Adhesive Strength Between a Nickel-Chromium Alloy and a Resin Cement Subjected to Different Surface Treatments *

Resistencia adhesiva entre una aleación de níquel-cromo y un cemento resinoso sometidos a diferentes tratamientos de superficie

Resistência adesiva entre uma liga de níquel-cromo e um cimento resinoso submetido a diferentes tratamentos de superfície

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Abstract:

Background: The constant need to improve adhesive systems leads manufacturers to develop new materials that offer optimal results. Manufacturers claim that new adhesives produce a strong and durable bond between resin cements and restorative materials including metals. Purpose: To measure the adhesive resistance between a resin cement and a nickel-chromium (Ni-Cr) alloy at 24 hours and after aging. Methods: This was an in vitro experimental study. 40 Ni-Cr metallic cylinders were assigned to 4 groups (n=10). The surfaces were mechanically sanded and abraded with 50 µm Al_2O_3 particles (mechanical surface treatment). The chemical surface treatment included a metallic primer, silane, and universal adhesive. Subsequently, two cubes of nanohybrid resin were cemented to each metal cylinder with resin cement, under a constant load of 0.98 N for 5 minutes. An immediate shear test (24 hours) and after aging (5,000 thermal cycles) was carried out. Results: The highest values of adhesive resistance were obtained with the GSUNIVERSAL group both when it was measured immediately and when aged. Conclusions: The mechanical and chemical treatment based on 10-MDP plus silane improved the adhesion between a Ni-Cr alloy and a resin cement, even after aging. **Keywords:** adhesive interface, adhesive strength, dental materials, dentistry, metal alloy, metallic primer, resin cement, surface treatment, universal adhesive.

Resumen:

Antecedentes: La constante necesidad de mejorar los sistemas adhesivos lleva a los fabricantes a desarrollar nuevos materiales que ofrecen resultados óptimos. Ellos aseguran que los nuevos adhesivos producen una unión fuerte y duradera entre los cementos resinosos y los materiales restauradores, incluso metales. Objetivo: Medir la resistencia adhesiva entre un cemento resinoso y una aleación de níquel-cromo (Ni-Cr) a las 24 horas y posterior al envejecimiento. Métodos: Se realizó un estudio experimental in vitro. Se utilizaron 40 cilindros metálicos de Ni-Cr que conformaron 4 grupos (n=10). Las superficies se lijaron mecánicamente y con partículas de Al_2O_3 de 50 µm (tratamiento superficial mecánico). Para el tratamiento superficial químico se empleó un diseño experimental que consistió en colocar: un imprimador metálico, silano y adhesivo universal. Posteriormente, a cada cilindro de metal se le cementaron dos cubos de resina nanohíbrida con cemento resinoso, bajo una carga constante de 0,98 N durante 5 minutos. Se realizó un ensayo de cizallamiento inmediato (24 horas) y después del envejecimiento (5.000 ciclos térmicos). Resultados: Los valores más altos de resistencia adhesiva se obtuvieron con el grupo GSUNIVERSAL tanto cuando fue medido inmediatamente como cuando fue envejecido. Conclusiones: Los tratamiento mecánico y químico a base de 10-MDP más silano mejoran la adhesión entre una aleación de Ni-Cr y un cemento resinoso, inclusive después de envejecida.

Palabras clave: adhesivo universal, aleación metálica, cemento resinoso, imprimador metálico, interfase adhesiva, materiales dentales, odontología, resistencia adhesiva, tratamiento superficial.

Resumo:

Antecedentes: A constante necessidade de melhorar os sistemas adesivos leva os fabricantes a desenvolver novos materiais que oferecem ótimos resultados. Os fabricantes afirmam que os novos adesivos produzem uma ligação forte e durável entre cimentos resinosos e materiais restauradores, incluindo metais. Objetivo: Medir a resistência adesiva entre um cimento resinoso e uma liga de níquel-cromo (Ni-Cr) após 24 horas e após o envelhecimento. Métodos: Este foi um estudo experimental in vitro. 40 cilindros metálicos de Ni-Cr foram divididos em 4 grupos (n=10). As superfícies foram lixadas mecanicamente e com partículas de Al.O. de 50 µm (tratamento superficial mecânico). O tratamento químico da superfície incluiu primer metálico, silano e adesivo universal. Posteriormente, dois cubos de resina nanohíbrida foram cimentados a cada cilindro metálico com cimento resinoso, sob carga constante de 0,98 N por 5 minutos. Foi realizado um teste de cisalhamento imediato (24 horas) e após envelhecimento (5.000 ciclos térmicos). Resultados: Os maiores valores de resistência adesiva foram obtidos com o grupo GSUNIVERSAL tanto quando medido imediatamente como quando envelhecido. Conclusões: O tratamento mecânico e químico à base de 10-MDP mais silano melhorou a adesão entre uma liga de Ni-Cr e um cimento resinoso, mesmo após o envelhecimento.

Palavras-chave: adesivo universal, cimento resinoso, força adesiva, interface adesiva, lixa metálica, materiais dentários, odontologia, primer metálico, tratamento da superfície.

INTRODUCTION

Metallic structures for prosthetic restorations, such as single crowns, short bridges, intra-radicular posts, and inlays made of nickel-chromium alloys (1,2) are still frequently used due to their rigidity, high resistance to corrosion (3,4), and relatively low cost (1). However, this type of restoration can fail due to detachment or de-cementation, mainly due to marginal mismatches and moisture infiltration (5,6). Achieving greater longevity of the restoration relies on obtaining a strong and lasting adhesive bond of the cement-adhesive-metal interface (7). This bond has been a challenge in restorative dentistry because the prosthetic pieces are usually cemented with powder-liquid cements. These cements have the disadvantage that they easily degrade in the presence of moisture. To overcome this problem, currently, it is possible to have resin cements that do not have this limitation. However, ensuring a proper bond between a resin cement and a metal substrate is not as simple as just mixing a powder and a liquid. On the contrary, studies show that a treatment must always be applied to the surface of the metal. Surface treatments to improve adhesion are mechanical, chemical, or a combination of both (1,8).

For all these reasons, manufacturers of restorative materials are increasingly introducing new adhesives, which have complex molecules and compositions promising to chemically adhere to a metal alloy (9-13). The literature reports that the composition of these materials incorporates active monomers, whether sulfur, phosphoric, or carboxylic, such as biphenyl-dimethacrylate (BPDM) and 10 methacryloxydecyl dihydrogen phosphate (10-MDP), which could act on the metal surface with particularly good results (1,7,14,15). However, dental clinicians and students could be confused and overwhelmed by these new products and techniques, since most studies have focused on adhesion to enamel and dentin and not so much on the bonding of metal alloys to different types of materials such as ceramics or zirconia (1,16).

Indeed, few investigators have evaluated the efficacy of these agents on base metal alloys, let alone sufficient studies on nickel-chromium alloys (7). Studies indicate that the combination of these new molecules with coupling agents such as silanes would improve their adhesive potential since these also function as a bridge to connect the different types of materials (10,12). Conclusive results are not yet available. In the same way, resin cements today are gaining popularity because they improve adhesion with their capability to produce a chemical bond with the oxide layer created on the metal surface (17). In addition, they better resist the stress generated during chewing due to their resinous matrix (18).

Taking into consideration all these aspects, the following research question was posed: In the nickel-chromium alloy, will the BPDM-based metal primer together with the resin cement present higher adhesive resistance or strength (AR) to shear than the 10-MDP molecule after applying artificial aging cycles? The purpose of this study was to measure the bond strength between a resin cement and a nickel-chromium alloy against different surface treatments at 24 hours (immediate) and after aging. The null hypothesis was that the AR would be the same in all groups at 24 hours and after applying the aging cycles. We conducted this study because, in recent years, most of the research has focused on adhesion to enamel and dentin, but not enough evidence has been provided on the adhesive bonding of metal alloys to resin cements using metallic primers or universal adhesives based on 10-MDP.

MATERIALS AND METHODS

This was an experimental *in vitro* study. The sample size was calculated after conducting pilot tests and submitting the results to a power test (Minitab 19®, Pennsylvania, USA). The study sample consisted of 40 metallic Ni-Cr detection cylinders (BesQual®, USA) with a diameter of 12.5 mm and a height of 7 mm, which were divided into 4 groups (n=10 per group), and 803 mm x 3 mm x 4 mm cubes of nanohybrid resin (Neofil®, Kerr, USA). The type of resin used is commonly available in the market.

The experiment operators created a custom aluminum matrix to elaborate the resin cubes. The matrix was held with a small pressure wrench that was placed on a glass tile. To pack the nanohybrid resin inside the matrix, we used a titanium gutta-percha obturator and a composite modeling instrument and light-cured each resin layer for 40 s with a curing light (LED.C Woodpecker®, China).

The 40 metal cylinders were randomly divided into 4 groups (i.e., GCONTROL, GZPRIMER, GSPLUS, and GSUNIVERSAL) and mechanically polished using an electric micromotor (Marathon 3 Champion®, Saeyang Microtech Co. Ltd., China) with 400 and 600 grit metal sandpaper for 20 s on each grit (7,18). The mechanical surface treatment was the same in all groups: sandblasting with 50 μm Al2O3 particles (Dentaurum®, USA), at a 10-mm distance measured with a 0.25-mm orthodontic wire attached to the nozzle of the dental office blaster (Bio Art®, Sao Paulo, SP, Brazil), with circular movements for 20 s (7,19). After the sandblasting, we stored the cylinders in a plastic container to avoid contamination and implemented the adhesion protocol.

A chemical surface treatment following each manufacturer's indications was applied to all groups, with the exception of the GCONTROL group. A BPDM-based priming agent (Z-Prime® plus, Bisco, USA) was used with the GZPRIMER group. The primer contained a carboxylic acid monomer to promote chemical adhesion in the chromium oxide on the metal surface through one of its methacrylate terminals (20-22). The primer was applied with a micro-brush, evenly moistening the adhesion surface. Finally, an air blast was applied for 5 s with the oil-free 3-way syringe.

The GSPLUS group received a thin layer of silane (Monobond®-N, Ivoclar Vivadent AG, Liechtenstein), leaving it for 60 s and then air blasting any excess with an oil-free 3-way syringe. Z-Prime™ plus was then applied with a micro-brush, evenly saturating the adhesion surface, and blowing air for 5 s with the 3-way syringe.

A thin layer of Monobond®-N was applied to the GSUNIVERSAL group, allowing it to act for 60 s and eliminating any excess with a blast of air from the 3-way syringe. Afterwards, a 10-MDP-based adhesive (Single Bond Universal, 3M® ESPE, USA) was applied by rubbing the surface for 20 s and then gently air blasted with the 3-way syringe for 5 s. Finally, the adhesive was light-cured for 10 s with the curing light. Figure 1 summarizes the adhesion protocols.

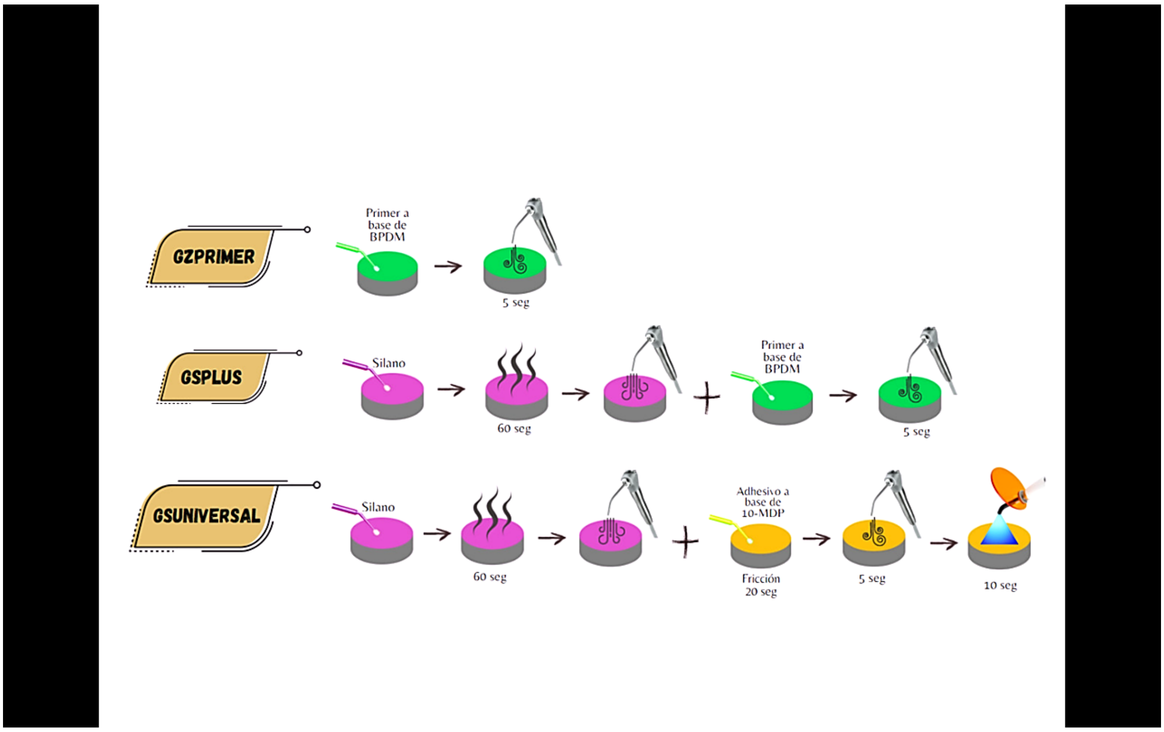

FIGURE 1
Adhesion Protocol
Source: the authors.

The protocol included the application of a dual resin cement (Allcem®, FGM, Joinville, Brazil), which was mixed for 8 s, in the 4 groups of samples with a 1:1 ratio. A small fraction of cement was placed at one end of the resin cube with a fine marten fur brush. We did not use micro-applicators because we observed fibers of the micro-applicator under the microscope in the pilot tests, which could interfere with adhesive resistance. The resin cube was carefully adhered to the metal surface and immediately cemented under a constant load of 0.98 Newtons for 5 min.

Three minutes after starting to mix the cement, we removed any excess with an OMS periodontal probe. Immediately afterwards, we removed the constant load and light-cured each side for 40 s. As a final product, we obtained two trial pieces cemented on a metallic cylinder (Figure 2). After completing the cementation protocol, we waited 20 min to store the cylinders in water at room temperature.

Two measurements (immediate and aged) were taken on each sample via a universal testing machine (Muver/5053®, Software Muver Cx Server Lite) in which the test bodies were sheared at a speed of 1.0 mm/min until failure (Figure 2). These parameters followed the ISO 29022:2013 standard (23).

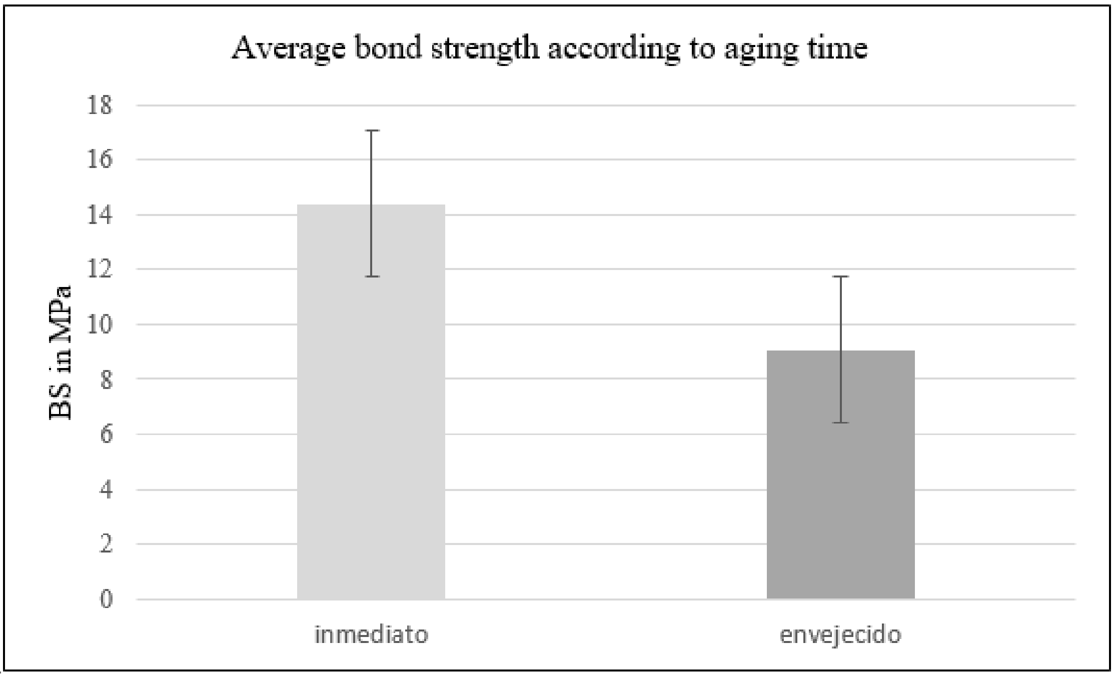

FIGURE 2
Shear Bond Strength Test
Source: the authors.

For the aging of the samples, 5,000 thermal cycles of thermal stress were carried out in a thermal cycler, with temperatures between + 5 ºC and + 55 ºC with a duration of 30 s and a transfer time of 5 s. This number of cycles is equivalent to 6-month aging in the oral cavity (24). The methods for artificial aging of the *in vitro* adhesive interface are storage in water and thermocycling. Thermocycling was used because it partly simulates the conditions of the mouth. Temperature variations can expand and shrink the resin-based materials involved in this interface, which can compromise the bonding and reduce the AR between the resin cement and the Ni-Cr metal alloy (7,25–27).

The shear bond strength was calculated by dividing the force (measured in Newtons and given by the machine) by the area in mm2 of the adherent section of the resin cube (which was calculated with the help of a Starrett® Digital Caliper, USA) and the resulting values were expressed in MPa. The type of failure was classified as: a) adhesive failure, if the sandblasting pattern is observed in 3 or more quadrants; b) cohesive failure, if cement remains are observed in 3 or more quadrants; and c) mixed failure, if cement remains and sandblasting pattern in a 2:2 ratio in the quadrants are observed (7,19,28). Adhesive failures were observed by light microscope (Oxbird®, China) at 50X magnification.

Statistical Analysis

All the data were recorded in an Excel® spreadsheet and analyzed using the Minitab® Statistical 19 software (State College, Pennsylvania-USA). Descriptive statistics included the AR mean and standard deviation (SD) for each group. The data were subjected to the Anderson Darling normality test, which, with a p = 0.065, showed that the data were normal. Finally, a two-way ANOVA (surface treatment and aging time) was performed, followed by a Tukey Post Hoc test.

RESULTS

The average and SD of the shear bond strength for each group are presented in Table 1.

TABLE 1
Mean Bonding Strength (MPa) and SD for All Groups

Groups	N	Immediate BS (MPa)		Aged BS (MPa)	
		Mean	SD	Mean	SD
GCONTROL	10	12,3	±0,79	8,2	±1,65
GZPRIMER	10	12,2	±2,49	9,1	±1,79
GSPLUS	10	13,4	±2,80	8,9	±2,56
GSUNIVERSAL	10	19,6	±5,02	12,3	±2,32

Source: the authors.

The highest AR average was obtained with the GSUNIVERSAL group, both immediate (19.6 MPa) and aged (12.3 MPa). The other groups showed average values between 12.2 and 13.4 MPa when measured immediately. Regarding the after-aging values, there was a decrease in the AR at averages between 8.2 MPa (control group) and 9.1 MPa (GZPRIMER group), which had the second-best value. Neither spontaneous detachment was observed at 24 h, nor during and after thermocycling in any of the groups. The ANOVA analysis showed there was a significant difference in surface treatment and aging time with p = 0.000 (Table 2).

TABLE 2
Two-Factor ANOVA. Surface Treatment and Aging Time

Factor	DF	ADJ SC	ADJ MC	F	P
Surface treatment	4	442,13	110,532	14,80	0,000
Aging time	1	423,53	423,526	56,70	0,000
Error	74	552,72	7,469		

Source: the authors.

The Tukey's Post Hoc test, with 95 % confidence, showed that the GSUNIVERSAL group was statistically different from the other groups in the surface treatment factor. The other groups were statistically equal among themselves (Table 3).

TABLE 3
The Post hoc Tukey Test *

Surface treatment	N	Mean	Group
GSUNIVERSAL	20	15,9305	A
GZPRIMER	10	11,7658	B
GSPLUS	20	11,0915	B
GCONTROL	20	10,2205	B
GZPRIMER	10	9,5752	B

* Means that do not share a letter are significantly different.
Source: the authors.

The results of Tukey's pairwise comparisons in the aging time factor indicate that there was a significant difference between the two groups The average was 14.37 MPa for the immediate group and 9.05 MPa for the aged ones (Figure 3).

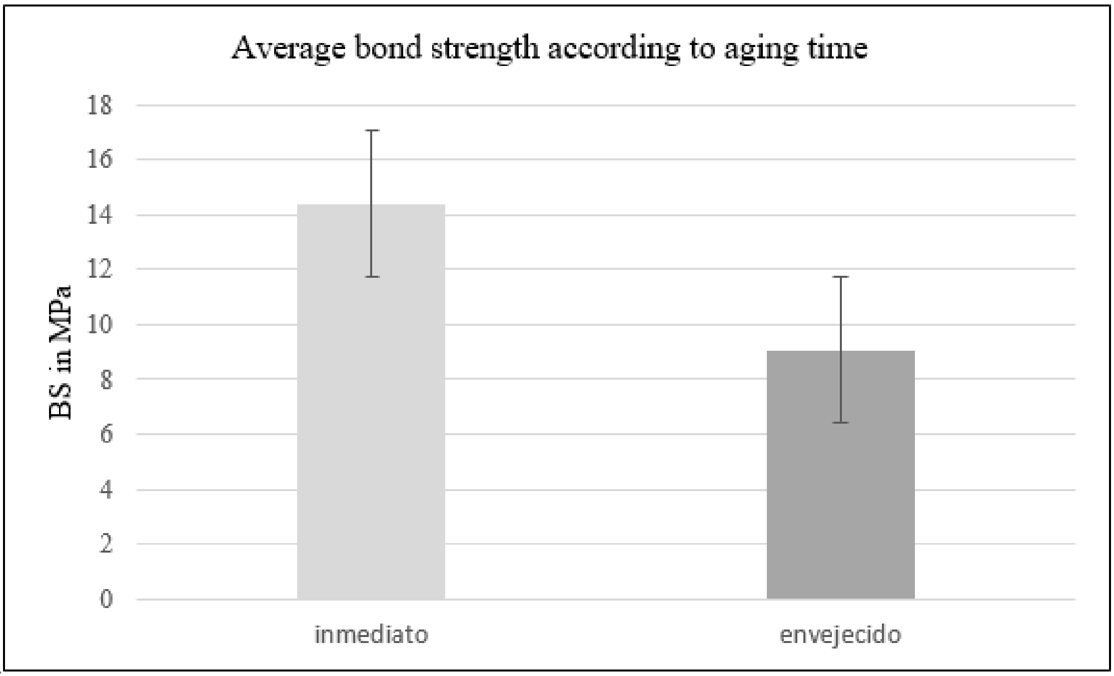

FIGURE 3
Tukey's Pairwise Comparison of Immediate and Aged Groups
Source: the authors.

In what has to do with the different types of failures, they were represented in percentages and can be seen in Table 4. Cohesive-type failures were the most prevalent with frequencies ranging from 50 % to 100 %, in all groups, with the exception of the control group that only presented 10 % of this type of failure. An interesting finding was the 50 % frequency of mixed failures for the aged GSUNIVERSAL group. GCONTROL adhesive failures were 90 % when they were from the immediate group and 50 % from the same group. The most representative microphotographs of the types of failure can be seen in Figure 4.

TABLE 4
Failure Mode Percentage of All Groups

Bond Strength	Group	Failure mode (percentage)		
		Adhesive	Cohesive	Mixed
Immediate	GCONTROL	90	10	0
	GZPRIMER	0	100	0
	GSPLUS	0	100	0
	GSUNIVERSAL	0	100	0
Aged	GCONTROL	50	50	0
	GZPRIMER	0	100	0
	GSPLUS	0	100	0
	GSUNIVERSAL	0	50	50

Source: the authors.

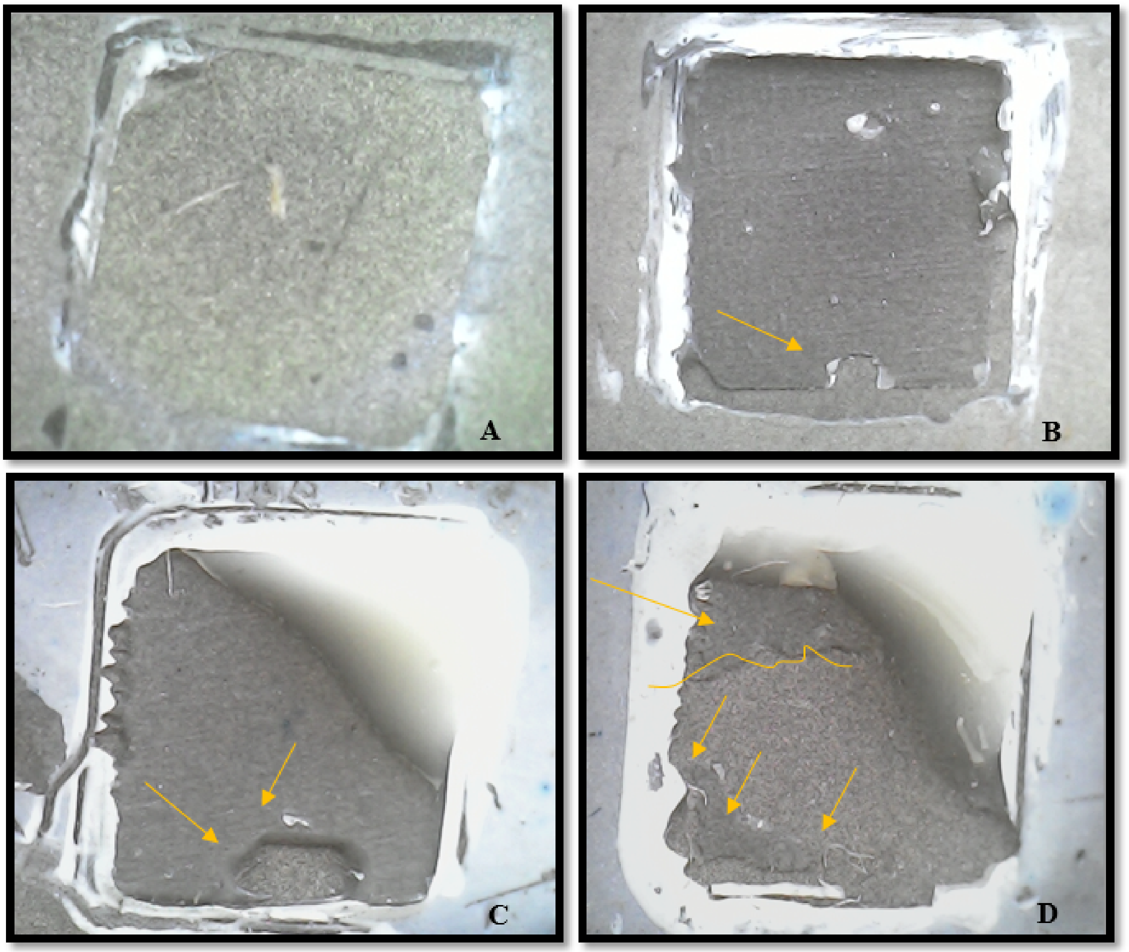

FIGURE 4
Failure modes: (A) Adhesive failure, no cement remains. (B) Cohesive failure, the yellow arrow indicates a fractured cement rest. (C) Cohesive failure, including substrate, the yellow arrow shows the fracture of the cement. (D) Mixed failure, where yellow arrows show the remains of fractured cement. Photomicrographs with an optical microscope, 50x magnification.
Source: the authors.

DISCUSSION

Achieving an adequate adhesive bond between a metallic substrate and a resin cement leads to the success and longevity of a prosthetic restoration. The present investigation was conducted with the purpose of evaluating the effect on the adhesive resistance produced by a metallic primer based on BPDM and one based on 10-MDP between a resin cement and a nickel-chromium alloy.

Sandblasting proved to be a good mechanical method to improve adhesion since it produces greater surface roughness, cleaning of surface contaminants, which determines an increase in surface energy, and a more uniform sandblasting pattern for retention (1,7,18). This is the reason all groups withstood the entire study without detachment, even in the control group with no metallic primer. However, the AR values in the latter group were the worst after aging (8.3 MPa), which indicates the need to complement the sandblasting with some type of chemical bond.

The granulation of the sand is also important (25). There are in the dental market particles with different granulations, such as 25, 50, 100, 110, and even 200 µm. It can be inferred that each granulation achieves a different effect on the sandblasted surface. For this research, 50 µm aluminum oxide particles were used following parameters that have been established in various studies (8,9). It is likely that with a larger particle size the results will be different. More research is needed on this matter.

Regarding the GSUNIVERSAL group that reached the highest adhesion values, both immediately and after aging, these results only confirm what was reported by researchers such as Nima, *et al.* (1), Papadogiannis, *et al.* (16), Jamel, *et al.* (18), Al-Heou and Swed (29), and Bankoğlu, *et al.* (30). The GSUNIVERSAL group was sandblasted and immediately afterwards silane and a universal adhesive (Single Bond Universal®, 3M, St. Louis. Missouri. USA) were applied. This adhesive was our gold standard due to the favorable capacity of chemical bonding to various substrates that the 10-MDP molecule presents. This molecule is formed by two ends: a methacrylate ($CH_3 – CH_2 =$) and a phosphate radical PO4 (7,29,31) with a long chain of 10 carbons in the middle. The methacrylate end connects to an equal end that is present in resins and resin cements (25). On the other side, the phosphate end connects to the oxygen present on the surface of the metal alloy (4,32,33) by means of covalent bonds (34).

While it is true that these bonds are extraordinarily strong, they can weaken with changes in temperature. Indeed, our study only confirmed what was reported by Marchesi, *et al.* (35), who found that after having kept the samples in water for one-year, low values were obtained when using a universal adhesive system, since the stability of the interface gets lost over time. In the long term, 10-MDP can absorb moisture due to the phosphate radical, which would cause a degradation of the polymer network and consequently, a reduction in the bond strength of the materials (16,36,37). In any case, in our study, despite the fact that the initial values decreased with aging, they are still interesting and good.

To try to reverse this phenomenon, we applied a silane (Monobond®-N Ivoclar Vivadent, Liechtenstein), as the manufacturer recommends for connecting metals. In addition, researchers reported that these compounds improve surface moistening of the adhesive (12).

Silanes also have two functional ends, a methacrylate and a SiO-. It is through one of the free valences of O_2 that the union to the metallic alloy occurs. However, the silanes used in dentistry are small molecules that could not resist hydrolytic degradation. The results of this group indicate that it could be indicated to place a silane prior to an adhesive with 10-MDP. However, Jung, *et al.* (25), mention that the different chemical treatments do not improve the adhesive bond between the resin cement and the metal alloy, since the previously sandblasted irregular surface tends to fill up. All of this could explain why AR values decreased with aging, although they remained high compared to the other groups.

Finally, regarding the type of failure, in this group it could be seen on Table 4 that the cohesive failures were 100 % in the immediate and the aged samples. This indicates that the adhesion with this treatment was particularly good between the substrates, that the cement had to fracture before detaching (38). These results suggest that the application of a silane layer, followed by an adhesive agent, could have contributed to improving the AR values, as reported by Nima, *et al.*, (1).

Regarding the GZPRIMER and GSPLUS groups, they showed the second highest values in our study. The Z-Primer contains a bonding agent designed to act on dental zirconia. It is highly likely that when used in a metallic alloy its performance may be less effective. The BPDM molecule has two benzene rings inside and a HEMA group with two OH- and two O= groups at both ends. These ends react with the OH- hydroxyl terminals present on the surface of a zirconia or a metal.

After comparing the results of these groups with the control group, we can reaffirm that a chemical treatment is necessary regardless of the molecule, whatever it is, as it ensures a better adhesion. Our results confirm findings by Fonseca, *et al.* (7), who obtained similar values using metal primers applied to a Ni-Cr alloy before and after thermocycling. Although manufacturers suggest using a metallic primer before applying a resin cement, Di Francescantonio, *et al.* (15), reported that this procedure does not necessarily increase the bond strength because these primers have different functional monomers, which may interact differently with the oxide layer on the alloy surface. We disagree with them, since we found that the application of a BPDM-based metallic primer did improve adhesion forces.

Regarding the groups after aging, the explanation may be the short length of the spacer chain of the BPDM monomer, which can make it lose its effectiveness in the presence of humidity (39). However, those were not bad values despite being stressed with heat and cold. Recent studies have suggested that the effect of thermocycling depended on the chemical bonding potential of the functional monomer at the interface, since these materials have different compositions, which contributes to the long-term stability of the adhesive bond (7,20,40,41).

With the GSPLUS group, the combination of silane with the BPDM-based metal primer may have contributed to a competitive relationship. They would try to connect with the hydroxyl terminals found on the surface of the metal, which somehow, instead of potentiating this union, end up harming it, causing the AR to be lower than expected. It will be necessary to investigate whether this primer by itself is capable of reaching higher values of junction resistance. Also, it could be assumed that when a primer combined with another coupling agent is applied after sandblasting, irregularities in the alloy surface can be filled in somewhat affecting the overall bond strength (34).

Regarding the resin cement used in the study, we observed that the combination of chemical treatments, along with the mechanical treatment, does improve the adhesion between the cement and the Ni-Cr alloy. Resin cements have low solubility in the oral environment, which allows them to withstand the hydrolytic degradation of the oral environment (8,42), unlike traditional powder-liquid cements. In addition, its methacrylate groups react with the methacrylate group of the adhesives ensuring high adhesion forces. One factor to consider is polymerization shrinkage. It is known that this phenomenon is related to the volume of the resin. A small volume will reduce the effects of contraction and being a dual-action cement, it will provide a slower reaction of the components, which will reduce the contraction stress (15,32,40,43). This partly explains the results obtained in all groups.

Finally, low values after artificial aging might arise due to various situations. Taira and Kamada (27) argue that the adhesive bond always tends to decrease due to the stress produced by heat and cold (30,38,44). This was verified in this study.

CONCLUSIONS

Sandblasting followed by application of a silane plus 10-MDP-based adhesive improved the bond strength between a resin cement and Ni-Cr brilliance, both immediately and after aging. A mechanical and chemical surface treatment is always necessary to improve adhesion values between a Ni-Cr layer and a resin cement. All surface treatments, mechanical and chemical, lose effectiveness over time in a Ni-Cr lesion.

RECOMMENDATIONS

It is recommended to sandblast and treat the surface of a Ni-Cr metal alloy with chemical bonding agents before cementing a restoration with resin cements. More *in vitro* studies as well as clinical follow-ups are needed before having conclusive results.

Conflict of interest: The authors declare not to have conflict of interest.

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Notes

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