Fracture Resistance of a CAD/CAM Nanoceramic Repaired with Two Surface Treatments: *In Vitro* Study *

Resistencia a la fractura de una nanocerámica CAD/CAM reparada con dos tratamientos de superficie: estudio *in vitro*

Resistência à fratura de uma nanocerâmica CAD/CAM reparada com dois tratamentos de superfície: estudo in vitro

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ABSTRACT

Background: Fractures of restorations in the mouth are inevitable. When this happens, the clinician should be able to repair them in a simple and reliable way. **Purpose**: To measure adhesion on a composite for Cad/Cam when it has been sandblasted or acid etched**. Methods:** 50 Brava Block sheets were divided into 5 groups of n= 10; and aged at 5000 thermal cycles. 100 resin cylinders of 3 mm diameter were cemented to the sheets. The groups: aluminum oxide (AL10) and aluminum oxide with silane (ALS 10) were sandblasted and the hydrofluoric acid (HF10) and hydrofluoric acid with silane (HFS10) groups were etched. The AHS group (aluminum oxide plus hydrofluoric acid with silane) was sandblasted, and acid etched. The cylinders were cemented with resin cement. All groups were sheared in a universal testing machine. Adhesion failures were analyzed under a 40X microscope. All the statistics were worked with an $\alpha = 0.05$. **Results:** The groups had similar adhesion

strengths. As they aged, their values decreased to 11.15 and 8.15 MPa, on average. The failures were: Adhesives 12 %; Cohesive 68 % and mixed 20 %, higher cohesive and mixed prevalence in sandblasted groups with ruptures of 80-90 %. **Conclusions:** Both treatments produce similar FA between a Cad/Cam composite and a resinous cement. However, the sandblasted groups suffered a higher percentage of material fractures.
Keywords: adhesive strength; Cad/Cam; dental adhesion; dental composite; dental etching; dental materials; dental polymer; dentistry; resin cement; sandblasting; silane; surface treatment; universal adhesive; universal dental adhesive

RESUMEN

Antecedentes: Las fracturas de las restauraciones en boca son inevitables. Cuando ello sucede, el clínico debería poder repararlas de una manera sencilla y confiable. **Objetivo**: Medir la adhesión en un composite para Cad/Cam, cuando ha sido arenado o grabado con ácido. **Métodos**: 50 láminas de Brava Block fueron divididas en 5 grupos de n= 10; y envejecidas a 5000 ciclos térmicos. 100 cilindros de resina de 3 mm de diámetro fueron cementados a las láminas. Los grupos: óxido de aluminio (AL10) y oxido de aluminio con silano (ALS 10) fueron arenados y los grupos ácido fluorhídrico (HF10) Y ácido fluorhídrico con silano (HFS10) fueron grabados. El grupo (óxido de aluminio más acido fluorhídrico con silano) AHS fue arenado y grabado con ácido. Los cilindros se cementaron con cemento de resina. Todos los grupos fueron cizallados en una máquina de ensayo universal. Las fallas en la adhesión fueron analizadas con un microscopio a 40X. Toda la estadística se trabajó con un α= 0,05. **Resultados:** Los grupos tuvieron similares fuerzas de adhesión. Al ser envejecidos, sus valores disminuyeron a 11,15 y 8,15 MPa, en promedio. Los fallos fueron: Adhesivos 12 %; Cohesivos 68 % y mixtos 20 %, mayor prevalencia cohesiva y mixta en los grupos arenados con rupturas del 80-90 %. **Conclusiones**: Ambos tratamientos producen similares FA entre un composite para Cad/Cam y un cemento resinoso. Sin embargo, los grupos arenados sufrieron un mayor porcentaje de fracturas del material.
Palabras clave: adhesión dental; adhesivo universal; arenado; Cad/Cam; cemento de resina; composite dental; fuerza adhesiva; grabado dental; materiales dentales; odontología; polímero dental; silano; tratamiento de superficie

RESUMO

Antecedentes: Fraturas de restaurações em boca são inevitáveis. Quando isso acontece, o clínico deve ser capaz de repará-los de forma simples e confiável. **Objetivo:** Medir a adesão em um compósito para Cad/Cam, quando este foi jateado ou ácido. **Métodos:** 50 placas Brava Block foram divididas em 5 grupos de n= 10; e envelhecido em 5000 ciclos térmicos. 100 cilindros de resina de 3 mm de diâmetro foram cimentados às placas. Os grupos: óxido de alumínio (AL10) e óxido de alumínio com silano (ALS 10) foram jateados e os grupos ácido fluorídrico (HF10) e ácido fluorídrico com silano (HFS10) foram atacados. O grupo AHS (óxido de alumínio mais ácido fluorídrico com silano) foi jateado e atacado com ácido. Os cilindros foram cimentados com cimento resinoso. Todos os grupos foram cisalhados em uma máquina de ensaios universal. As falhas de adesão foram analisadas em microscópio de 40X. Todas as estatísticas foram trabalhadas com α= 0,05. **Resultados:** Os grupos tiveram forças de adesão semelhantes. À medida que envelheciam, seus valores diminuíam para 11,15 e 8,15 MPa, em média. As falhas foram: Adesivos 12 %; Coesivo 68 % e misto 20 %, maior prevalência de coesivo e misto em grupos jateados com rupturas de 80-90 %. **Conclusões:** Ambos os tratamentos produzem FA semelhantes entre um compósito Cad/Cam e um cimento resinoso. No entanto, os grupos jateados sofreram uma maior porcentagem de fraturas de material.
Palavras-chave: adesão dentária; adesivo dentário universal; adesivo universal; ataque dentário; Cad/Cam; cimento resinoso; compósito dentário; força adesiva; jateado; materiais dentários; odontologia; polímero dentário; silano; tratamento da superfície

INTRODUCTION

New polymeric and ceramic materials for indirect restorations are appearing in the dental market. Due to their unique compositions, manufacturers promise better mechanical and optical properties, when compared to the old indirect resins or misnamed ceromers (1). Nanoceramic composites are made of an inorganic phase in the form of ceramic nanoparticles and another phase in the form of a polymer, which is generally TEGDMA, UDMA and BIS/GMA. They all come in blocks for Cad/Cam (2,3).

Due to the ceramic phase in their composition, these materials have excellent gloss, particularly good translucency and allow the dentist to choose from a wide range of colors (4). While its resinous phase allows it to absorb masticatory forces, which ensures a particularly good resistance to fracture (5), with a modulus of elasticity of 160 MPa. Brava Block allows for quite simple repairs or adjustments with composite resin being polymerized directly in the mouth (6). According to the manufacturers themselves, these materials have a flexural strength ranging from 160 to 220 MPa (6-8).

The previous materials to make indirect restorations came in syringes in pastes and the technician had to mold the dental structure, being a longer process and prone to internal defects, they also required light-curing, on the other hand, the Cad/Cam carves pre-cured blocks, everything is mechanical and computerized. Therefore, the mechanical properties are superior, since the grinding of the tooth is no longer performed on a block of plaster, reducing failures when performing indirect restorations (9-11). Due to these characteristics, these composites are ideal for performing treatments such as veneers, single crowns, in incisors and canines, as well as inlays in premolars, with particularly good results in the medium term when they are cemented with the correct adhesive technique (11). Despite this, fractures in the mouth may be unavoidable (12). Unfavorable situations such as: accidents, parafunctional habits, or simply hard foods can cause fractures in these restorations. When this happens, it would be ideal to be able to repair them without having to remove the entire restoration (13, 14).

However, the literature on this is scarce. Sandblasting and acid etching (15) are currently used to repair a ceramic polymer, mentioning that sandblasting is better, however, sandblasting can cause damage to the surface of the material (2). Furthermore, there are no long-term clinical trials on CAD/CAM nanoceramic blocks (10).

If one of these restorations had to be repaired, its main requirement would be to withstand a minimum adhesion force of 10 MPa (16). Considering that, at the time of the repair, the restoration was aged due to the extreme conditions of the mouth, and therefore the residual polymerizable monomers are usually very scarce (17); a method that ensures a quality repair depending on the composition of the material should be investigated.

Many techniques have been described to achieve good bond strengths to ceramic and polymeric materials (4,18). Mechanical procedures such as sandblasting or hydrofluoric acid etching are among the most common (1,4).

By means of these two techniques it is possible to produce micro irregularities to increase the surface, and therefore improve adhesion. However, both have disadvantages. With sandblasting, authors point out that microfractures (19-21) occur that could trigger a new breakage of the restorative material. In contrast, the use of hydrofluoric acid may not be effective on resinous materials. Taking all this background into account, this paper proposes to investigate: Which of these two techniques produces better adhesion forces in a nanoceramic composite for Cad/Cam?

MATERIALS AND METHODS

This was an experimental *in vitro* study. Table 1 shows the trade names, the manufacturer, the batch, and the composition of the materials used in this investigation.

TABLE 1

Materials used in the study

Material	Maker	Lote	Composition
Brava Block	FGM, Joinville. Santa Catarina, Brazil	070121 20310107	80 % silica and 20 % resin matrix. 0.5-40 μm particles
Llis Composite	FGM (Brazil)	240621	BisGMA, BisEMA, TEGDMA, Camphoroquinone
Aluminum oxide sand	Bio Art	74417	50 μm particles
Hydrofluoric acid	Eufar	C14A455	9.6 % hydrofluoric acid
Silane	Ultradent (USA)	CD31A	3-methacryl oxy propyl trimethoxysilane, ethanol, water
Single Bond Universal	3M ESPE (USA)	10604[a]	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, plugger, ethanol, water, initiators, silane
Allcem dual cement	FGM (Brazil)	180121	Base paste: Bis-GMA, Bis-EMA, and TEGDMA, camphoroquinone, barium-aluminum-silicate microparticles, silicon dioxide nanoparticles. Catalyst paste: methacrylic monomers and dibenzoyl peroxide and stabilizers, barium-aluminum glass microparticles

Source: the authors

Five blocks of Brava Block (FGM. Joinville. Sta. Catarina. Brazil) were cut into 50 14x7x1mm slices with a cutting machine using a 0.3mm thick diamond blade, under continuous irrigation. In total, five groups of N=10 were formed. The ceramic surface to be bonded was polished with 600, 1000 and 1200 number sandpaper, each for 1 min.

Once polished, all the sheets were aged for 5000 cycles, which correspond to 6 months of aging according to Alnafaiy, *et al*. (12), with a thermocycler (GreatSolutions, Quito, Ecuador). One cycle consists of a water bath at 55 °C and 5 °C for 30 s each (17, 22, 23).

At the same time, 100 conventional resin cylinders (Llis Composite, FGM, Joinville. Sta. Catarina. Brazil) of 3 mm in diameter and 2 mm in height were made using a metal matrix previously designed by the researchers. These cylinders will be cemented on the aged Brava Block sheets.

The AL10 and ALS10 groups were sandblasted with an office sandblaster (Bio Art Jato. Sao Paulo. Brazil) for 15 s with circular movements, with 50 µm particles of AL2O3, (Aluminum oxide sand, Bio Art, Brazil) at a distance of 10mm measured by a piece of wire connected to the nozzle of the blaster.

Acid etching in the HF10 and HFS10 groups was performed using 9.6 % Hydrofluoric acid (Hydrofluoric Acid, Eufar, Colombia) for 1min. Subsequently, the acid was washed with 20 cc of distilled water by means of a syringe, and dried with oil-free air (hair dryer) for 5 s.

Finally, the AHS group was sandblasted for 7 s and then etched with hydrofluoric acid for 1 min. In order to see if this combination of the two methods was better.

After the mechanical treatment, the HFS10, ALS10 and AHS groups received a chemical surface treatment, which consisted of placing a bonding agent, silane (Ultradent, South Jordan, UTA, USA). This was placed with the tip of a microbrush, rubbing actively for 20 s and then allowed to evaporate for 60 s and we dried the excess with cold, oil-free air for 5 s.

Next, an adhesive with 10 MDP was used. Single bond Universal (St Paul, MN, Minnesota, USA), in all groups, rubbed for 20 s and blown with air, without oil contamination, indirectly for 5 s. It was then light-cured for 20 s with a halogen light lamp (Guilin Woodpecker Medical Instrument, Guilin: China).

Finally, each of the Brava Block sheets thus treated was divided in half (left and right). A resin cylinder was cemented in each half, using an Allcem resin cement (FGM. Joinville, Sta. Catarina, Brazil) that was brought to one end of the cylinder by means of the tip of a 0.5 mm diameter periodontal probe. Once this procedure was completed, a standard force of 1 N was placed on the other end of the cylinder to simulate the pressure exerted by a clinician. Excess cement was removed with the tip of a microbrush, and the test body was polymerized.

The adhesive interface was light-cured with the same light source for 40 s on each side. Finally, the test bodies were stored in water at 37 °C for 7 days, after which time the left half was subjected to the shear test in a universal testing machine (Muver/5053, Software Muver Cx Server Lite) at a speed of 1.0 mm/min, once this procedure was finished, the samples of the right half were aged (5000 cycles) before being tested again (Figure 1).

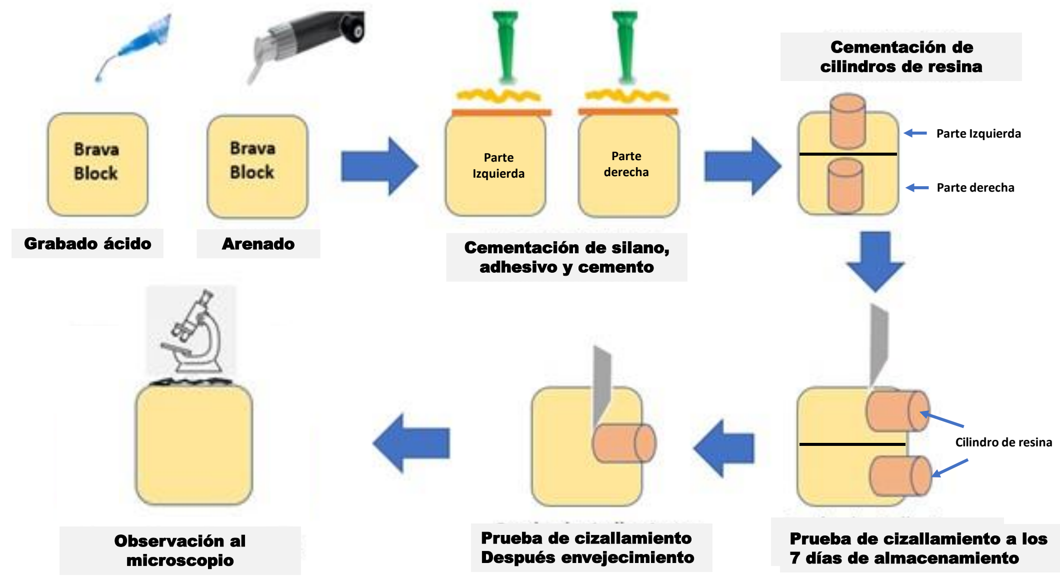

FIGURE 1
Experimental protocol used in the study
Source: the authors

The adhesion force results were collected in MPa. For this, the formula was used: FA=F/a, where,

FA = adhesion force
F= maximum detachment force of the resin cylinders
a= area of the resin cylinder

The area of the cylinder was calculated by means of the formula: $a=\pi r^2$

Finally, each of the detached adhesive interfaces were reviewed under a 40X optical microscope (Oxbird. Canton. China) and classified as Adhesive when no cement is observed at the interface, Cohesive, when cement or cement remains are observed. resin in one of the quadrants of the interface and Mixed when the remains of cement are found in at least two of the quadrants of the adhesive interface.

The statistics were performed with the Minitab 19 program. Minitab Statistical Software. State College. Pennsylvania. USA Data were analyzed with Anderson Darling normality and Levene Homogeneity tests. Once these statistical assumptions were verified, the two-way Anova test (surface treatment and aging) was performed, followed by a Tukey post hoc test, with a value of $p = < 0.05$, which was taken as a reference. to confirm or rule out differences between groups.

RESULTS

5 groups each with N = 10 were analyzed. To obtain the number of samples, three pilot tests were conducted beforehand and their results were subjected to a power test.

Each N had 2 resin cylinders cemented that were evaluated, one of them after 7 days of storage, and the other one was evaluated after the aging process. Taking a total of 100 trials.

In Table 2 it can be seen that the HF10 group that was evaluated after 7 days of storage presented the highest values of adhesion force (13.67 MPa), in this group 9.6% hydrofluoric acid was used as treatment. surface, but no silane was applied. On the other hand, the lowest value in the immediate group occurred in the HFS10 group, with an average value of 11.15 MPa. It must be considered that, in the immediate group, the FA values do not show a difference between them.

TABLE 2

Mean and standard deviation of FA by shearing of groups AL10, ALS10, HF10, HFS10 and AHS evaluated after 7 days of storage and subsequent aging *

Groups	N	Average FA (stored 7 days)	Std. Dev. (±)	Average FA aged	Std. Dev. (±)
AL10	10	12,59 A	5,97	11,15 A	4,35
ALS10	10	11,57 A	2,49	10,60 A	2,99
HF10	10	13,67 A	3,25	8,15 B	2,48
HFS10	10	11,15 A	3,74	7,30 B	3
AHS	10	12,23 A	3,98	10,20 A	3,38

*Groups that do not share a letter are significantly different.

Source: The authors.

Regarding the aged group, it can be seen that the FA values reached by the tested groups immediately decreased after being subjected to thermocycling of 5000 cycles, corresponding to 6 months of clinical use in the mouth, according to such Alnafaiy *et al* (12) and Simancas Y et al (24). The group with the least FA was HFS10 with an average value of 7.30 MPa. While the AL10 group was the one that reached the highest FA value with 11.15 MPa (table 2).

Regarding inferential statistics (Table 3), the two-way Anova test was applied and showed that the aging factor (p= 0.000); was responsible for the difference between these groups. While the surface treatment factor (p= 0.237) and the interaction between surface treatment plus aging (p= 0.255) did not show differences.

TABLE 3

Two-way Anova analysis for the Factors Surface treatment (Tto de sup), Aging (Envej) and the interaction Surface treatment with Aging (Tto de sup*Agej)

Factor	**GL**	**SC Ajust.**	**MC Ajust.**	**Valor F**	**Valor p**
Tto de sup	4	78,48	19,62	1,41	0,237
Envej	1	184,13	184,13	13,23	0,000
Tto de sup*Envej	4	75,63	18,91	1,36	0,255
Error	90	1253,03	13,92		
Total	99	1591,27			

TABLE 4
Shows the Tukey analysis with a confidence of 95 %, it can be confirmed that the aging factor was the cause of the AF averages of the groups tested after 7 days of storage 12.24 MPa and then of aged 9.54 MPa, were different. FA values decrease significantly after thermocycling, regardless of the surface treatment used

Groups	FA 7 días de alamcenamiento		FA después del envejecimiento	
	Average	**Agrupación**	**Media**	**Agrupación**
AL10	12,59	**A B**	11,15	**A B**
ALS10	11,571	**A B**	10,60	**A B**
HF10	13,67	**A**	8,151	**B**
HFS10	11,15	**A B**	7,308	**B**
AHS	12,23	**A B**	10,20	**A B**

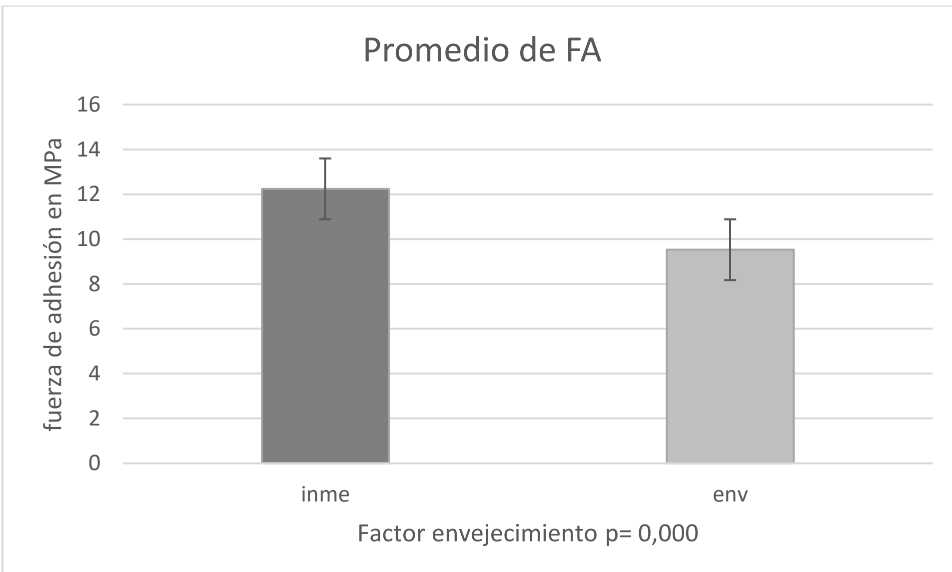

FIGURE 2
Adhesion force between the immediate groups (7 days of storage) and after aging when they were analyzed by the Tukey test

Among the predominant types of failure in the study groups these were: cohesive and mixed, with the exception of the aged HF10 and HFS10 groups where a higher percentage of adhesive failure of 50% and 60% respectively is shown.

In Table 5 it can be observed that when evaluating the adhesive failures, the groups that were sandblasted and evaluated after 7 days of storage and after the aging process presented a higher prevalence of cohesive and mixed failures.

TABLE 5

Types of adhesion failure found after analysis under the microscope

INMEDIATO (7 días después almacenado)	ADHESIVO %	COHESIVO %	MIXTO %
AL10	10	70	20
ALS10	0	80	20
HF10	10	90	0
HFS10	30	50	20
AHS	10	50	40
ENVEJECIDO	ADHESIVO %	COHESIVO %	MIXTO %
AL10	10	60	30
ALS10	20	60	20
HF10	50	40	10
HFS10	60	10	30
AHS	40	60	0

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Six representative microphotographs of the type of failures found in the material detachment test.

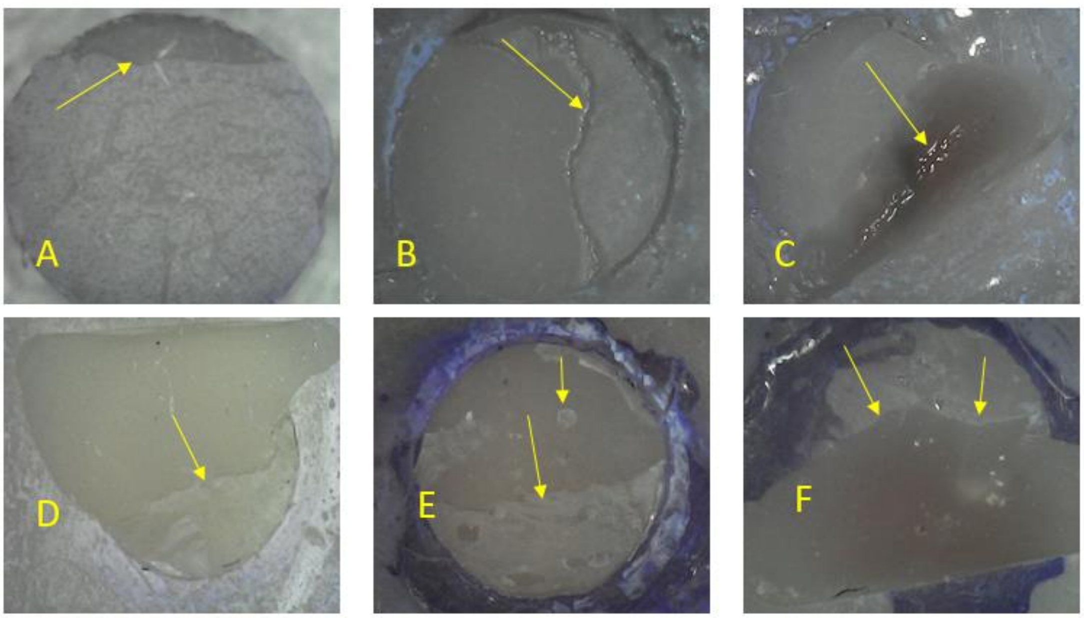

FIGURE 1

Most representative micrographs of the type of adhesive failure found in the test

To an adhesive failure on an interface sandblasted and tested immediately. The arrow shows the limit where resinous cement was. B shows a mixed fault. The arrow marks the limit between cement and ceramic. C shows cohesive failure. It is observed that the ceramic broke in its entire thickness and reveals the bottom of the test body. Images D to F show aged test bodies. The arrows indicate the areas where moisture penetrated and produced hydrolysis and therefore fracture of the cement. F shows a cohesive type of fracture after the material has aged, in the AHS group. You can see the large fracture that occurred in the ceramic with this surface treatment.

DISCUSSION

The present investigation measured the shearing FA of an aged nanoceramic composite, after being subjected to two surface treatments: sandblasting and acid etching, with the aim of knowing what would happen if, when trying to repair a fracture that could occur in this material when it has already been in clinical use.

Within the limitations of the study, in Latin America it is difficult to find the same materials used in other continents. The lack of institutional and state financing, in addition, we do not have specialized laboratories in the universities, this means that researchers have to travel to other places, and this could influence the results, it is impossible to replicate in a laboratory all the natural conditions of the environment due to this, our results are not entirely conclusive. Therefore, further research is needed in other circumstances, for example: presence of bacteria, chewing force, longer aging time, etc.

The results showed that both sandblasting, and acid etching reached similar adhesion values. Regarding the interaction, surface treatment and aging, no significant statistical difference was found ($p= 0.010$), but the aging factor did show differences between the groups ($p= 0.000$). Therefore, the null hypothesis was partially accepted.

This indicates that it is possible to adhere to this material by means of these two techniques, with similar values (15). However, when analyzing the adhesive interface under the microscope, it was possible to observe that, in the groups subjected to sandblasting, mostly cohesive and mixed type failures were predominant. Our investigation found that this type of failure occurred with the sandblasting technique. of failures in 80-90%, the same ones that were found by Lise (3).

Sandblasting with AL_2O_3 blasting is a widely used technique to improve adhesion between different substrates (5, 25-27). Through this technique it is possible to obtain a rough surface that increases the surface area for adhesion between resin cement and ceramic (28). In addition, sandblasting removes layers of contamination and forms large irregular grooves in the ceramic surface, allowing resin to infiltrate the surface of the material (12, 17). This improves the adhesion forces between a resinous cement and a restorative material (17).

The findings of the present investigation only confirm what was reported by Niizuma, *et al.* (2) and Cinar, *et al.* (20), who state that sandblasting can cause damage to the polymer surface by generating microcracks that cause cracks that can continue to grow with the stress of temperature changes and end up fracturing the material.

This internal damage occurs due to the force and the short distance at which the sand particles are thrown onto the ceramic. It is quite possible that the direction and size of the sand particle may be decisive when creating microcracks (20, 29).

It is known that sand particles can come in sizes of 20, 30, 50, 100, 110 and up to 200 µm. (25). It is obvious to assume that the smaller the size, the impact and damage of the ceramic will be less. While the larger the size, the impact force will be more intense. In our work we use 50 µm sand and found catastrophic damage to the body of the material. We believe that with larger particles the internal damage will be worse (29).

All of this is important because most clinicians delegate this procedure to the dental technician, without knowing the particle size with which the sandblasting was performed. This information can be crucial when cementing an indirect restoration, as it can be part of its success or failure.

The finding of a high percentage of cohesive and mixed fractures of the material when it was sandblasted can be a problem to consider. It does not seem to be a good idea to sandblast a nanoceramic composite to try to repair it in case it has fractured, since it could break again (29).

Another issue that must be considered is that sandblasting with aluminum oxide particles directly into the mouth of patients should be avoided at all costs, as the person could inhale the Al2O3 particles and serious lung problems could be triggered (30). Therefore, faced with so many disadvantageous findings, we would rule out this method to suggest it as a technique to want to repair one of these composites directly in the mouth (23).

Regarding acid etching, authors state that etching with hydrofluoric acid (HF) selectively dissolves the surface filler of the block, to form microporosities, notches and grooves, making it rough, providing micromechanical retention and increasing its surface energy (31-33). The composition of Brava Block is 80% inorganic matrix in the form of a glass-ceramic; therefore, this polymer can be treated with this technique and obtain adhesion values equal to those of sandblasting. The findings of this work only confirm what was reported by DP Lise et al in 2017 (3).

Hydrofluoric acid provides micromechanical retention, because it dissolves the glassy phase of the nanoceramic, making it rough, producing micro irregularities and increasing surface energy to improve AF (2, 21). Brava Block has 80% silica in its composition, the action of the HF dissolved this silica matrix (6), and for this reason the HF had an effect similar to sandblasting; In addition, in the groups subjected to acid etching, no fractures and breaks of the material were observed. This leads us to assume that microfractures are not produced by means of this technique, as stated in the study by Niizuma Y et al (2) where it is recommended to use HF as a surface treatment. Mixed failures were 0% in the immediate HF10 group and barely 10% in the aged group. Therefore, in case of fractures of this material, it will be better to attack it with acid in case you want to repair it. Obviously, all the special care will have to be taken, due to the danger of this acid, when working on the soft tissues of a patient.

An interesting observation was that all groups had good FA values when evaluated immediately. This is crucial because this material would not need the prior placement of a silane (18, 34) to have good adhesion, and this could save the dentist time and material.

Regarding the adhesive used in this study, it is known that 10 MDP methacryloyl oxidecyl dihydrogen phosphate has particularly good results joining silica-based vitreous substrates with resinous materials with methacrylate terminals (4, 7). The 10 MDP has two functional ends. One of them is a methacrylate end capable of reacting with resinous cement and the other end is a phosphate end that is capable of chemically bonding to the silica of ceramic. The long chain of 10 carbons makes it difficult for moisture to penetrate from one end of the adhesive interface to the other. This explains why the adhesion values did not decrease to critical values when they were aged (35, 36).

The groups treated with ALS10, HFS10 and AHS silane had, on average, similar adhesion values to those groups not treated with this molecule (Table 2). These results indicate that it would be unnecessary to place silane in this new restorative material, since a layer of adhesive with MDP is enough to obtain good results.

The AHS group was a hybrid of surface treatments. Sandblasting was used, followed by hydrofluoric acid. Hypothesizing that AF would increase with the combination of these treatments. However, AF was not higher than the rest of the groups. What could be appreciated was that cohesive failures were high (60 %). Therefore, we can mention that using these two surface treatments does not increase the adhesion force and was even the cause of a higher percentage of cohesive and mixed fractures of the material (Table 5).

Regarding the method used, thermocycling has been found to be an ideal technique to simulate the aging conditions of restorative materials in the mouth (21, 37). Indeed, in this investigation, it was possible to verify how the adhesion values decreased when the adhesive and the cement were stressed by means of heat and cold. In this way it is possible to understand how restorations perform intraorally after some time of use. While it is true that it is impossible to replicate all the situations that can be found in vivo; At least with this simulation technique, researchers can know that the adhesive interface degrades over time, even with the best techniques recently developed (11).

CONCLUSIONS

With the limitations of the study, we can conclude that: Both sandblasting with Al2O3 particles and etching with 9.6 % hydrofluoric acid were able to generate similar bond forces between a resin cement and a nanoceramic composite.

Sandblasting produced a greater number of cohesive and mixed type fractures.

Etching with 9.6 % hydrofluoric acid had a lower incidence of cohesive and mixed type fractures.

FA values decreased in all groups after being aged.

Our results are still not conclusive. Further research is needed with larger samples with other conditions related to these results with other studies.

RECOMMENDATIONS

Further investigations with different chemical formulations of bonding agents are recommended.

It is recommended to replicate the present study with materials similar to those of the authors.

It is recommended to conduct studies in patients in the medium and long term.

Conflict of interest: The authors declare that they have no conflict of interest.

Contribution of the authors: MCC design and conception of the investigation of the investigation, statistics, wrote and corrected the final version of the article. KRF, conception of the research, conducted the experimental part, wrote and corrected the article. VAT, wrote, reviewed and corrected the results. ABS, assisted in the experimental part, wrote the methodology and corrected the article. JTG assisted in the experimental part, wrote the methodology and corrected the article.

References

1. Cevallos GQ, Flores LC, Guamán KG, Gonzaga MC, Bedoya PC, López VQ, **CascanteCalderón MG**. Influencia del arenado sobre la resistencia adhesiva de un polímero HPP para CAD CAM. Odontol Activ Rev Cient. 2022; 7(1): 17-22. https://doi.org/10.31984/oactiva.v7i1.624
2. Niizuma Y, Kobayashi M, Toyama T, Manabe A. Effect of etching with low concentration hydrofluoric acid on the bond strength of CAD/CAM resin block. Dent Mater J. 2020 Dec 3; 39(6): 1000-1008. https://doi.org/10.4012/dmj.2018-398
3. Lise DP, Van Ende A, De Munck J, Vieira L, Baratieri LN, Van Meerbeek B. Microtensile Bond Strength of Composite Cement to Novel CAD/CAM Materials as a Function of Surface Treatment and Aging. Oper Dent. 2017 Jan/Feb; 42(1): 73-81. https://doi.org/10.2341/15-263-L
4. Guimarães HAB, Cardoso PC, Decurcio RA, Monteiro LJE, de Almeida LN, Martins WF, Magalhães APR. Simplified Surface Treatments for Ceramic Cementation: Use of Universal Adhesive and Self-Etching Ceramic Primer. Int J Biomater. 2018 Dec 31; 2018: 2598073. https://doi.org/10.1155/2018/259807.
5. Skorulska A, Piszko P, Rybak Z, Szymonowicz M, Dobrzyński M. Review on Polymer, Ceramic and Composite Materials for CAD/CAM Indirect Restorations in Dentistry-Application, Mechanical Characteristics and Comparison. Materials (Basel). 2021 Mar 24; 14(7): 1592. https://doi.org/10.3390/ma14071592
6. FMG Group. Brava Block. 2017. https://fgmdentalgroup.com/latam/productos-protesicos/brava-block/
7. Cedillo Valencia JDJ, Solano González AC. Monobond Etch & Prime, primer acondicionador monocomponente, que permite el grabado y la silanización de las superficies cerámicas en un solo paso. Revista ADM. 2018; 75(6): 358-369.
8. Sonmez N, Gultekin P, Turp V, Akgungor G, Sen D, Mijiritsky E. Evaluation of five CAD/CAM materials by microstructural characterization and mechanical tests: a comparative in vitro study. BMC Oral Health. 2018 Jan 8; 18(1): 5. https://doi.org/10.1186/s12903-017-0458-2
9. Marchesi G PA, Nicolin V, Turco G, Lenarda D. Materiales CAD / CAM en el consultorio : tendencias actuales de usos clínicos Abstracto. 2021; 10(11): 1-14.
10. Grzebieluch W, Mikulewicz M, Kaczmarek U. Resin Composite Materials for Chairside CAD/CAM Restorations: A Comparison of Selected Mechanical Properties. J Healthc Eng. 2021 Apr 28; 2021: 8828954. http://doi.org/10.1155/2021/8828954
11. Berkman M, Tuncer S, Tekçe N, Karabay F, Demirci M. Microtensile bond strength between self-adhesive resin cements and resin based ceramic CAD/CAM block. Odovtos-International Journal of Dental Scienc 2021; 23(1): 116-125. http://dx.doi.org/10.15517/ijds.2021.43670.

12. Alnafaiy S, Labban N, Maawadh A, Alshehri H, Albaijan R. Repair bond strength of composite resin to aged resin and glass-matrix CAD/CAM ceramic materials using two different repair systems. Coatings. 2021; 11(11): 1331.
13. Subasi MGAG. Reparar las resistencias de unión de nanocerámicas de resina envejecidas y no envejecidas. 2017; 364-370.
14. Demirel G, Baltacıoğlu İH. Influence of different universal adhesives on the repair performance of hybrid CAD-CAM materials. Restor Dent Endod. 2019 May 20; 44(3): e23. htt://doi.org/10.5395/rde.2019.44.e23
15. Mohammed Said MMMZ T. El efecto del envejecimiento y los tratamientos superficiales sobre la resistencia a la unión por micro-cizallamiento del material nanocerámico. 2021:1-19.
16. Hernández MJ. Aspectos prácticos de la adhesión a dentina. Av odontoestomatol 2004; 20(1): 19-32.
17. Günal-Abduljalil B, Önöral Ö, Ongun S. Micro-shear bond strengths of resin-matrix ceramics subjected to different surface conditioning strategies with or without coupling agent application. J Adv Prosthodont. 2021 Jun; 13(3): 180-190. http://doi.org/10.4047/jap.2021.13.3.180
18. Politano G, Van Meerbeek B, Peumans M. Nonretentive Bonded Ceramic Partial Crowns: Concept and Simplified Protocol for Long-lasting Dental Restorations. J Adhes Dent. 2018; 20(6): 495-510. http://doi.org/10.3290/j.jad.a41630
19. Yoshihara K, Nagaoka N, Maruo Y, Nishigawa G, Irie M, Yoshida Y, Van Meerbeek B. Sandblasting may damage the surface of composite CAD-CAM blocks. Dent Mater. 2017 Mar; 33(3): e124-e135. http://doi.org/10.1016/j.dental.2016.12.003
20. Cinar S, Altan B, Akgungor G. Comparison of bond strength of monolithic CAD-CAM materials to resin cement using different surface treatment methods. J. Adv. Oral Res 2019; 10(2): 120-127. https://doi.org/10.1177/23202068198620
21. Cekic-Nagas I, Ergun G, Egilmez F, Vallittu PK, Lassila LV. Micro-shear bond strength of different resin cements to ceramic/glass-polymer CAD-CAM block materials. J Prosthodont Res. 2016 Oct; 60(4): 265-273. http://doi.org/10.1016/j.jpor.2016.02.003
22. Said AM, Zohdy MM, Morsy TS. The effect of aging and surface treatments on micro-shear bond strength of resin nano-ceramic material using two resin cements. J Dent Oral Sci. 2020; 2(1): 1-14.
23. May MM, Rodrigues C da S, da Rosa JB, Herrmann JP, May LG. Enfoques de tratamiento superficial y adhesión en la red cerámica infiltrada con polímeros: influencia en la fuerza de unión al cemento de resina. Brasil. J. ciencia oral. 8 de febrero de 2021; 20(00): e211670.
24. Simancas Pereira Y, Rosales Leal J, Vallejo Bolaños E. Efecto del termociclado y el acondicionamiento en el sellado de una resina microhibrida. Acta odontol venez. 2012.
25. Papadopoulos K, Pahinis K, Saltidou K, Dionysopoulos D, Tsitrou E. Evaluation of the Surface Characteristics of Dental CAD/CAM Materials after Different Surface Treatments. Materials (Basel). 2020 Feb 22; 13(4): 981. http://doi.org/10.3390/ma13040981
26. Awad MM AL, Almahdy A. Salud bucal de BMC sobre la fuerza de unión microtensil a la cerámica híbrida. 2021; 1:–28.
27. Sarahneh O, Günal-Abduljalil B. The effect of silane and universal adhesives on the micro-shear bond strength of current resin-matrix ceramics. J Adv Prosthodont. 2021 Oct; 13(5): 292-303. http://doi.org/10.4047/jap.2021.13.5.292.
28. Souza J, Fuentes MV, Baena E, Ceballos L. One-year clinical performance of lithium disilicate versus resin composite CAD/CAM onlays. Odontology. 2021 Jan; 109(1): 259-270. http://doi.org/10.1007/s10266-020-00539-3
29. Tanış MÇ, Akçaboy C. Effects of Different Surface Treatment Methods and MDP Monomer on Resin Cementation of Zirconia Ceramics an In Vitro Study. J Lasers Med Sci. 2015 Fall; 6(4): 174-81. http://doi.org/10.15171/jlms.2015.15.
30. Nordberg G. Metales: Propiedades químicas y toxicidad productos químicos. Enciclopedia de salud y seguridad en el trabajo. 2017; 2: 63.
31. Azevedo VLB, de Castro EF, Bonvent JJ, de Andrade OS, Nascimento FD, Giannini M, Cavalli V. Surface treatments on CAD/CAM glass-ceramics: Influence on roughness, topography, and bond strength. J Esthet Restor Dent. 2021 Jul;33(5):739-749. http://doi.org/10.1111/jerd.12734.
32. Leyva Del Rio D, Sandoval-Sanchez E, Campos-Villegas NE, Azpiazu-Flores FX, Zavala-Alonso NV. Influence of Heated Hydrofluoric Acid Surface Treatment on Surface Roughness and Bond Strength to Feldspathic Ceramics and Lithium-Disilicate Glass-Ceramics. J Adhes Dent. 2021 Dec 3; 23(6): 549-555. http://doi.org/10.3290/j.jad.b2288275
33. Veríssimo AH, Moura DMD, Tribst JPM, Araújo AMM, Leite FPP, Souza ROAE. Effect of hydrofluoric acid concentration and etching time on resin-bond strength to different glass ceramics. Braz Oral Res. 2019; 33: e041. http://doi.org/10.1590/1807-3107bor-2019.vol33.0041
34. Murillo-Gómez F, Goes MFD. Effect of different silane treatments on long-term bonding between non-etched glass-ceramic and resin cement. Odovtos Int. J. Dental Sc. 2017; 19(2): 33-46. : http://dx.doi.org/10.15517/ijds.v0i0.27950
35. Ríos Szalay E, Garcilazo Gómez A, Guerrero Ibarra J, Meade Romero I, Miguelena Muro K. Estudio comparativo de la resistencia al desplazamiento de cuatro cementos en zirconia. Rev odontol MEX. 2017; 21(4): 235-240.
36. Corts Rover JP, Abella R. Protocolos de cementado de restauraciones cerámicas. Act odontol. 2013: 37-44.

37. Sismanoglu S, Yildirim-Bilmez Z, Erten-Taysi A, Ercal P. Influence of different surface treatments and universal adhesives on the repair of CAD-CAM composite resins: An in vitro study. J Prosthet Dent. 2020 Aug; 124(2): 238.e1-238.e9. http://doi.org/10.1016/j.prosdent.2020.02.029

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