

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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DOI : <https://doi.org/10.11144/Javeriana.uo42.frcc>
Submission Date: 16 January 2023
Acceptance Date: 9 May 2023
Publication Date: 19 December 2023

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ABSTRACT

Background: Fractures of oral restorations are inevitable. When they occur, the clinician should be able to repair them in a simple and reliable manner. **Purpose:** To measure the bond strength (BS) of a CAD/CAM composite when it has been sandblasted or acid-etched. **Methods:** 50 Brava Block veneers 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 veneers. The groups, aluminum oxide (AL10) and aluminum oxide with silane (ALS 10), were sandblasted. The groups, hydrofluoric acid (HF10) and hydrofluoric acid with silane (HFS10) were etched. The group, aluminum oxide plus hydrofluoric acid with silane (AHS) was sandblasted and acid-etched. The cylinders were cemented with resin cement. All groups were sheared on a universal testing machine. Bond failures were analyzed with a light microscope at 40X (p = 0.05). **Results:** The groups had similar bond strengths. When aged, their

values decreased to 11.15 and 8.15 MPa on average. The failures were adhesive (12%), cohesive (68%), and mixed (20%). A higher frequency of cohesive and mixed failures was observed in the sandblasted groups with 80-90 % fractures. **Conclusions:** Both treatments produced similar FA between a Cad/Cam composite and a resin 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 fuerza de adhesión (FA) 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 óxido de aluminio con silano (ALS 10), fueron arenados. Los grupos ácido fluorhídrico (HF10) y ácido fluorhídrico con silano (HFS10) fueron grabados. El grupo óxido de aluminio más ácido 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 de luz a 40X ($p = 0,05$). **Resultados:** Los grupos tuvieron similares fuerzas de adhesión. Al ser envejecidos, sus valores disminuyeron a 11,15 y 8,15 MPa en promedio. Las fallas fueron adhesivas (12%), cohesivas (68%) y mixtas (20%). Mayor frecuencia de fallas cohesivas y mixtas se observó en los grupos arenados con rupturas del 80-90 %. **Conclusiones:** Ambos tratamientos produjeron 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 na boca são inevitáveis. Quando isso acontece, o médico deve ser capaz de repará-los de forma simples e confiável. **Objetivo:** Medir a resistência de adesão (RA) em um compósito Cad/Cam, quando este foi jateado ou gravado com ácido. **Métodos:** 50 folhas de Brava Block foram divididas em 5 grupos de n=10 e envelhecidas a 5000 ciclos térmicos. 100 cilindros de resina de 3 mm de diâmetro foram cimentados nas placas. Os grupos óxido de alumínio (AL10) e óxido de alumínio com silano (ALS 10) foram jateados. Os grupos ácido fluorídrico (HF10) e ácido fluorídrico com silano (HFS10) foram condicionados. O grupo de óxido de alumínio mais ácido fluorídrico de silano (HSA) foi jateado e atacado com ácido. Os cilindros foram cimentados com cimento resinoso. Todos os grupos foram cizalhados em uma máquina de testes universal. As falhas de adesão foram analisadas em microscópio óptico de 40X ($p = 0,05$). **Resultados:** Os grupos apresentaram forças de adesão semelhantes. À medida que envelheciam, seus valores diminuíram para 11,15 e 8,15 MPa em média. As falhas foram adesivas (12%), coesivas (68%) e mistas (20%). Foi observada maior frequência de falhas coesivas e mistas nos grupos jateados com rupturas de 80-90%. **Conclusões:** Ambos os tratamentos produziram RA semelhantes entre um compósito Cad/Cam e um cimento resinoso. Porém, os grupos jateados sofreram maior percentual de fraturas do 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 on the dental market. Due to their unique compositions, manufacturers promise improved mechanical and optical properties when compared to the old indirect resins, wrongly called ceromers (1). Nanoceramic composites have an inorganic phase in the form of ceramic nanoparticles and a polymer phase, which is usually TEGDMA, UDMA, or BIS/GMA. These materials consist of glass-ceramic blocks for Cad/Cam (Brava Blocks®, FGM Dental Group, Joinville, Brazil) (2,3). Due to the ceramic phase of their composition, these materials have excellent gloss, very good translucency, and allow the dentist to choose from a wide range of colors (4). Moreover, their resinous phase allows these materials to absorb masticatory forces,

which ensures good fracture resistance (5), whose modulus of elasticity is 160 MPa. Brava blocks allow for easy repairs or adjustments with a composite resin that is polymerized directly in the mouth (6). These materials have a flexural strength between 160 and 220 MPa (6-8).

Composites used in indirect restorations used to be presented in syringes with the paste and the dental technician had to model the tooth structure, which was a long process and susceptible to internal defects. In addition, the materials were photopolymerized. In contrast, with Cad/Cam, the pre-polymerized blocks are prepared in a completely mechanical and digital process; for this reason, its mechanical properties are better, in addition to the fact that the preparation of a tooth is no longer done on a stone block, thus reducing the possibility of failure when performing indirect restorations (9-11).

Due to these characteristics, the new composites are excellent for treatments such as veneers and single crowns on incisors and canines, as well as inlays on premolars. Their results are optimal in the medium term when cemented using the correct adhesive technique (11). Nevertheless, oral fractures may still be unavoidable (12). Unfavorable situations such as accidents, parafunctional habits, or hard foods may cause fractures in restorations. When this happens, it would be ideal for the dentist to be able to repair fractures without having to remove an entire restoration (13,14). The literature on this issue is limited. Currently, sandblasting and acid etching are used to repair a ceramic polymer (15). Niizuma, *et al.*, (2) mention that sandblasting is better; however, sandblasting can cause damage to the surface of the material (2). In addition, there are no long-term clinical trials on Cad/Cam nanoceramic blocks (10).

If a restoration were to be repaired, the main requirement would be to withstand a minimum bond strength of 10 MPa (16). At the time of repair, a restoration may be aged by the physiological conditions of the mouth. In such cases, polymerizable residual monomers are often scarce (17). A method that ensures a quality repair according to the composition of the material should be investigated. Different techniques have been described to achieve acceptable bond strengths to ceramic and polymeric materials (4,18). Thus, mechanical procedures such as sandblasting and etching with hydrofluoric acid are the most commonly used (1,4). With both techniques, micro-irregularities can be produced to increase the surface area and improve adhesion. However, they have disadvantages. Several authors (19-21) point out that, on the one hand, sandblasting produces microfractures that could trigger new fractures of the restorative material. On the other hand, hydrofluoric acid may not be effective on resinous materials. Considering the problem stated above, the present study set out to answer the question, Which of the two techniques, sandblasting and acid etching, produces better adhesion forces in a nanoceramic composite for Cad/Cam?

MATERIALS AND METHODS

An experimental *in vitro* study was conducted. Five Brava Blocks were sectioned into 50 sheets of 14 x 7 x 1 mm using a 0.3 mm thick diamond cutting machine with continuous irrigation. Table 1 lists the brands, manufacturers, batch, and composition of the materials used in the study. Five groups of n = 10 sheets were created. Each ceramic surface to be bonded was polished with #600, #1000 and #1200 sandpaper for 1 min. Once polished, all the sheets were aged by subjecting them to 5000 cycles, equivalent to 6 months of aging, as described by Alnafaiy, *et al.*, (12) with a thermal cycler (GreatSolutions®, Quito, Ecuador). Each cycle consisted of a water bath at 55 °C and 5 °C for 30 s (17,22,23).

Likewise, 100 conventional resin cylinders (Llis Composite®, FGM, Joinville, Santa Catarina, Brazil) with a diameter of 3 mm and a height of 2 mm were produced using a metal matrix previously designed by the researchers. These cylinders were cemented onto the aged Brava Block sheets.

TABLE 1
Materials Used in the Study

Material	Manufacturer	Batch	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 (Colombia)	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

The AL10 and ALS10 groups were treated with a dental office sandblaster (Bio Art Jato®, Sao Paulo, Brazil) for 15 s with circular movements. 50 µm particles of AL22O3 (aluminum oxide sand, Bio Art®, Brazil) were used at a distance of 10 mm and measurements were made with a piece of wire connected to the nozzle of the sandblaster. Acid etching in the HF10 and HFS10 groups was performed with 9.6% hydrofluoric acid (Eufar®, Colombia) for 1 min. Subsequently, the acid was washed with 20 cm² 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 observe whether the combination of both methods gave better results.

After mechanical treatment, the HFS10, ALS10, and AHS groups received a chemical surface treatment consisting of the application of a silane bonding agent (Ultradent®, South Jordan, UT, USA). The silane was applied with the tip of a microbrush, actively rubbing for 20 s, then allowing it to evaporate for 60 s, and drying the excess with cold, oil-free air for 5 s. Then, a 10% MDP Single Bond Universal® adhesive (St Paul, MN, USA) was used in all groups, rubbing for 20 s and blowing with oil-free air 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 treated Brava Block sheets was divided into left and right halves. A resin cylinder was cemented onto each half using Allcem resin cement (FGM®, Joinville, Sta. Catarina, Brazil), which was brought to one end of the cylinder by means of the tip of a 0.5 mm diameter periodontal probe. After this procedure, a standard force of 1 N was applied to 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 specimen was polymerized.

The adhesive interface was photopolymerized with the same light source for 40 s per side. Finally, the test specimens were stored in water at 37 °C for 7 days. After this time, the left half was subjected to shear testing on a universal testing machine (Muver/5053®, Muver Cx Server Lite software) at a speed of 1.0 mm/min. After this procedure, the right half specimens were subjected to an aging process (5000 cycles) before being tested again (Figure 1).

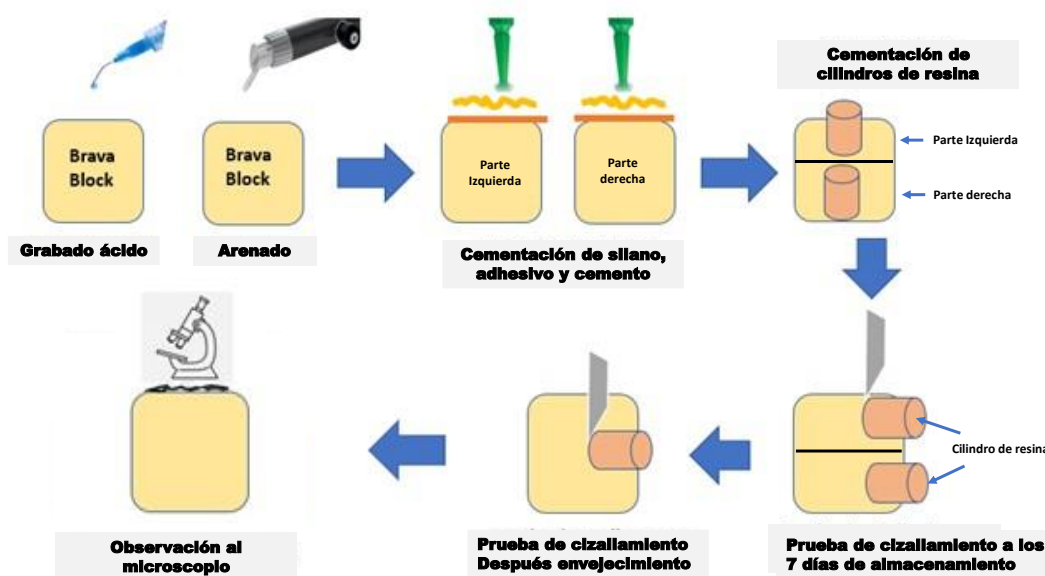


FIGURE 1
Experimental Protocol Used in the Study
Source: the authors

The adhesion force results were recorded in MPa. The formula $FA=F/a$ was used, where:

FA = adhesion force

F= maximum peeling force of the resin cylinders

a= area of the resin cylinder.

The area of the cylinder was calculated using the formula: $a=\pi r^2$ [$a=\pi r^2$].

Finally, each of the detached adhesive interfaces was observed under a light microscope at 40X magnification (Oxbird®, Canton, China). Interfaces were classified as adhesive when no cement was observed at the interface; cohesive when cement or resin residues were observed in one of the quadrants of the interface; and mixed when cement residues were present in at least two of the quadrants of the adhesive interface.

All statistical analysis was performed using Minitab 19® (Minitab Statistical Software, State College, PA, USA). Data were analyzed using Anderson Darling's normality test and Levene's homogeneity test. Once these statistical assumptions were met, a two-way ANOVA test (surface treatment and aging) was performed, followed by a Tukey post hoc test, with a p value = < 0.05, being taken as a reference to confirm or rule out differences between groups.

RESULTS

Five groups were analyzed, each with n = 10. To calculate the number of samples, three pilot tests were performed beforehand and their results were subjected to a power test. Each group had 2 resin cylinders cemented on the samples, which were tested, one of them after 7 days of storage and the other after the aging process. There were a total of 100 tests.

Table 2 shows that the HF10 group, which was tested after 7 days of storage, presented the highest adhesion strength values (13.67 MPa). In this group, 9.6 % hydrofluoric acid was used as a surface treatment, but no silane was applied. On the other hand, the lowest value in the immediate group was

presented in the HFS10 group, with an average value of 11.15 MPa. It should be noted that in the immediate group, the FA values did not show differences 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	3,00
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.

As regards the aged group, it can be observed that the FA values achieved by the groups tested immediately decreased after being subjected to a thermocycling of 5000 cycles corresponding to 6 months of clinical use in the mouth, according to Alnafaiy, *et al.*, (12) and Simancas, *et al.*, (24). The group with the lowest FA was HFS10 with an average value of 7.30 MPa, while the AL10 group had the highest FA value with 11.15 MPa (Table 2).

Concerning the inferential statistical analysis (Table 3), the two-way ANOVA test showed that the aging factor was responsible for the differences between these groups ($p = 0.000$). On the other hand, the surface treatment factor ($p = 0.237$) and the interaction between surface treatment plus aging ($p = 0.255$) did not show statistically significant 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 Adjust.	MC Adjust.	F	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			

Source: the authors.

Table 4 shows the results of the Tukey analysis with a confidence level of 95%. It is confirmed that the aging factor was the cause of the different FA averages of the tested groups after 7 days of storage (12.24 MPa) and after aging (9.54 MPa). The FA values decreased significantly after thermocycling, regardless of the surface treatment used (Figure 2).

TABLE 4

Tukey Analysis of Adhesion Strength at 7 Days of Storage and After Aging

Group	FA 7-day storage		FA after aging	
	Average	Grouping	Average	Grouping
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

Source: the authors.

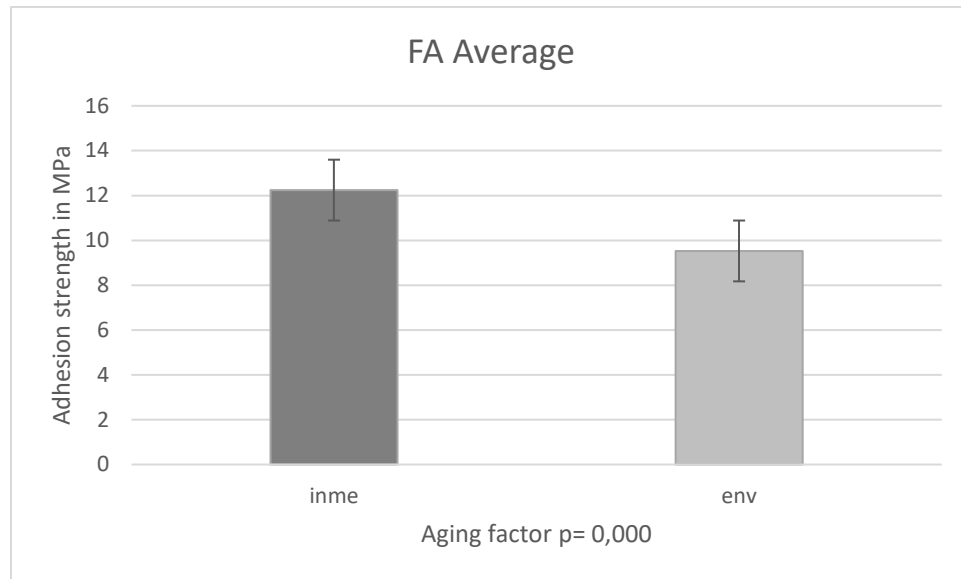


FIGURE 2

Adhesion Strength Between Immediate Groups (7 days of storage) and after aging (Tukey Test)

Regarding the types of failure, we found that the cohesive and mixed groups predominated, with the exception of the aged HF10 and HFS10 groups, in which there was a higher percentage of adhesive failure of 50% and 60%, respectively (Table 4). The types of failure were classified as adhesive, cohesive, and mixed. In the groups that were sandblasted and tested immediately, a higher frequency of cohesive and mixed failures was observed. Likewise, the sandblasted and tested groups once aged showed the same types of failures.

Table 5 shows that, when evaluating adhesive failures, the groups that were sandblasted and tested after seven days of storage and after the aging process, presented a higher frequency of cohesive and mixed failures.

TABLE 5

Types of adhesion failure found after analysis under the microscope (percentages)

Groups		Type of Failure		
		Adhesive	Cohesive	Mixt
Immediate (seven days after stored)	AL10	10	70	20
	ALS10	0	80	20
	HF10	10	90	0
	HFS10	30	50	20
	AHS	10	50	40
Aged	AL10	10	60	30
	ALS10	20	60	20
	HF10	50	40	10
	HFS10	60	10	30
	AHS	40	60	0

Source: the authors.

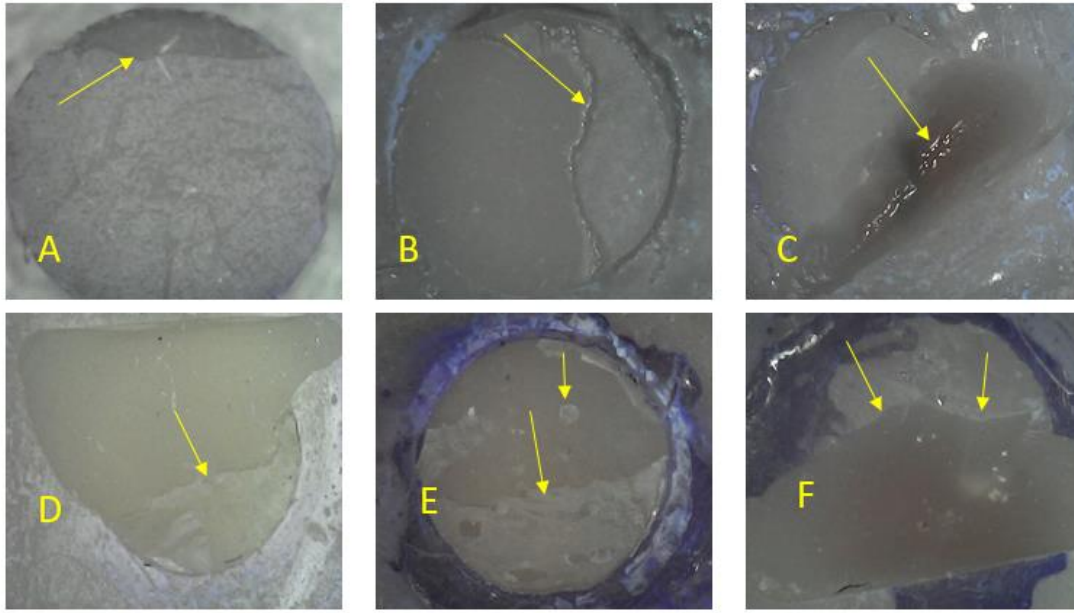


FIGURE 1

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

Figure 3 shows six microphotographs of the types of failures found in the material debonding test. A) An adhesive failure is observed in a sandblasted interface immediately tested. The arrow points to the boundary where the resin cement was. B) It shows a mixed failure, and the arrow marks the boundary between the cement and the ceramic. C) A cohesive failure is observed in which the ceramic broke throughout its thickness and the bottom of the test specimen can be seen. Images D to F show aged test specimens. In them, the arrows indicate the areas where moisture penetrated, producing hydrolysis and, therefore, fracture of the cement. Furthermore, F) allows a cohesive fracture to be observed after ageing of the material in the AHS group. A large fracture occurred in the ceramic with this surface treatment.

DISCUSSION

The present study measured the shear FA of an aged nanoceramic composite after being subjected to two surface treatments: sandblasting and acid etching. The purpose was to identify in a laboratory test what would happen if an attempt was made to repair a fracture that occurred in this material after it had already been used clinically. The study is justified by the fact that in Latin America the same materials used in other regions of the world are often not available. As for limitations, one can mention the lack of institutional and state funding, as well as the lack of specialized laboratories, so it is difficult to replicate all the conditions of the oral environment. For this reason, the inferences made about the findings can only be made in the context of the study alone. Other factors that could influence the effect studied include the presence of bacteria, chewing force, and a longer aging time, among others.

The results showed that both sandblasting and acid etching achieved similar adhesion values. Regarding the interaction between surface treatment and aging, the differences were not statistically significant ($p = 0.010$). However, the aging factor did show differences between the groups ($p = 0.000$). This suggests that it is possible to achieve adhesion to the material by means of these two techniques to obtain similar values (15). Even so, when analyzing the adhesive interface with the microscope, it was observed that, in most of the groups subjected to sandblasting, they suffered predominantly cohesive and

mixed type failures. In this study, this type of failure occurred in 80% to 90% with the sandblasting technique. They are similar to those reported by Lise, *et al.* (3).

Sandblasting with aluminum oxide (Al_2O_3) is a widely used technique to improve adhesion between different substrates (5,25-27). By means of this technique it is possible to obtain a rough surface that increases the surface area for adhesion between the resin cement and the ceramic (28). In addition, sandblasting removes contamination layers and forms large irregular grooves on the ceramic surface, allowing the resin to seep into the surface of the material (12,17). This improves the adhesion forces between a resin cement and a restorative material (17). The findings of this research confirm those reported by Niizuma, *et al.* (2) and Cinar, *et al.* (20). These authors found that sandblasting can cause damage to the polymer surface by generating microcracks that cause fissures that can continue to grow with the stress of temperature changes and end up fracturing the material.

This internal damage is caused by the force and short distance at which the sand particles are thrown at the ceramic. It is possible that the direction and size of the sand particle is a determining factor when creating microcracks (20,29). It is known that the size of the sand particles can be 20, 30, 50, 100, 110, and even 200 μm . (25). It is obvious to assume that the smaller the size, the less impact and damage to the ceramic will be. Whereas the larger the size, the more intense the impact force will be. In our study we used 50 μm sand and found large damages in the body of the material. We believe that with larger particles the internal damage will be greater (29). All of this is important because most clinicians delegate this procedure to the dental technician, without considering 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 may 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, as it could break again (29).

Another issue to consider is that sandblasting with Al_2O_3 particles directly into the patient's mouth should be avoided at all costs. The patient could inhale the aluminum particles, triggering serious lung problems (30). Therefore, in the face of so many disadvantageous findings, we would rule out this method and suggest it as a technique for repairing a composite of these directly in the mouth (23).

Regarding acid etching, some authors claim that hydrofluoric acid (HF) etching selectively dissolves the surface filler of the block to form microporosities, notches, and grooves. This roughens the material, which provides micromechanical retention and increases its surface energy (31-33). The Brava Block has an 80% inorganic matrix in the form of glass ceramic in its composition. For this reason, this polymer can be treated with this technique and obtain adhesion values equivalent to those of sandblasting. The findings of the present work only confirm what was described by Lise, *et al.*, in 2017 (3).

HF provides micromechanical retention because it dissolves the glassy phase of the nanoceramic making it rough, producing micro irregularities, and increasing the surface energy to improve FA (2, 21). Brava Block contains 80% silica. The action of HF dissolved such silica matrix (6), which is why HF had an effect similar to sandblasting. In addition, no fractures or breaks of the material were observed in the groups subjected to acid etching. This observation leads us to assume that microfractures do not occur through this technique as stated by Niizuma, *et al.* (2). It is recommended using HF as a surface treatment. Mixed failures were 0% in the immediate HF10 group and only 10% in the aged group. Therefore, in case of fractures of this material, it could be thought that it is better to attack it with acid if it is to be repaired. Obviously, special care should be taken, due to the danger of this acid, when working on a patient's soft tissues.

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

Regarding the adhesive used in this study, 10-methacryloyloxydecyl dihydrogen phosphate (10 MDP) is known to produce good results in bonding silica-based glass substrates to methacrylate-

terminated resinous materials (4,7). 10 MDP has two functional ends. One end is a methacrylate end capable of reacting with a resin cement and the other end is a phosphate end that is capable of chemically bonding to the silica of the ceramic. The long 10-carbon chain makes it very difficult for moisture to penetrate from one end to the other of the adhesive interface. This explains why the adhesion values did not decrease to critical values when aged (35,36).

The groups treated with silane ALS10, HFS10 and AHS 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 apply silane to 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 HF. We hypothesized that the FA would increase with the combination of these treatments. However, the FA was not higher than the rest of the groups. What was observed was that the cohesive failures were high (60%). Therefore, we can say that using these two surface treatments does not increase the adhesion strength and was even the cause of a higher percentage of cohesive and mixed fractures of the material (Table 5).

Regarding the method used, it has been proven that thermocycling is an ideal technique to simulate the aging conditions of restorative materials in the mouth (21,37). Indeed, in this study, it was found that adhesion values decreased when the adhesive and cement were stressed by heat and cold. In this way, it is possible to understand how restorations perform intraorally after some time of use. Although 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 know that the adhesive interface degrades over time, even with the best techniques developed recently (11).

CONCLUSIONS

Both sandblasting with Al₂O₃ particles and etching with 9.6% HF were able to generate similar FA between a resin cement and a nanoceramic composite.

Sandblasting produced a greater number of cohesive and mixed fractures.

Etching with 9.6% HF had a lower incidence of cohesive and mixed fractures.

FA values decreased in all groups after aging.

RECOMMENDATIONS

Our findings cannot be generalized, although they show trends. Further research is needed with random representative samples and under other conditions. Furthermore, new studies with different chemical formulations of the binding agents are recommended. The research protocol can be replicated with materials similar to the previous ones. Likewise, studies can be sought in patients in the medium and long term.

ACKNOWLEDGEMENTS

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

Contribution of the authors. MCC: idea and design of the study, statistical analysis, writing and revisions of the manuscript. KRF: idea of the study, conducting experiment, writing and revisions of the manuscript. VAT: writing and revisions of the manuscript. ABS: assistance during the experiment, writing of methods section, and revisions of the manuscript. JTG: assistance during the experiment, writing of methods section, and revisions of the manuscript.

References

1. Cevallos GQ, Flores LC, Guamán KG, Gonzaga MC, Bedoya PC, López VQ, Cascante Calderón 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/10.1155/2018/2598073>
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-proteticos/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. <http://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. Günel-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 microhíbrida. 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>

* Original research.

How to cite this article: Cascante-Calderón MG, Reascos Flores KA, Villacís Altamirano IM, Bayas Salinas AM, Taraguay Galindo JE. Fracture Resistance of a CAD/CAM Nanoceramic Repaired with Two Surface Treatments: *In Vitro* Study. Univ Odontol. 2023; 42. <https://doi.org/10.11144/Javeriana.uo42.frcc>