

The Effect of Concentration Based on Etching Time of Hydrofluoric Acid on Shear-Bond Strength of Porcelain Repair Material on Cohesive Fractures of Porcelain Fused to Metal Restoration

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Abstract

This research investigates the pivotal role of hydrofluoric acid (HF) concentrations and application times in optimizing the adhesive strength between feldspathic porcelain from Vita VMK Master and Ceromer composite resin in porcelain fused to metal (PFM) restorations. Renowned for their strength, durability, biocompatibility, and aesthetic appeal, PFM restorations require precise material manipulation to achieve optimal outcomes.

This present study examines how 9%, 9.5%, and 10% HF concentrations, applied for durations of 30, 60, 90, 120, and 150 seconds, influence the microstructural changes on the surface of feldspathic porcelain samples, affecting its cohesive fracture restoration properties with the Ceromer composite resin. A total of 64 cylindrical feldspathic porcelain samples, each measuring 7 mm in diameter and 3 mm in thickness, were treated with varying HF concentrations to investigate these effects. Our findings reveal that a 9.5% HF concentration applied for 60 seconds optimizes the etching process, achieving the highest adhesive strength by balancing etch depth with surface integrity, thereby facilitating a stronger bond with repair materials in PFM restorations.

The interaction between HF and the porcelain's glass matrix is critical, as HF's ability to etch the surface by dissolving the glass matrix results in surface roughness and porosity, both of which are instrumental in enhancing adhesive strength. Based on scanning electron microscopy (SEM) and atomic force microscopy (AFM) for a detailed analysis of the etching patterns and statistical tests to evaluate the shear-bond strengths., the results conclusively demonstrated that a 9.5% concentration of HF for 60 seconds represents the most effective treatment protocol, underscoring the critical influence of HF's chemical properties on the adhesive outcomes in PFM restorations.

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Introduction

Porcelain-fused-to-metal (PFM) restorations are a prevalent choice in fixed dental prosthetics, valued for their durable masticatory functions and aesthetic appeal. Such restorations are particularly popular for crowns and bridges, showing a remarkable ten-year success rate of about 95%.¹ Studies have reported encouraging survival rates for PFM restorations—98% after five years, 97% after ten years, and 85% after

fifteen years of intraoral usage.² PFM restorations offer several advantages, including good color stability, radiopacity, and commendable resistance to pressure and abrasion. However, they also have their drawbacks. The porcelain layer, despite its strength, is susceptible to brittleness, low tensile strength, and a high modulus of elasticity, making it prone to fractures under load.³ Clinical studies have indicated that the prevalence of porcelain fractures ranges between 5-10% for uses extending beyond ten years. Specific studies have delineated the occurrence of fractures, with a majority (65%) happening in the anterior region, followed by other areas such as the labial (6%), buccal (27%), incisal (5%), and occlusal (8%) regions. An analysis of 1,192 fractures revealed that 3% occurred in the PFM porcelain layer, predominantly on the maxillary

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labial surface (75%). When examining the failure of PFM restorations, it's observed that after five months of usage, metal fractures were at 6%, chipping at 13%, and cracked porcelain surfaces at 25%. Particularly in the anterior region, such fractures are treated as emergencies, necessitating immediate intervention through direct restoration techniques.⁴⁻⁶

The direct (intraoral) repair technique, employing composite resin, is a preferred method in dental restoration for its cost-effectiveness, time efficiency, and ease of application, particularly in urgent cases of porcelain fractures in anterior teeth. This technique's immediate applicability in a clinical setting makes it a valuable option.^{6,7} However, it faces limitations, such as reduced aesthetic appeal, diminished quality over time, and decreased strength compared to other methods. To overcome these limitations, surface treatment of the porcelain is crucial to enhance the adhesive bond between the porcelain and composite resin. This is achieved by creating a mechanical interlock at the micro-level between the two materials.^{8,9} Hydrofluoric acid (HF) is considered the gold standard for this surface treatment, not merely for its penetration but for its chemical etching effect on the porcelain surface, which significantly improves the micro-mechanical retention for the composite resin. Despite its effectiveness, HF's toxicity necessitates caution; its application should be minimal yet sufficient to ensure a robust adhesive bond, minimizing risks to oral tissues and preventing irritation, burns, and necrosis.^{9,10}

Hydrofluoric acid (HF) is commonly employed as a surface treatment agent, but its optimal usage is not entirely established. The choice of HF concentration and application time influences the bonding strength. Lower concentrations require a longer application time to achieve significant bond strength compared to higher concentrations.^{11,12} Research shows that a 10% HF concentration yields the highest bond strength, supported by studies recommending a 9-10% HF concentration for optimal bond strength within a brief period (1-2 minutes).¹³ Nonetheless, the application timing is crucial, as excessively short or long exposure can weaken the bond between the porcelain and the repairing material.¹⁴ Prolonged exposure does not necessarily enhance the bond strength and might even deteriorate it, causing surface cracks on the

porcelain, increasing the likelihood of fractures. Thus, the concentration and timing of HF application need careful consideration to maintain the integrity and strength of the restored surface.¹⁵

The bonding strength in dental restorations is influenced by the choice of composite resin repair material used. A higher filler content in the composite resin material tends to enhance the mechanical and physical properties, improving the overall bonding strength. Emerging technologies have introduced ceromers, initially used in direct restorations, as suitable materials for porcelain repair. In prosthodontics, ceromers are becoming essential due to their satisfactory bonding, aesthetic appeal, stability, resistance to shear forces, low polymerization shrinkage, and longevity. However, ceromers necessitate specialized curing tools for their application.¹⁶ Currently, there is limited research on the bonding strength between porcelain and ceromer composite resin repair materials, particularly regarding the application of HF. To expedite the intraoral application process without compromising the bond strength between porcelain and the repair material, researchers are investigating various HF concentrations and application times. They are exploring the use of 9%, 9.5%, and 10% HF concentrations applied for durations ranging from 30 to 150 seconds, aiming to understand their impact on cohesive behavior and bond integrity between the porcelain and repair materials. This study hypothesizes that the application of hydrofluoric acid (HF) as a surface treatment significantly enhances the adhesive strength between porcelain and composite resin in PFM restorations by creating a chemically etched surface that facilitates mechanical interlocking, thereby enhancing the bond strength between the porcelain and composite resin.

Materials and methods

Materials

This study utilized a carefully selected set of materials to investigate the effects of hydrofluoric acid (HF) concentrations on the adhesive strength between porcelain and composite resin in porcelain-fused-to-metal (PFM) restorations. Cylindrical metal matrix molds were employed to create porcelain specimens, each with a diameter of 7mm and a

thickness of 3mm. The porcelain, in powder and liquid forms from Vita VMK Master and chosen in the A3 color range, was mixed with distilled water to achieve the correct consistency for experimentation. Three different concentrations of hydrofluoric acid gel—9% (Ultradent™ Porcelain Etch), 9.5% (PPH CERKAMED Yellow Porcelain Etch), and 10% (DENTSCARE LTDA Condac Porcelana)—were used for surface treatment. To improve bonding properties, Silane (Ultradent™) and 3M ESPE Single Bond Universal Adhesive were applied. Ceromer composite resin with super high density, sourced from Ceramage, Japan, and selected in the A2 color range, was used to meet the experimental objectives. All chemical reagents were of analytical grade to ensure the integrity of the study's findings.

Study Design

was structured as an experimental laboratory study, employing a factorial design to explore the effects of multiple independent variables (HF concentrations and application durations) on a single dependent variable (adhesive strength). The sample size for this research can be determined using Federer's formula ¹⁷: $(t - 1)(r - 1) \geq 16$, where:

t = Number of treatment groups
 r = Number of replications/samples per group

$$(15 - 1)(r - 1) \geq 16$$

$$14(r - 1) \geq 16$$

$$14r - 14 \geq 16$$

$$14r \geq 30$$

$$r \geq 30/14 \rightarrow 2,14$$

Based on the calculations, the minimum sample size required for each group is ≥ 2 samples. To prevent bias, it was decided to increase this number to 4 samples per group, making a total of 64 samples used in the study.

Porcelain Specimen Preparation

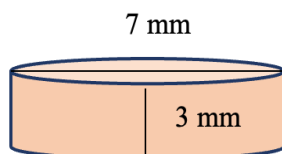


Figure 1. Dimension of Porcelain Block Sample.

Cylindrical feldspathic porcelain samples conforming to ISO 11405 standards were created using the described metal matrix molds.¹⁶ Figure 1 illustrates the porcelain block. The porcelain mixture was placed into these molds with a brush, and density was enhanced by vibration for 60 seconds. Samples were then heat-treated in a porcelain oven following the manufacturer's temperature protocol, preparing them for surface treatment.

Procedure of Surface Treatment with HF Variations

For the surface treatment process, various concentrations of hydrofluoric acid (HF), specifically 9%, 9.5%, and 10%, were applied, each undergoing a precise measurement of pH levels. The specimens were thoroughly cleaned and dried before and after the application of HF. Different HF concentrations were methodically applied to groups of four specimens, each subjected to varied treatment durations, ensuring that each specimen was meticulously treated and prepared.

Procedure Bonding Application

In the bonding application procedure, each porcelain surface underwent a systematic preparation process, starting with the application of acetone, followed by a layer of silane containing 3-MPS on the surface of all porcelain specimens with a brush and leave for 60 seconds at room temperature. Then proceed by applying a layer of bonding solution (single bond universal) on all surfaces of the porcelain specimens with a microbrush. The specimens were then finalized with a curing process, using a light cure device for 20 seconds to complete the preparation.

Characterizations and Statistical Analysis

Post-treatment, the surface condition of the samples was analyzed using Scanning Electron Microscopy (SEM) (Prisma E SEM, Thermo Fisher Scientific, U.S) at a 1500x magnification and roughness measurements utilizing Atomic Force Microscopy (AFM) (Nanosurf easyScan 2 Controller., Ltd., Japan) was carried out to assess etching patterns. Adhesive shear strength was measured using a Universal Testing Machine (UTM) (Tokyo Testing Machine MFG Co., Switzerland), equipped with a 10 kN load cell and operating at a crosshead speed of 0.5 mm/minute, in accordance with ISO 11405 standards. Failure

types were analyzed using a stereomicroscope with 20 times magnification, ensuring detailed insight into the adherence performance. Statistical analysis included a univariate normality test and an ANOVA one-way test to evaluate the effects of HF concentrations and application times on adhesive strength.

Results

In this study, the samples were organized into four main categories: one control group and three experimental groups, each subjected to different hydrofluoric acid (HF) concentrations (9%, 9.5%, and 10%). Within these experimental groups, 20 samples were further segmented based on the duration of HF application, ranging from 30 to 150 seconds.

The acidity level, or pH, of each HF concentration was measured to assess its potential impact on the porcelain surface. Our analysis found that the pH levels varied with HF concentration: the 9.5% HF solution had a pH of 2.70, indicating a more acidic environment compared to the 9% HF solution, which had the highest pH among the groups. The most acidic solution was the 10% HF concentration, with a pH of 2.58.

Shear-bond strength, a critical measure of adhesive effectiveness, was evaluated across all groups using a Universal Testing Machine (UTM). The results demonstrated a range of adhesive strengths, reflecting the nuanced effects of HF concentration and application time on bonding. For example, samples treated with 9% HF for 30 seconds exhibited shear-bond strengths between 8.098 MPa and 8.256 MPa. This variability underscores the intricate relationship between HF concentration, etching duration, and their collective influence on the bond strength of the porcelain samples. Each experimental condition revealed distinct outcomes, signifying the pivotal role of both HF concentration and application time in determining the effectiveness of the adhesive bond.

Effect of 9, 9.5, and 10% Hydrofluoric Acid Concentration and Time

The study utilized a One-way ANOVA test to analyze the effect of varying HF concentrations (9%, 9.5%, and 10%) and application times on the shear-bond strength of ceromer resin composite repair materials. The control group showed a mean shear-bond

strength of 4.475 ± 0.153 MPa. For a 30-second application time, the highest mean shear-bond strength was observed with 9.5% HF concentration (10.276 ± 0.047 MPa), followed by 9% HF (8.173 ± 0.070 MPa), and the lowest with 10% HF (7.378 ± 0.023 MPa). The significant difference in shear-bond strengths among these concentrations was confirmed by a p-value of 0.0001 ($p < 0.05$), indicating a statistically significant impact of HF concentration and application time on the bond strength in cohesive fractures of Porcelain Fused to Metal restorations as seen in Table 1.

No	Concentration	Etching Time (s)	p
1.	Control	No treatment	0.462
2.	9%	30	0.853
3.		60	0.886
4.		90	0.456
5.		120	0.599
6.		150	0.179
7.	9.5%	30	0.624
8.		60	0.279
9.		90	0.130
10.		120	0.652
11.		150	0.172
12.	10%	30	0.626
13.		60	0.991
14.		90	0.433
15.		120	0.488
16.		150	0.779

Table 1. Normality Data.

Before applying the One-way ANOVA, it was essential to ensure that the data were normally distributed, as assessed by the Shapiro-Wilk normality test. This test is suitable for datasets with a smaller number of observations. The normality test results indicated that the control group and all treatment groups, across various HF concentrations and etching times, had p-values greater than 0.05, suggesting that the data within each group were normally distributed. Specifically, the control group had a p-value of 0.462, and treatment groups ranged from 0.179 to 0.991 across different concentrations and application times, allowing the One-way ANOVA analysis to proceed with confidence as seen in Table 2.

After conducting shear-bond strength tests on the samples, each one was further

examined to identify the type of failure that occurred. This analysis involved using scanning electron microscopy (SEM) at 500- and 1500-times magnification a stereo microscope at 20 times magnification and displayed in Figure 2 and 3 respectively. Meanwhile, the investigation of surface topology was carried out to determine the effect of HF to the surface roughness, and the AFM results are shown in Figure 4. The failures observed were categorized into three types: adhesive failure, occurring at the porcelain/bonding interface; cohesive failure, occurring within the composite material or porcelain itself without interface damage; and mixed failure, which involved both the interface and the material.

The analysis revealed distinct patterns of failure across different groups. In the control group, every sample (100%) exhibited mixed failure types, indicating that failures were both at the interface and within the materials. Conversely, in the groups treated with HF concentrations of 9%, 9.5%, and 10%, cohesive failures were predominant, with all samples (100%) showing this type of failure and no instances (0%) of mixed failures were recorded and presented in Table 3.

Adhesive and mixed failures were absent across all HF etching groups. The occurrence of cohesive failures in the HF-treated groups and mixed failures in the control group were clearly visible in the SEM images. Specifically, Figures 3(a) and (b) illustrate cohesive failure in the etched porcelain, indicating a failure within the material itself. Additionally, Figures 3d and 3e depict the mixed failure observed on the etched porcelain surface, highlighting the dual nature of these failures. Figure 4c showcases mixed failure in the control samples, where the fracture spans across two surfaces: one part occurring within the porcelain or composite resin and the other at the interface between the two materials.

HF Concentration	Failure Types			Frequency (n)
	Cohesive	Mixed	Adhesives	
Control	0	4	0	4
9%	20	0	0	20
9.5%	20	0	0	20
10%	11	9	0	20
Total	51	9	0	64

Table 3. HF Etching Failure Types based on concentration.

Discussion

Hydrofluoric acid (HF) surface treatment currently stands as the gold standard in dental practice due to its ability to significantly increase the bond strength of porcelain restorations. When HF comes into contact with porcelain, it selectively dissolves the glass matrix because of fluoride's higher affinity to silicon compared to oxygen, enabling an ionic attack on the silanol bonds in glass ceramic.¹⁸ However, it must be noted that HF is toxic and can be harmful to tissues. Contact with oral tissues can lead to irritation, burns, and necrosis. Hence, controlling the contact of HF with oral tissues is crucial, especially during prolonged use, which underscores the preference for shorter application times.

A ceromer-based composite resin was used as a repair material in this study, taking advantage of its recent progress for both direct and indirect restorations. The silane used in dental applications, and that includes specifically 3-methacryloxypropyltrimethoxysilane, acting as a chemical binder that connects the organic (resin-based materials) with the inorganic (porcelain).¹⁹ Prior to shear-bond testing, all samples were incubated in distilled water at 37°C for 24 hours according to ISO 11405 standards, simulating short-term oral cavity conditions to distinguish materials resistant to wet environments. The shear-bond was assessed against different HF concentrations over application times of 30, 60, 90, 120, and 150 seconds between the ceromer composite resin and feldspathic porcelain. The shear-bond strength test was employed as it is commonly used, simple in specimen preparation, and cost-effective.

The application of 9%, 9.5%, and 10% HF for 30 seconds on cohesive fractures showed significant differences in shear-bond strength in this study. The average shear-bond strength for 9% HF-etched samples was 8.173 MPa, for 9.5% HF- was 10.276 MPa, and for 10% HF- was 7.378 MPa, with the lowest shear-bond strength observed in the 10% HF etching, yet still higher than the control group at 4.475 MPa. SEM and AFM observations revealed that 9%, 9.5%, and 10% HF etching produced different morphological patterns on the porcelain surface. A 30-second application of 9% HF created a porous pattern due to the dissolution of the glass

matrix by HF. Such surface roughness, as evidenced by the application of 9.5% HF, has been shown to improve shear-bond strength. Conversely, 10% HF application led to a decrease in bond strength due to a more dominant crack pattern than the pore pattern, with the higher HF concentration causing more extensive and deeper matrix dissolution, increasing defects and the risk of crack propagation, which may weaken the porcelain surface.^{20,21}

At a 60-second etching time, the average shear-bond strength for 9% HF-etched samples was 8.373 MPa, for 9.5% HF it was 11.990 MPa, and for 10% HF it dropped to 6.231 MPa. The lowest bond strength was recorded with the 10% HF etching, yet this was still above the control group's 4.475 MPa. SEM and AFM images revealed a smooth control sample surface with polishing scratch patterns, whereas surfaces etched with 9%, 9.5%, and 10% HF showed increased roughness. Each concentration resulted in a unique surface topography, defined by the quantity and size of the patterns produced post-etching. The 9% HF etching at 60 seconds led to more pronounced pore and groove patterns on the porcelain surface, contributing to a rougher surface due to the increased dissolution of the glass matrix. A study reported that etching up to 60 seconds enhances surface irregularities and undercuts from dissolved ceramic particles, essential for adequate micromechanical retention.²⁰ The application of 9.5% HF for 60 seconds resulted in even rougher surfaces compared to 9% HF, with more dominant and larger pore and groove patterns visible in SEM and AFM images, facilitating better composite resin penetration.

Conversely, 10% HF for 60 seconds produced smoother morphological patterns with wider crack formations, which led to decreased bond strength when application time was increased to 60 seconds. The presence of cracks indicates potential material weakness, and a smooth surface is also undesirable since it lacks micro-retention, leading to lower bond strength. One of the reasons on this lower bond-strength is due to smoother surfaces (less irregularity), contributing to reduced shear-bond strength.²² Furthermore, 10% HF may cause deeper and more extensive glass phase dissolution, jeopardizing material integrity due to an increased defect population within the material's

internal microstructure, potentially heightening crack propagation risk under stress²³. Similarly, a previous study also reported that using 10% HF for 60 seconds results in greater and deeper dissolution, amplifying damage and crack propagation risk, thereby diminishing the bond strength between porcelain and composite resin material.²⁴

The findings of cohesive failure type suggest a good bond strength between porcelain and ceromer repair material. Failure analysis on 9% HF etching revealed cohesive failures. Although failure type analysis alone is not sufficient to evaluate the bond produced, the minimum bond strength required for porcelain repair material is between 8-9 MPa.²⁵ Therefore, HF at a concentration of 9% (8.373 MPa) and 9.5% (11.990 MPa) meet the desired values. In contrast, the 10% HF (6.231 MPa) did not reach the desired minimum value. Thus, the bond strength with 9% and 9.5% HF application for 60 seconds achieved the minimum value with the desired cohesive failure type for cohesive fracture repair in PFM restorations.

Etching with 9% HF for 90 seconds showed an increased dissolution of the glass matrix. The morphology of pores and grooves represents the initial form of further dissolution in the glass matrix. The dissolution of the glass matrix in porcelain occurs when HF etching operates not only superficially but also in three dimensions. Therefore, the stronger the etching protocol (higher etching concentration and longer application time), the deeper HF can dissolve the glass matrix (SiO₂) within the glass phase.^{23,26} Fluoride acid can also dissolve leucite crystals depending on the concentration of HF and the time of application. The results of etching with 9.5% HF for 90 seconds showed an increase in surface roughness, which affects the shear-bond strength. The images of the porcelain surface etched with 9.5% HF revealed enlarged pore patterns bounded by needle-shaped crystals.

The average shear-bond strength for 9.5% HF remains higher compared to 9% HF over 90 seconds. This is due to the morphology of surface roughness etched with 9.5% HF being more uniform compared to 9% HF. HF can influence the resulting morphological structure to be larger and reduce the flexural strength. Etching with 10% HF for 90 seconds produced a morphology of crack patterns with extensive smooth surfaces. The resulting average shear-

bond strength decreased when compared to the application for 60 seconds. This may be due to the increased area of smooth surface compared to the number of cracks, thereby reducing retention in the composite material. Failure analysis for etching over 90 seconds showed that with 9% HF etch and 9.5% HF etch, 100% cohesive failure was obtained. For 10% HF etching, a cohesive failure type of 75% and 25% mixed was obtained. Cohesive and mixed failure types are expected because they indicate a good bond strength between porcelain and the ceromer repair material.

The average bond strength for 9% HF etch was 8.458 MPa, for 9.5% HF it was 10.321 MPa, and for 10% HF it was 5.359 MPa over 150 seconds. The lowest shear-bond strength was produced with 10% HF etching; however, this value was higher compared to the control group at 4.475 MPa. Based on the morphology images from SEM and AFM, the etched surfaces were rougher compared to the control surfaces. The porcelain surfaces etched with 9%, 9.5%, and 10% HF concentrations resulted in different morphological patterns on the porcelain surface. Etching with 9% HF for 120 seconds, in addition to the morphology of pore and crack patterns seen in SEM and AFM, showed the presence of reaction products from the acid reaction on the ceramic surface. HF applied for a certain time will produce less adherent fluorosilicate reaction products (Na, K, Ca, and Al) on the porcelain surface after treatment. The reaction products can weaken the resin-porcelain bond and may cause clinical failure. The application of 9.5% HF for 120 seconds showed a higher average shear-bond strength compared to 9% HF for 120 seconds. The morphology pattern on the porcelain surface etched with 9.5% HF showed a rougher pattern. This is evident with more dominant pore and groove morphology in SEM. Whereas 9% HF for 120 seconds, in addition to producing a rough surface, also showed the presence of reaction products on the porcelain surface.

A stereomicroscope was used to examine the mixed failure type at the interface in the control with 10% hydrofluoric acid (HF) using a stereomicroscope did not show a different pattern. The difference became apparent when using Scanning Electron Microscopy (SEM), where the surface of the porcelain etched with 10% HF displayed the presence of residual

bonding agent. This indicates the occurrence of interlocking of the bonding agent with the rough porcelain surface, thereby resulting in a micromechanical bond.²⁴ A different result was shown on the control surface with mixed interface failure type, where a smoother surface was observed. This suggests that the micromechanical bond formed is weak due to the non-rough surface, which could affect the resulting bond strength. The increased mixed failure type with 10% HF indicates a decrease in bond strength. The increase in mixed failure type suggests that the strength of the bonded material and the adhesive are balanced. Although mixed failure type is not included in the desired failure types, mixed failures can still be acceptable in determining bond strength.²⁷

The average bond strength for 9% HF-etched samples was 9.228 MPa, for 9.5% HF it was 10.683 MPa, and for 10% HF it was 5.093 MPa over 150 seconds. The lowest shear-bond strength was observed with 10% HF etching; however, this value was higher compared to the control group at 4.475 MPa. Based on the SEM and AFM morphology images, the etched surfaces were rougher compared to the control surfaces. With the use of 10% HF, the morphology of the porcelain surface showed a pattern of cracks more dominant than pores. The cracks produced are a form of defect that can reduce the bond strength between the repair material and the porcelain. In one study, the use of 10% HF can lead to greater and deeper solubility, increasing damage that can result in the risk of crack propagation.²⁴ Such conditions lower the bond strength between porcelain and the resin composite material. SEM analysis of 10% HF also revealed images of reaction products on the surface. The acid etching reaction with hydrofluoric acid that results in poorly adherent reaction products can weaken the resin-porcelain bond and may cause clinical failure.²⁴

The bond strength is affected by the surface roughness of porcelain produced by HF etching. Hydrofluoric acid in contact with porcelain selectively dissolves the glass matrix contained within it. The dissolution of the glass matrix is a result of a chemical reaction that transforms it from a solid to a liquid state. As a result of the glass matrix dissolution, the porcelain surface becomes porous, creating roughness. With 10% HF, a 100% mixed failure

type was obtained. Examination of the mixed failure type at the interface with the control using 10% HF on SEM (Figure 3 and 4) shows a different pattern. The surface of the porcelain etched with 10% HF shows the presence of residual bonding agent, whereas the control surface appears smoother.

Despite the observed variability in bond strength attributed to different HF concentrations and the limitations arising from using hydrofluoric acid of different brands—which could affect the consistency of application due to variations in viscosity—the study provides valuable insights into the nuanced effects of HF etching on porcelain surfaces. Specifically, it highlights the critical role of HF concentration in achieving optimal surface roughness for effective bonding, with 9.5% HF demonstrating superior bond strength. This finding challenges the prevailing assumption that higher concentrations of HF invariably lead to better etching outcomes, revealing a complex relationship between HF concentration, surface roughness, and bond strength. Moreover, the identification of mixed failure types as a predominant failure mode under certain conditions offers a nuanced understanding of failure mechanisms in porcelain repair, contributing to the broader discourse on dental restoration durability. Future research could explore the long-term effects of different HF concentrations on bond durability, incorporating a broader range of HF brands to assess the impact of product-specific properties on etching effectiveness. Additionally, extending the simulation of oral cavity conditions to include long-term aging processes, such as thermocycling, would offer deeper insights into the durability of the bond over time. Such studies could ultimately lead to the development of standardized etching protocols that maximize restoration longevity while minimizing the risk of

failure, thereby enhancing patient outcomes in dental restoration.

Conclusion

In conclusion, this research has elucidated the significant impact of hydrofluoric acid concentration on the adhesive strength of porcelain repair materials in cohesive fracture restoration of Porcelain Fused to Metal. The study determined that concentrations of 9%, 9.5%, and 10% hydrofluoric acid consistently influenced the bonding strength across various application durations—30, 60, 90, 120, and 150 seconds—with a compelling p-value of 0.0001, firmly indicating statistical significance ($p < 0.05$). A pivotal clinical implication derived from these findings is that a 9.5% hydrofluoric acid concentration for a minimal application time of 60 seconds yields the optimal adhesive strength for bonding porcelain to repair materials. This insight offers a valuable guideline for clinical practices, balancing efficacy and efficiency in porcelain restoration procedures, and can potentially enhance the longevity and success of Porcelain Fused to Metal restorations.

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Declaration of Interest

We declare there is no conflict of interest in this study.

No	Shear-bond strength (Mpa)															
	Control	30 seconds			60 seconds			90 seconds			120 seconds			150 seconds		
		9%	9.5%	10%	9%	9.5%	10%	9%	9.5%	10%	9%	9.5%	10%	9%	9.5%	10%
1	4.328*	8.203	10.291	7.377	8.346	11.931*	6.377**	8.903	9.016*	5.551	8.516	10.533**	5.310	9.152*	10.913	5.275**
2	4.367	8.256**	10.232*	7.411**	8.332*	11.943	6.269	8.979**	9.858	5.468*	8.427	10.175*	5.292*	9.205	10.997**	5.154
3	4.651**	8.137	10.246	7.371	8.398	11.990	6.188	8.841*	9.893**	5.590	8.540**	10.238	5.395	9.316**	10.749	5.006
4	4.557	8.098*	10.338**	7.355*	8.417**	12.099**	6.093*	8.915	9.150	5.606**	8.352*	10.338	5.441**	9.239	10.076*	4.937*

Table 2. Average Value of Shear Bond Strength Based on HF Concentration Group and Acid Etching Time Used.

Note: *the smallest; ** the biggest

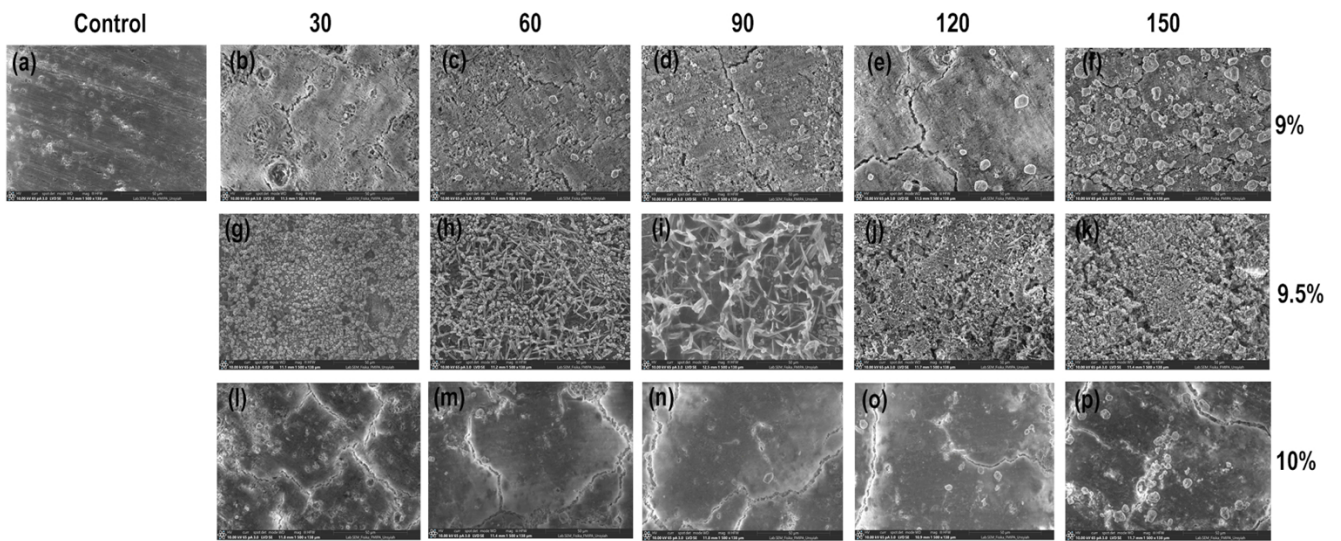


Figure 2. SEM image (1500x magnification) of the porcelain surface a. control. Application of 9% HF concentration during; b. 30s; c. 60s; d. 90s; e. 120s; and f. 150s. In etching 9.5% HF concentration during, g. 30s; h. 60s; i. 90s; j. 120s and k. 150s. In etching the HF concentration is 10% during; l. 30s; m. 60s; n. 90s; o. 120s; and p. 150s.

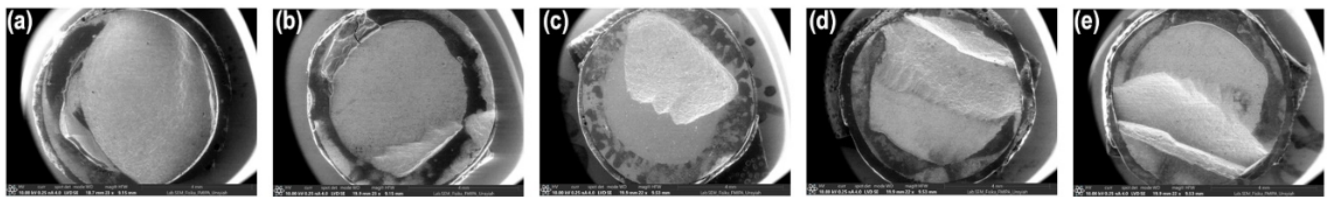


Figure 3. SEM image magnification 50x Type of failure in the sample, a. and b. cohesive failure type in the etched porcelain. c. The mixed failure in the control, d. and e. the mixed failure in the etched porcelain surface.

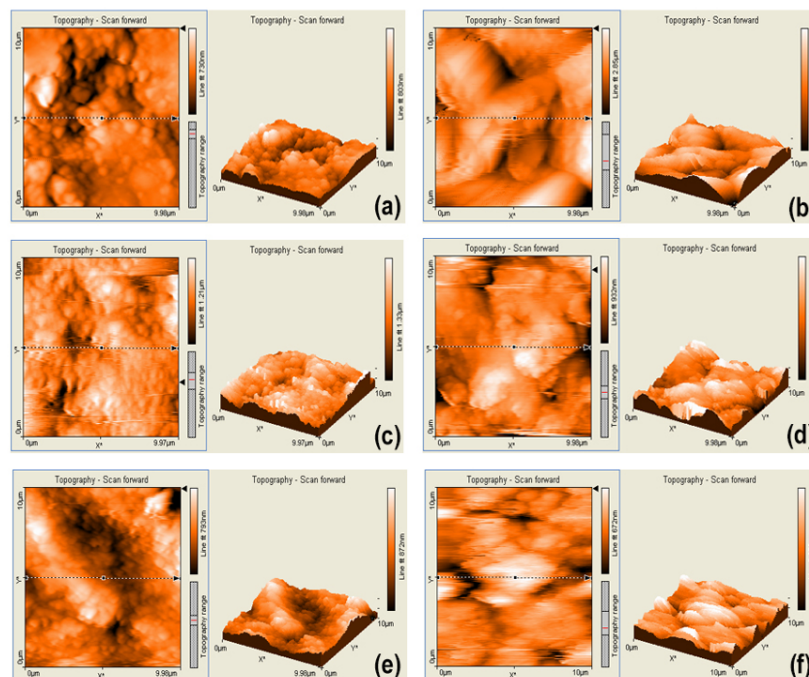


Figure 4. AFM image of the porcelain surface a. control. Application of 9.5% HF concentration during; b. 30s; c. 60s; d. 90s; e. 120s; and f. 150s.

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