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Effect of thermocycling and surface treatments on the bond strength of a hybrid PICN ceramic

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ABSTRACT

This study evaluated the bond strength of a hybrid PICN ceramic before and after being subjected to 5000 cycles of thermocycling. PICNs are a promising alternative in the field of CAD/CAM dental restorations; however, their adhesive behavior with chemical surface treatments such as silane and 10-MDP, and micromechanical treatments such as hydrofluoric acid and sandblasting is not yet fully understood. ***Aim:*** *To compare the bond strength of a hybrid PICN ceramic treated with different surface protocols before and after a thermocycling process.* ***Materials and methods:*** *An in-vitro experimental study was conducted. Forty PICN ceramic slices were prepared and divided into four groups. Each group received a specific surface treatment (sandblasting or acid etching) and a different adhesive technique (silane + adhesive or adhesive only). Composite cylinders of 3 mm diameter mm were bonded to each slice, and shear strength was measured on a universal testing machine immediately, and after a period of thermocycling. Statistics: Data were analyzed by ANOVA followed by Tukey's post hoc test. All statistics were analyzed with a 95% confidence interval.* ***Results:*** *The group subjected to sandblasting followed by silane and universal adhesive achieved the highest adhesion values, both immediately and after thermocycling (16.3 MPa and 11.2 MPa, respectively), and the group subjected to hydrofluoric acid etching and adhesive had the lowest values, both immediately and after thermocycling (8.6 MPa and 5.4MPa).* ***Conclusions:*** *Cementation of a hybrid ceramic treated with sandblasting, silane and a 10-MDP-based adhesive ensures high bond values, even when aged under hot, humid conditions. Thermocycling significantly reduced the adhesive strength in all groups, the decrease being more noticeable in those that did not include silane.*

Keywords: *tooth adhesion - surface - treatments - ceramics - hybrid*

Efecto del termociclado y tratamientos superficiales en la resistencia de adhesión de una cerámica híbrida PICN

RESUMEN

En este estudio se evaluó la fuerza de adhesión de una cerámica PICN híbrida, sometida a 5000 ciclos de termociclado. Las PICN representan una alternativa prometedora en el campo de las restauraciones dentales CAD/CAM; sin embargo, su comportamiento adhesivo con tratamientos de superficie químicos como el silano y la molécula de 10MDP y tratamientos micromecánicas como el uso de ácido fluorhídrico y arenado aún no se conoce por completo. ***Objetivo:*** *Comparar la fuerza de adhesión de una cerámica PICN híbrida tratada con diferentes protocolos de superficie antes y después de un proceso de termociclado.* ***Materiales y métodos:*** *Se realizó un estudio experimental in vitro en el que se prepararon 40 láminas de cerámica PICN y se dividieron en cuatro grupos. Cada grupo recibió un tratamiento superficial específico (chorro de arena o grabado ácido) y una técnica adhesiva distinta (silano + adhesivo o sólo adhesivo). Se adhirieron cilindros de composite de 3 mm de diámetro a cada lámina y se midió la resistencia al cizallamiento inmediatamente y posterior a un periodo de termociclado en una máquina de ensayos universales. Estadísticas: Los datos se analizaron mediante ANOVA seguido de la prueba post hoc de Tukey. Todas las estadísticas se analizaron con un intervalo de confianza del 95%.* ***Resultados:*** *El grupo que se sometió a arenado seguido de silano y adhesivo universal alcanzó los valores de adhesión más altos, tanto inmediatamente como después del termociclado (16,3 MPa y 11,2 MPa, respectivamente) y el grupo sometido al ataque de ácido fluorhídrico y adhesivo mostro los valores más bajos tanto inmediatamente como después del termociclado (8,6 MPa y 5,4MPa).* ***Conclusiones:*** *La cementación de una cerámica híbrida tratada con chorro de arena, silano y un adhesivo a base de 10-MDP garantiza altos valores de adhesión, incluso cuando envejece en condiciones de humedad y calor. El termociclado redujo significativamente la resistencia adhesiva en todos los grupos, siendo más notoria la disminución en los que no incluyeron silano.*

Palabras clave: *adhesión dental - superficie - tratamientos - cerámicas - híbrida*

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INTRODUCTION

Advances in CAD/CAM have led to the development of new dental ceramics such as VITA ENAMIC, also known as PICN (Polymer Infiltrated Ceramic Network). This is a hybrid ceramic composed of a porous ceramic matrix and a polymeric matrix, which combines the esthetics of ceramic with the strength of resin. Thanks to this unique combination, it offers superior optical and mechanical properties, providing a very natural esthetic appearance and withstanding masticatory forces[1-3]. According to the manufacturer[4], the versatility of VITA ENAMIC makes it suitable for a wide range of esthetic restorations in the anterior sector.

However, adhesion to resin cements is a critical factor in ensuring the durability of these restorations. May et al.[5] state that the long-term success of restorations depends on establishing a reliable bond between the restorative material and the luting agent. A proper luting technique can significantly reduce the risk of fractures and microleakage, and adequate adhesion of dental materials to the tooth structure and the interface ensures the long-term success of dental restorations.

Numerous studies have evaluated the efficacy of different surface treatments, such as sandblasting with aluminum oxide, etching with hydrofluoric acid, and applying silane agents and universal adhesives, especially those containing 10-MDP, known for its chemical affinity with ceramic phases[2,6-9]. However, discrepancies remain as to which protocol is best for hybrid ceramics such as VITA ENAMIC[5].

The aim of this study was to determine the bond strength of a hybrid ceramic to a resinous cement, comparing different surface treatments before and after thermocycling. Few studies include the factor of aging by thermocycling, a standardized technique that simulates oral thermal stress and evaluates bond durability over time.

The working hypothesis was that surface treatment with sandblasting, followed by silane and a 10-MDP-based universal adhesive would provide the highest bond strength to PICN hybrid ceramic, even after aging.

MATERIALS AND METHODS

The materials used in this study are described in Table 1.

Procedure

Sample size: A sample size of 40 was selected to guarantee adequate distribution of the data and minimize random error, considering a standard deviation of up to ± 3 (Table 1).

PICN specimens: Four 12x14x18 mm VITA Enamic blocks (VITA Zahnfabrik, Bad Säckingen, Germany) intended for CAD/CAM use were cut into slices 12 mm long, 14 mm wide and 1 mm thick using a cutting machine (Mini CNC XYZ26187, GreatSolutions) and a diamond cutting disc 0.5 mm thick with a 22 mm radius, to obtain 40 slices.

Surface standardization: To standardize the ceramic surfaces, they were sanded with 600, 1000 and 1200 grit silicon carbide sandpaper sheets for one minute each, following a uniform direction.

Composite resin cylinders: 80 cylinders were made of composite resin (Llis Composite, FGM, Joinville, Santa Catarina, Brazil) (Table 1) using an aluminum matrix with a perforation of 1.5 mm radius and 2 mm long, to ensure that they would all be the same size.

The 40 VITA Enamic slices were distributed randomly into the following 4 experimental groups (n=10):

- **AAC group:** Sandblasting + adhesive + cement
- **ASAC group:** Sandblasting + silane + adhesive + cement
- **AFAC group:** Hydrofluoric acid + adhesive + cement
- **AFSAC group:** hydrofluoric acid + silane + adhesive + cement

The specimens in groups AAC and ASAC were sandblasted with 50 µm Al_2O_3 particles (Bioart, Sao Paulo, Brazil) from a distance of 10 mm from the sandblaster nozzle (STD, Bio-Art, Sao Paulo, Brazil) for 15 s, applied perpendicularly over the entire surface at a pressure of 2 bars.

The specimens in groups AFAC and AFSAC were treated with 10% hydrofluoric acid for one minute, then washed with running water for 60 s, and dried with a jet of oil-free cold air (hair dryer).

Two resin cylinders were cemented to each surface-treated PICN slice. To do so, first, silane was applied in a thin layer with a microbrush for 20 s on half of the sandblasted slices (n=10) and half of the hydrofluoric acid-treated slices (n=10), and left to act for 60 s for its complete evaporation. Then, all 40 specimens were coated with an adhesive

Table 1. Materials used in the study

MATERIAL	MANUFACTURER	BATCH	COMPOSITION
VITA ENAMIC[4]	Vita Zahnfabrik (Alemania)	73340	Ceramic component (86% by weight and 75% by volume): Silicon dioxide (SiO2), aluminum oxide (Al2O3), sodium oxide (Na2O), potassium oxide (K2O), boron trioxide (B2O3), zirconium dioxide (ZrO2), calcium oxide (CaO). Polymeric component (14% by weight and 25% by volume): UDMA (urethane dimethacrylate), TEGMA (triethylene glycol dimethacrylate). [4]
SINGLE BOND UNIVERSAL[10]	3M ESPE (USA)	10604ª	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane. [10]
Silane MONOBOND-N (Universal primer)	Ivoclar Vivadent AG Liechtenstein	Z0226T	Alcoholic solution of silane methacrylate, phosphoric acid methacrylate and methacrylate sulfide.[27]
ALL CEM DUAL CEMENT[11]	FGM (Brazil)	190421	Base paste: Bis-GMA, Bis-EMA and TEGDMA, camphorquinone, co-initiators, Barium glass microparticles-aluminosilicates, silicon dioxide nanoparticles, inorganic pigments and preservatives. Catalyst paste: methacrylic monomers and dibenzoyl peroxide and stabilizers, Barium-aluminum glass microparticles. [11]
LIS[28] COMPOSITE	FGM (Brazil)	011021	Bis-GMA, Bis EMA, TEGDMA camphorquinone, and silane, micronized barium-aluminum silicate glass, pigments and nano silica.[28]
ALUMINUM OXIDE SAND	Bio Art	76584	50 µm aluminum oxide particles
CONDAC PORCELANA 10%	FGM	230622	10% Hydrofluoric Acid

containing 10-MDP, rubbed for 20 seconds on the surface and polymerized for 20 seconds with an LED light (Woodpecker). Subsequently, resin cement was applied to one end of each cylinder with a 0.5 mm diameter periodontal probe, and two cylinders attached to each PICN specimen. Excess cement was carefully removed so as not to move the cylinders, and then light-curing was performed using the Woodpecker LED B lamp (Woodpecker, Guilin, Guangxi, China).

Each group of test specimens (consisting of two cylinders) was divided into two subgroups. The first subgroup was subjected to shearing immediately, while the second subgroup was aged with 5000 cycles of thermocycling before shearing. The tests were performed on a universal testing machine (Muver/5053, Muver Cx Server Lite Software) at a speed of 1.0 mm/min (Fig. 1).

The shear strength data obtained in newtons were converted to MPa using the following formula:

$$RU = \frac{F}{a}$$

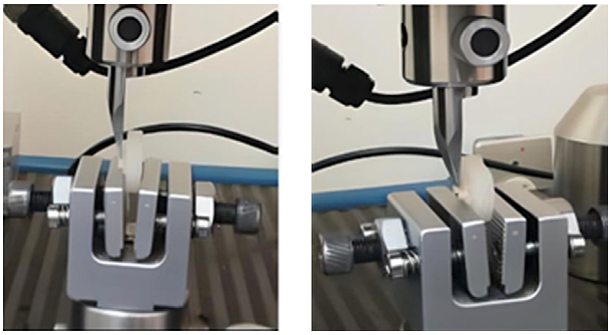

Fig. 1: Universal testing machine used to perform the shear tests.

where:

- RU = Bond strength.
- F = Maximum force at which the resin cylinder was peeled off, expressed in newtons
- a = Area of the base of the resin cylinder, expressed in mm².

The area of the cylinder was calculated using the formula:

$$A = \pi * r^2 \; [a=\pi r^\wedge 2]$$

where:

- π= 3.1416
- r2 = 2.25

Statistics:
Statistics were performed using Minitab 20. software (Minitab Statistical Software. State College. Pennsylvania. USA). Data were subjected to two-way ANOVA (surface treatment and aging) followed by Tukey's post hoc test with a value of $p \leq 0.05$, which was taken as a reference to confirm or rule out statistical differences between groups.

RESULTS

Four groups of n=10 were analyzed; each test specimen with 2 cylinders cemented on its surface, one tested immediately, and the other after simulated aging.
A total 80 ceramic-resin cement interfaces were evaluated. Table 2 shows the average results in MPa and the standard deviations of bond strength for each group.
Adhesion was highest in the immediate ASAC group, at 16.3 MPa. In this group, abrasion with aluminum particles was used as surface treatment, and silane was applied. The lowest value in the immediate group was in AAC, at 9.98 MPa.
Bond strength decreased in all groups after thermocycling (5000 cycles), which is equivalent to 6 months of clinical use in the mouth, according to Alnafaiy S et al[12].
After thermocycling, average bond strength was lowest in AAC, at 6.6 MPa, and highest in ASAC, at 11.2 MPa.
A two-way analysis of variance (ANOVA) was performed to evaluate the effect of surface treatment and aging on bond strength. Tukey's post hoc test was used to determine significant differences between groups (Table 3).
Tukey's analysis with a 95% confidence interval confirmed that after aging, there was no significant difference between groups, regardless of the surface treatment applied (Table 4).

Table 2. Mean and standard deviation of shear bond strength of the AAC, ASAC, AFAC, AFSAC groups tested immediately and after aging

GROUP	Immediate Bond strength (MPa)		Aged bond strength (MPa)	
	Mean	Std. Dev.	Mean	Std. Dev.
AAC	9.98	1.68	6.60	1.14
ASAC	16.30	3.36	11.20	2.19
AFAC	10.58	2.04	6.77	0.98
AFSAC	12.94	3.10	9.73	2.91

Table 3. Two-way ANOVA: showed that both the aging factor (p = 0.000) and the surface treatment factor (p < 0.001) had a significant effect on the differentiation of the groups. However, the interaction between surface treatment and aging was not significant (p = 0.570).

Factor	df	Sum of squares	Mean square.	F	P
Surface Treatment	3	389.70	129.899	23.93	< 0.001
Aging	1	300.73	300.735	55.40	<0.001
Surface Treatment * Aging	3	10.99	3.664	0.67	0.570
Error	72	390.88	5.429		
Total	79	1092.30			

Table 4. Tukey's Analysis

Surface Treatment *Aging	N	Mean	Group *			
ASAC immediate	10	16.30	A			
AFSAC immediate	10	12.94		B		
ASAC aged	10	11.20		B		
AFAC immediate	10	10.58		B		
AAC immediate	10	9.98		B	C	
AFSAC aged	10	9.73		B	C	D
AFAC aged	10	6.77			C	D
AAC aged	10	6.60				D

*Values that do not share a letter differ significantly.

DISCUSSION

Dental material bonding protocols are fundamental to the success of restorative treatment. Many protocols have been developed and are claimed to be the best; however, their efficacy has not yet been fully proven.
This study analyzed the micro-shear bond strength between a hybrid ceramic (VITA ENAMIC) and a resinous cement (AllCem) when different surface treatments were applied.
Our results show that using silane in the bonding protocol is essential to achieve an optimal bond between the hybrid ceramic and resin cement. The group treated with sandblasting, silane and 10-MDP-based universal adhesive exhibited the best bond strength values, while the groups without silane showed significantly lower bonding. These findings support the hypothesis that silane significantly improves bonding in this type of material.
Silane works well for cementing this type of ceramic because it improves the chemical bonds between

silica and methacrylate. The results of the present study agree with those reported by several other authors[7, 13-15].

Silane contains a methacrylate terminal –a small bifunctional molecule, technically called 3-methacryloyloxypropyltrimethoxysilane– which can bind to any ceramic containing silica, as does Vita Enamic (Table 1). It forms covalent bonds at one end (silicon dioxide, Si-O-Si), while the other end binds to the resinous matrix of the cement through the methacrylate group[13].

To achieve this bonding, first, the silane is activated by acetic acid, which converts it to silanol (SiOH). This silanol will bind to an inorganic surface. After the etching reaction, a hydroxyl group (-OH) is produced. The hydroxyl group can lose a proton (H+), facilitating the formation of siloxane bonds (–Si–O–Si–) through condensation.. These covalent bonds are strong when reacted with glass ceramics, as demonstrated in our study. Meanwhile, the organic group (CH3-CH2-) binds to methacrylate-based resins[14, 16].

In the current study, the groups treated with silane only did not have the highest bond strength. The best performing groups were those in which a 10-MDP-based universal adhesive (Single Bond Universal 3M St. Louis, Missouri, USA) was applied after silane. These results confirm the report by the International Academy of Adhesive Dentistry, which suggests that the application of adhesive after silanization could improve resin infiltration into the etched surface[17].

A universal adhesive containing copolymers of methacrylate-modified polyacrylic acid, 10-methacryloyloxyalkyl dihydrogen phosphate (MDP) and silane could bind to both phases (ceramic and resin) of the polymer-infiltrated ceramic network[18]. This occurs because the methacrylate end of the adhesive will bind to the same end which is present in resins and resin cements. The phosphate end of the adhesive will bind to the oxygen at the siloxane end of the previously silanized ceramic. All this determines a strong bond between both materials, which was observed in our study. Even after stress by heat and cold, the strength remained high[19, 20].

One of the most widely used techniques for surface treatment of ceramic materials is sandblasting. According to Fouquet V et al.[17], sandblasting significantly increases the adhesion values for PICN. Abrasion increases the surface roughness of the material, creating irregularities that increase surface energy and improve wettability, thereby improving micromechanical retention[21].

Care is needed for sandblasting. Praisuwanna N [8,14] notes that when a sandblaster is used, pressure, time, distance, and aluminum oxide particle size must be carefully controlled in order to avoid producing microcracks in the ceramic. This was demonstrated by Cevallos[3] and Cascante, et al.[22], who reported that all their in vitro ceramic samples presented fractures when subjected to 50 µm sand. Moreover, abrasion with aluminum oxide particles applied directly on the patient involves the risk of the patient and the practitioner inhaling the dust, which could lead to silicosis, a slowly progressive fibrotic lung disease[19].

Hydrofluoric acid (HF) treatment of a surface selectively dissolves the glassy phase of the ceramic by reacting with the silica, and eliminates the organic part (polymers), producing pores up to 10µ deep, and creating a microstructure that favors bonding strength[20]. The use of low-concentration hydrofluoric acid ensures a gentler surface treatment than sandblasting, and does not produce microcracks, thereby minimizing the risk of subsequent fractures in the ceramic. The results of the groups treated with this technique were similar to those that were sandblasted. Indeed, no significant difference in bond strength was observed between the groups after aging, in agreement with Kömürcüoğlu several other authors[7, 23, 24].

For all these reasons, our findings confirm the Vita Enamic manufacturer's recommendation to treat this ceramic exclusively with hydrofluoric acid. According to Niizuma et al.[25], 4% is an adequate concentration of hydrofluoric acid to avoid damaging the surface of the material and to improve bond strength.

The cementation protocol applied a dual resinous cement whose resinous matrix contains methacrylate monomers such as Bis-GMA, TEGDMA and UDMA, which are also present in the polymeric network of the hybrid ceramic Vita Enamic. The monomers of both materials would therefore be joined by covalent bonds, thereby improving bond strength[24].

Dental restorations are continuously exposed to adverse conditions in the oral cavity, including temperature changes, constant humidity and mechanical forces, which can compromise the durability of the adhesive interface. It is therefore essential to simulate these conditions during in vitro studies by using standardized aging protocols such as thermocycling, which aims to replicate the thermal stress experienced by restorative materials in the mouth during the ingestion of food and beverages at different temperatures[12].

There is a consensus in scientific literature that thermal cycling between 5 °C and 55 °C adequately simulates intraoral thermal fluctuations. The International Organization for Standardization (ISO TR 11405) recommends a protocol of 5000 cycles to represent approximately six months of clinical aging[26]. During this process, water can penetrate the polymer network due to its small molecular size and high molar concentration, which can lead to phenomena such as plasticization, hydrolysis and degradation of the resin cement. These effects directly impact adhesive strength and compromise the long-term stability of the adhesive system[5,22].

In this context, the present study included thermocycling as a critical variable to evaluate the adhesive behavior of PICN hybrid ceramics. The results show a significant decrease in bond strength after thermal aging, highlighting the importance of selecting surface treatment protocols that provide adhesive stability even under adverse conditions. The group treated with sandblasting, silane and universal adhesive with 10-MDP was shown to maintain superior adhesion after thermal cycling, making it the most effective protocol among those evaluated.

This study has some limitations. Being an in vitro study, the results may not fully reflect clinical conditions. In addition, the number of thermocycling cycles used may not be fully representative of clinical aging.

CONCLUSIONS

Within the limitations of an in vitro study, the following conclusions can be drawn:

- Surface treatment combining sandblasting, silane application and use of a 10-MDP-based universal adhesive provides the highest adhesive strength values in PICN hybrid ceramics, both before and after simulated aging by thermocycling.
- After 5000 cycles of thermocycling, adhesion values decreased in all groups, with no statistically significant difference between them. This could be attributed to hydrolytic degradation of the resinous cement induced by water penetration at the adhesive interface.
- These findings highlight the importance of employing surface conditioning protocols that enhance adhesive stability against thermal challenges in the oral environment. However, long-term clinical investigations are required to validate the efficacy of these treatments under real-world conditions.

CONFLICT OF INTERESTS

The authors declare no potential conflicts of interest regarding the research, authorship, and/or publication of this article

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