exists regarding the long-term durability of composite bonding to SDF-treated enamel after artificial aging [26]. Furthermore, direct comparisons between etch-and-rinse and self-etch universal adhesive strategies under prolonged aging conditions remain scarce. Since the mechanism of interaction between adhesives and SDF-modified enamel may differ substantially depending on the bonding approach employed, a better understanding of these relationships is necessary to guide evidence-based restorative protocols [27].

Evaluating the performance of adhesive systems following thermocycling and extended water storage is particularly relevant for clinicians adopting SMART and other minimally invasive restorative techniques [28]. Determining whether etch-and-rinse strategies provide superior bond durability compared with self-etch approaches could influence clinical decision-making and improve restoration longevity in SDF-treated teeth. Moreover, identifying adhesive protocols capable of maintaining stable bond strength over time would contribute to enhanced treatment outcomes and patient satisfaction.

Therefore, the present study aimed to evaluate the long-term durability of composite bonding to SDF-treated enamel using universal adhesives applied in etch-and-rinse and self-etch modes. Bond performance was assessed following thermocycling and extended water storage to simulate oral aging conditions. The findings of this investigation may provide valuable insights into the optimal adhesive strategy for restoring SDF-treated enamel and contribute to the development of predictable, durable restorative protocols in minimally invasive dentistry.

METHODOLOGY

Study Design

This in vitro experimental study was conducted to evaluate the long-term durability of composite bonding to Silver Diamine Fluoride (SDF)-treated enamel using universal adhesive systems applied in etch-and-rinse and self-etch modes after thermocycling and extended water storage. Ethical clearance was obtained from the Institutional Research and Ethics Committee prior to commencement of the study.

Sample Size Calculation

A total sample size of 100 extracted human permanent premolars was selected based on previous bond strength studies and power analysis (power = 80%, $\alpha = 0.05$), ensuring adequate statistical significance among the study groups.

Sample Selection

One hundred freshly extracted, caries-free human premolars extracted for orthodontic purposes were collected. Teeth exhibiting cracks, restorations, hypoplastic defects, fluorosis, enamel fractures, or developmental anomalies were excluded. After debridement of soft tissue remnants and calculus, the teeth were stored in 0.1% thymol solution at room temperature until use.

Specimen Preparation

The crowns were cleaned using a rubber prophylaxis cup and non-fluoridated pumice slurry. Buccal enamel surfaces were flattened using 600-grit silicon carbide abrasive papers under continuous water irrigation to obtain standardized bonding surfaces. Each tooth was embedded in self-cure acrylic resin blocks exposing the buccal enamel surface.

Group Allocation

The specimens were randomly divided into four groups (n = 25 each):

Group I (Control Etch-and-Rinse)

No SDF application; universal adhesive applied in etch-and-rinse mode.

Group II (Control Self-Etch)

No SDF application; universal adhesive applied in self-etch mode.

Group III (SDF + Etch-and-Rinse)

38% Silver Diamine Fluoride applied followed by universal adhesive in etch-and-rinse mode.

Group IV (SDF + Self-Etch)

38% Silver Diamine Fluoride applied followed by universal adhesive in self-etch mode.

SDF Application

In Groups III and IV, 38% Silver Diamine Fluoride solution was applied to the prepared enamel surface using a microbrush for 1 minute according to the manufacturer's instructions. Excess material was gently air-dried and specimens were stored in artificial saliva at 37°C for 24 hours before adhesive procedures.

Adhesive Procedures

Etch-and-Rinse Protocol (Groups I and III)

The enamel surface was etched with 37% phosphoric acid gel for 15 seconds, rinsed thoroughly with water for 20 seconds, and air-dried. A universal adhesive was then actively rubbed onto the surface for 20 seconds, gently air-thinned, and light-cured for 10 seconds using an LED curing unit with an intensity of 1200 mW/cm^2.

Self-Etch Protocol (Groups II and IV)

The universal adhesive was directly applied to the enamel surface without prior phosphoric acid etching. The adhesive was scrubbed for 20 seconds, air-thinned, and light-cured for 10 seconds.

Composite Build-Up

A cylindrical Teflon mold (4 mm diameter × 4 mm height) was positioned over the bonded enamel surface. Nanohybrid resin composite was placed incrementally in 2-mm layers and each increment was light-cured for 20 seconds. Following polymerization, the molds were carefully removed.

Aging Procedures

Thermocycling

All specimens were subjected to 10,000 thermocycles between 5°C and 55°C with a dwell time of 30 seconds and transfer time of 10 seconds to simulate approximately one year of clinical service.

Extended Water Storage

Following thermocycling, specimens were stored in distilled water at 37°C for six months. The storage medium was renewed weekly to prevent contamination and maintain consistent conditions.

Shear Bond Strength Testing

After completion of the aging procedures, shear bond strength testing was performed using a Universal Testing Machine. A chisel-shaped loading blade was positioned at the composite-enamel interface and load was applied at a crosshead speed of 1 mm/min until bond failure occurred.

The maximum failure load was recorded in Newtons (N) and converted to Megapascals (MPa) using the formula:

Bond Strength (MPa) = Failure Load (N) / Bonded Surface Area (mm^2)

Failure Mode Analysis

The fractured surfaces were examined under a stereomicroscope at 40× magnification and categorized as:

- Adhesive failure (at enamel-adhesive interface)
- Cohesive failure within composite
- Cohesive failure within enamel

- Mixed failure

Statistical Analysis

Data were analyzed using SPSS software version 26.0. Mean and standard deviation values of bond strength were calculated for all groups. Normality was assessed using the Shapiro-Wilk test. Intergroup comparisons were performed using one-way Analysis of Variance (ANOVA) followed by Tukey's post hoc test. The distribution of failure modes was analyzed using the Chi-square test. A p-value <0.05 was considered statistically significant.

RESULTS

A total of 100 enamel specimens were evaluated for long-term composite bond durability following thermocycling and six months of water storage. Mean shear bond strength (SBS) values and failure mode distributions were analyzed among the four study groups.

Shear Bond Strength Analysis

Group I (Control Etch-and-Rinse) demonstrated the highest mean bond strength (24.82 ± 2.94 MPa), followed by Group III (SDF + Etch-and-Rinse) (22.96 ± 2.71 MPa). Lower bond strength values were observed in Group II (Control Self-Etch) (20.41 ± 2.63 MPa), while Group IV (SDF + Self-Etch) exhibited the lowest mean bond strength (17.85 ± 2.48 MPa).

Table 1. Comparison of Shear Bond Strength Among Study Groups

Group	n	Mean SBS (MPa)	SD
Group I (Control Etch-and-Rinse)	25	24.82	2.94
Group II (Control Self-Etch)	25	20.41	2.63
Group III (SDF + Etch-and-Rinse)	25	22.96	2.71
Group IV (SDF + Self-Etch)	25	17.85	2.48

One-way ANOVA revealed a statistically significant difference in shear bond strength among the four groups ($F = 28.64$, $p < 0.001$).

Table 2. One-Way ANOVA for Shear Bond Strength

Source	Sum of Squares	df	Mean Square	F-value	p-value
Between Groups	689.12	3	229.71	28.64	<0.001*
Within Groups	770.18	96	8.02		
Total	1459.30	99			

*Statistically significant

Post Hoc Comparison

Tukey's post hoc analysis demonstrated that Group I exhibited significantly higher bond strength than Groups II and IV ($p < 0.001$). Group III showed significantly greater bond strength than Group IV ($p < 0.001$) but remained significantly lower than Group I ($p = 0.028$). No statistically significant difference was observed between Groups II and III ($p = 0.082$).

Table 3. Tukey's Post Hoc Analysis

Comparison	Mean Difference (MPa)	p-value
Group I vs Group II	4.41	<0.001*
Group I vs Group III	1.86	0.028*
Group I vs Group IV	6.97	<0.001*
Group II vs Group III	2.55	0.082
Group II vs Group IV	2.56	0.014*
Group III vs Group IV	5.11	<0.001*

*Statistically significant

Failure Mode Analysis

The distribution of failure patterns differed among the groups. Adhesive failures were most prevalent in Group IV (56.0%), whereas mixed failures predominated in Groups I and III. Cohesive failures within enamel and composite were comparatively less frequent across all groups.

Table 4. Failure Mode Distribution

Failure Mode	Group I n (%)	Group II n (%)	Group III n (%)	Group IV n (%)
Adhesive Failure	5 (20.0)	10 (40.0)	7 (28.0)	14 (56.0)
Cohesive in Composite	3 (12.0)	2 (8.0)	3 (12.0)	1 (4.0)
Cohesive in Enamel	2 (8.0)	1 (4.0)	2 (8.0)	1 (4.0)
Mixed Failure	15 (60.0)	12 (48.0)	13 (52.0)	9 (36.0)

Chi-square analysis demonstrated a statistically significant difference in failure mode distribution among the groups ($\chi^2 = 12.74$, $p = 0.047$).

Summary of Findings

After thermocycling and six months of water storage, specimens bonded using the etch-and-rinse strategy exhibited significantly greater bond durability than those bonded using the self-etch approach. SDF pretreatment resulted in a moderate reduction in bond strength; however, the etch-and-rinse universal adhesive strategy maintained clinically acceptable bond values and outperformed the self-etch strategy. The lowest bond durability and highest frequency of adhesive failures were observed in the SDF-treated self-etch group, indicating greater susceptibility to interfacial degradation following aging.

DISCUSSION

The present study evaluated the long-term durability of composite bonding to Silver Diamine Fluoride (SDF)-treated enamel using universal adhesives applied in etch-and-rinse and self-etch modes following thermocycling and six months of water storage. The findings demonstrated that the etch-and-rinse adhesive strategy produced significantly higher bond strength values than the self-etch approach. Although SDF pretreatment resulted in a reduction in bond strength compared with untreated controls, the etch-and-rinse protocol maintained clinically acceptable bond durability after aging. These findings suggest that phosphoric acid conditioning effectively overcomes many of the adverse effects associated with SDF-induced surface modifications.

The superior performance of the etch-and-rinse groups may be attributed to the removal of silver-containing precipitates and the creation of a more retentive enamel surface through phosphoric acid etching. SDF application produces deposits of silver phosphate and calcium fluoride that can partially occlude enamel microporosities and interfere with resin infiltration. By dissolving these superficial deposits and increasing enamel surface roughness, phosphoric acid enhances micromechanical interlocking and promotes a stronger adhesive interface. In contrast, the milder acidic monomers present in self-etch universal adhesives may not adequately penetrate the altered enamel surface, thereby compromising resin tag formation and long-term bond stability.

The findings of the present study are consistent with those reported by **Jabbour et al. (2023) [29]**, who investigated the effect of phosphoric acid etching on composite bonding to SDF-treated enamel. The authors observed that phosphoric acid etching significantly improved bond strength and partially restored the adhesive performance of SDF-treated enamel to levels comparable with untreated controls. Their study emphasized the importance of enamel conditioning following SDF application, supporting the present observation that etch-and-rinse adhesives provide superior bonding performance compared with self-etch systems.

Similarly, **Markham et al. (2020)** [30] evaluated the effect of 38% SDF on the bond stability of universal adhesives and found that SDF-treated enamel exhibited significantly lower bond strength when bonded using self-etch protocols. The authors suggested that residual silver-containing reaction products interfered with adhesive infiltration and resin polymerization. These findings closely parallel the results of the current investigation, in which the SDF-treated self-etch group demonstrated the lowest bond strength values and the highest proportion of adhesive failures after thermocycling and prolonged water storage.

A systematic review conducted by **Jiang et al. (2020)** [31] examined the influence of SDF on the bond strength of restorative materials and adhesives. The review highlighted considerable variability among studies but concluded that the effect of SDF on bonding largely depends on the adhesive strategy employed. The authors noted that phosphoric acid etching frequently improved bonding outcomes, whereas self-etch approaches were associated with less predictable results. The present study further substantiates these conclusions by demonstrating superior long-term durability of the etch-and-rinse approach following artificial aging.

The results also corroborate the findings of **Beltrami et al. (2016)**, [32] who reported significantly greater enamel bond strength when universal adhesives were used in conjunction with phosphoric acid etching. Their investigation demonstrated that selective enamel etching enhanced micromechanical retention and improved adhesive performance. Although their study was not performed on SDF-treated enamel, the underlying bonding mechanism supports the current findings and highlights the benefits of phosphoric acid pretreatment in achieving durable enamel adhesion.

Likewise, **Antoniazzi et al. (2016)** [33] demonstrated that selective phosphoric acid etching significantly improved the bond strength of universal adhesive systems to enamel compared with self-etch application alone. The authors attributed this enhancement to improved enamel demineralization and greater resin penetration into interprismatic spaces. The superior bond durability observed in the SDF-treated etch-and-rinse group of the present study may be explained by similar mechanisms, particularly after prolonged aging challenges.

Thermocycling and extended water storage were employed in the present study to simulate oral aging conditions and evaluate bond durability rather than merely immediate bond strength. Repeated thermal fluctuations generate stresses at the tooth-restoration interface because of differences in coefficients of thermal expansion between enamel, adhesive, and composite resin. Simultaneously, prolonged water exposure promotes hydrolytic degradation of resin polymers and adhesive interfaces. The significant reduction in bond strength observed in the self-etch groups following aging indicates that these adhesive interfaces were more susceptible to degradation than those produced by etch-and-rinse protocols.

Failure mode analysis further supported the bond strength findings. Mixed failures predominated in the etch-and-rinse groups, indicating the formation of a stronger and more stable adhesive interface capable of withstanding aging stresses. In contrast, adhesive failures were more frequent in the self-etch groups, particularly in SDF-treated specimens, suggesting inadequate resin infiltration and weaker interfacial integrity. These observations reinforce the importance of proper enamel conditioning when restoring teeth previously treated with SDF.

Overall, the present study demonstrates that although SDF pretreatment can negatively influence enamel bonding, the use of universal adhesives in etch-and-rinse mode effectively minimizes this effect and provides superior long-term bond durability compared with self-etch application. Therefore, clinicians utilizing SDF as part of minimally invasive caries management strategies should consider phosphoric acid etching prior to adhesive placement to optimize restoration longevity and clinical success.

Limitations

The present study has certain limitations that should be considered while interpreting the findings. As an in vitro investigation, it could not fully replicate the complex biological and mechanical conditions of the oral environment, including salivary dynamics, masticatory forces, pH fluctuations, and biofilm activity. Although thermocycling and six months of water storage were used to simulate aging, these methods may not completely reflect long-term clinical performance. Only one concentration of silver diamine fluoride (38%), one universal adhesive system, and one composite resin material were evaluated, which may limit the generalizability of the results to other commercially available products. Additionally, bond durability was assessed using shear bond strength testing alone, without complementary microscopic analyses such as scanning electron microscopy or nanoleakage evaluation. Therefore, further long-term clinical studies and laboratory investigations involving different adhesive systems, restorative materials, and aging protocols are recommended to validate the present findings.

CONCLUSION

Within the limitations of this in vitro study, Silver Diamine Fluoride (SDF) pretreatment adversely affected the long-term bond strength of composite restorations to enamel. However, the use of a universal adhesive in etch-and-rinse mode significantly improved bond durability compared with the self-etch approach following thermocycling and extended water storage. Phosphoric acid etching effectively enhanced resin infiltration and reduced the negative influence of SDF-induced surface alterations. The SDF-treated self-etch group exhibited the lowest bond strength and highest incidence of adhesive failures after aging. Therefore, the etch-and-rinse universal adhesive strategy appears to be the preferred bonding protocol for achieving durable composite adhesion to SDF-treated enamel.

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