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Evaluation of the adhesive bond strength to zirconia using different adhesive systems with 10-MDP.

INVESTIGATION

Evaluación de la resistencia de unión adhesiva a la zirconia utilizando distintos sistemas adhesivos con 10-MDP

Avaliação da resistência adesiva à zircônia utilizando diferentes sistemas adesivos com 10-MDP.

Abstract

Objective: To analyze the immediate shear bond strength and the strength after a period of delay to zirconia restorations using adhesive systems available in our market containing the 10-MDP molecule.

Methodology: Twenty sintered zirconia specimens were randomly divided into four groups based on the chemical conditioning system used (Z Prime Plus, Peak ZM, Single Bond Universal, and Tetric N-Bond Universal), which were applied following the manufacturer's instructions. The zirconia surface was pretreated with 50-micrometer aluminum oxide sandblasting for 15 seconds, at a distance of 10 mm and a pressure of 0.25 MPa. The corresponding chemical conditioning agent was then applied, followed by the fabrication of four dual resin cement buttons. Two of the four buttons underwent microshear testing 24 hours after preparation. Subsequently, the specimens were stored in distilled water at 37°C for six months, after which the remaining two buttons were subjected to microshear testing. The results were analyzed using two-way ANOVA and Tukey's post hoc test.

Results: No significant differences were observed when analyzing the chemical conditioning system nor the delay factor. When the factors were analyzed separately, no differences were found between the materials at 24 hours; however, at 6 months, the Z Prime Plus group showed a statistically significant decrease.

Conclusions: The results of this in vitro study suggest that universal adhesives with 10-MDP exhibit an immediate bond strength similar to that of zirconia-specific chemical surface conditioners. After a period of delay, universal adhesives appear to have better stability in bond strength.

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Resumen

Objetivo: analizar la resistencia de unión cizallamiento inmediata y después de un periodo de envejecimiento en restauraciones de zirconia utilizando sistemas adhesivos que contienen la molécula 10-MDP en su composición.

Metodología: 20 especímenes de zirconia sinterizada se dividieron aleatoriamente en 4 grupos según el sistema de acondicionamiento químico a utilizar (Z Prime Plus, Peak ZM, Single Bond Universal, Tetric N-Bond Universal), los cuales se aplicaron siguiendo las indicaciones del fabricante. La superficie de zirconia fue pretratada con un arenado a óxido de aluminio de 50 micrómetros de tamaño, durante 15 segundos a una distancia de 10 mm y una presión de 0.25 MPa. Posteriormente se aplicó el agente de acondicionamiento químico correspondiente y finalmente se confeccionaron 4 botones de agente de fijación resinoso resinoso dual. 2 de los 4 botones fueron sometidos al ensayo de microcizallamiento de forma inmediata luego de 24 hrs. Seguidamente los especímenes fueron almacenados en agua destilada a 37ºC durante 6 meses y pasado ese periodo se sometió al microcizallamiento los 2 botones restantes. Los resultados obtenidos se analizaron mediante ANOVA de 2 vías y un test posthoc de Tukey.

Resultados: No se encontraron diferencias significativas al analizar el factor del sistema de acondicionamiento química ni al analizar el factor envejecimiento. Al analizar los factores independientemente, no se encontraron diferencias entre ninguno de los materiales a las 24 horas, sin embargo, a los 6 meses el grupo Z Prime Plus presentó una disminución estadísticamente significativa.

Conclusiones: Los resultados de este estudio in vitro sugieren que los adhesivos universales con 10-MDP presentan una resistencia de unión inmediata similar a los acondicionadores de superficie químicos específicos para zirconia. Después de un periodo de envejecimiento, los adhesivos universales parecen tener una mejor estabilidad en la resistencia de unión.

Palabras Clave: Microcizallamiento, Zirconia, Cementado Adhesivo

Resumo

Objetivo: analisar a resistência de união imediata ao cisalhamento e após período de envelhecimento em restaurações de zircônia utilizando sistemas adesivos disponíveis em nosso mercado que contenham a molécula 10-MDP em sua composição.

Metodologia: 20 corpos de prova de zircônia sinterizada foram divididos aleatoriamente em 4 grupos de acordo com o sistema de condicionamento químico a ser utilizado (Z Prime Plus, Peak ZM, Single Bond Universal, Tetric N-Bond Universal), os quais foram aplicados seguindo as instruções do fabricante. A superfície da Zircônia foi pré-tratada com jato de areia de óxido de alumínio de 50 micrômetros de tamanho, por 15 segundos a uma distância de 10 mm e pressão de 0,25 MPa. Posteriormente foi aplicado o agente condicionador químico correspondente e por fim foram confeccionados 4 botões duplos de cimento resinoso. 2 dos 4 botões foram submetidos ao teste de microcisalhamento imediatamente após 24 horas. Os corpos de prova foram então armazenados em água destilada a 37ºC por 6 meses e após esse período os 2 botões restantes foram submetidos ao microcisalhamento. Os resultados obtidos foram analisados por meio de ANOVA de 2 fatores e teste posthoc de Tukey.

Resultados: Não foram encontradas diferenças significativas ao analisar o fator sistema de condicionamento químico ou ao analisar o fator envelhecimento. Ao analisar os fatores de forma independente, não foram encontradas diferenças entre nenhum dos materiais às 24 horas, porém às 6 meses o grupo Z Prime Plus apresentou diminuição estatisticamente significativa.

Conclusões: Os resultados deste estudo in vitro sugerem que os adesivos universais com 10-MDP apresentam resistência de união imediata semelhante aos condicionadores químicos de superfície específicos para zircônia. Após um período de envelhecimento, os adesivos universais parecem ter melhor estabilidade na resistência de união.

Palavras-chave: Microcisalhamento, Zircônia, Cimentação Adesiva

Introduction

Currently, the development of ceramic biomaterials, such as zirconia for dental applications, is booming. Zirconia restorations are applied using digital systems based on computer-aided design and manufacturing (CAD/CAM). [1,2] Yttria-stabilized tetragonal zirconia polycrystal (YTZP) was initially introduced as a substructure for fully ceramic restorations. Zirconia restorations in dentistry, both for tooth- and implant-supported crowns and bridges, became popular due to their high fracture toughness compared to silica-based ceramics. Zirconia exhibits a flexural strength exceeding 900 MPa, fracture toughness ranging from 4 to 8 MPa√m, and an elastic modulus of 210 GPa.[3-5] In addition to its mechanical superiority, zirconia offers exceptional biocompatibility,[1,6] high chemical stability, [2,7] and favorable optical properties. [6,7]

However, when zirconia restorations are made on substrates with poor mechanical retention, their clinical performance largely depends on establishing strong adhesion between the cementing agent and the zirconia.[8-10] Adhesive treatments used for other dental ceramics are not effective on zirconia surfaces because it is chemically inert and non-polar, making it resistant to acid attacks due to the absence of vitreous phases in its composition. Furthermore, the lack of silica in its composition prevents chemical bonding with silane agents. [11-13]

Therefore, several methods have been proposed for bonding zirconia restorations, including resin-modified glass ionomer cements and both conventional and self-adhesive resin bonding agents. The use of resin bonding agents on zirconia requires chemical preconditioning with monomers containing specific functional groups. The application of products containing phosphate ester monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), has been extensively documented. When applied after mechanical pretreatment of the zirconia surface, they significantly increase adhesive strength. [6] Conditioning with 10-MDP monomers is considered a non-destructive chemical treatment that functionalizes the inert surface of YTZP, enabling chemical bonding to the surface. [14-16]

In an effort to simplify adhesive protocols, manufacturers have incorporated monomers with specific functional groups, such as 10-MDP and silane agents, into multipurpose adhesive systems called Universal adhesives. These systems are designed to be faster, less technique-sensitive, and easier to use, forming adhesive bonds with different substrates—including metals, vitreous ceramics, dentin, and enamel—without requiring surface pretreatment. [16,17] Sandblasting with alumina particles on the zirconia surface, combined with the application of specific functional monomers, remains the protocol established in the literature and recommended by several manufacturers. [4,18-20]

However, the durability of this bond under artificially induced delay conditions remains controversial. [18-21] The establishment of an optimal bonding protocol for zirconia restorations has yet to be defined, making it an area of significant scientific interest and development. Establishing such protocols would improve the clinical performance and longevity of zirconia restorations. Given the above, this study aimed to analyze the immediate shear bond strength and the bond strength after a period of delay in zirconia restorations using adhesive systems containing the 10-MDP molecule in their composition. The null hypothesis to be tested is that no differences will be found in shear bond strength nor durability among the different chemical conditioning agents.

Methodology

SPECIMEN PREPARATION

A partially sintered zirconia disc (Ceramill Zolid, Amann Girrbach AG; Koblach, Austria) was cut using a microcutter (LECO VC50, LECO; St. Joseph, MI, USA) to create rectangular specimens measuring 20 mm x 20 mm x 3 mm. Five specimens were prepared for each group, resulting in a total of 20 specimens. After milling, the specimens were sintered according to the manufacturer's specifications using a Ceramill Therm 3 high-temperature furnace (Amann Girrbach AG; Koblach, Austria). The sintering process was carried out at 1450 °C for 2 hours with a heating rate of 8 °C/min. Upon completion, the samples were allowed to cool gradually to room temperature, following the manufacturer's instructions.

The fully sintered samples were embedded in PP tubes using acrylic resin, with one surface of each test specimen left exposed. The exposed zirconia surfaces were sequentially polished with 220, 400, and 600-grit silicon carbide sandpaper to standardize the surfaces. Between each sanding stage, the zirconia surfaces were rinsed with distilled water for 10 seconds. At the end of the polishing process, the specimens were cleaned in an ultrasonic bath with 99% isopropyl alcohol for 3 minutes.

After cleaning, the specimens were sandblasted with 50-micrometer aluminum oxide particles (Basic Classic, Renfert, Germany) for 15 seconds, maintaining a distance of 10 mm and a pressure of 0.25 MPa. The specimens were then ultrasonically cleaned again for 3 minutes in 99% isopropyl alcohol.

Subsequently, the specimens were randomly divided into four groups (www.randomizer.org) based on the chemical conditioning agent applied: SBU Group - Single Bond Universal (3M ESPE; St. Paul, MN, USA), TBU Group - Tetric N-Bond Universal (Ivoclar Vivadent; Schaan, Liechtenstein), PZM Group - Peak-ZM (Ultradent; South Jordan, UT, USA), and ZP Group - Z Prime (Bisco; Schaumburg, IL, USA). The composition of the chemical conditioning agents used in this study is summarized in Table 1. The chemical conditioning agents were applied to the polished and sandblasted surface of the zirconia specimens in strict accordance with the manufacturer's instructions.

TABLE 1

Material, manufacturer, composition, lot, and application procedure of the conditioning agents used

MATERIAL	MANUFACTURER	COMPOSITION **	LOT	APPLICATION PROCEDURE
Single Bond Universal (SBU)	3M ESPE (St. Paul, MN, USA)	10-MDP, Bis-GMA, HEMA, functional methacrylate co-polymer, filler, ethanol, water, initiators, and silane.	00221A	▪ Apply the adhesive. ▪ Leave it to react for 20 seconds. ▪ Dry with air for 5 seconds.
Tetric Bond Universal (TBU)	Ivoclar Vivadent (Schaan, Liechtens-tein)	10-MDP, Bis-GMA, HEMA, UDMA, DDMA, filler, ethanol, and camphorquinone.	Y43918	▪ Apply the adhesive. ▪ Leave it to react for 20 seconds. ▪ Dry with air for 5 seconds ▪ Light-cure for 10 seconds
Peak ZM (PZM)	Ultradent (South Jordan, UT, USA)	10-MDP, HEMA, ethyl alco-hol.	BJ4T1	▪ Apply the primer for 3 seconds ▪ Dry with air
Z Prime Plus (ZP)	Bisco (Schaumburg, IL, USA)	10-MDP, Bisphenol A Glycidyl Methacrylate, HEMA, ethanol.	17005432	▪ Apply 1-2 layers, evenly moistening the bonding surface. ▪ Dry with an air syringe for 3-5 seconds.
RelyX ARC	3M ESPE (St. Paul, MN, USA	**PASTE A:** Silica treated with silane, TEGDMA, BisGMA, function-alized dimethacrylate poly-mer, triphenylantimony. **PASTE B:** Silica treated with silane, TEGDMA, BisGMA, func-tionalized dimethacrylate polymer, BTM, BPO.	6724817	▪ Dispense the cement into a mixing block and mix for 10 seconds. ▪ Apply a layer of cement to the bonding surface. ▪ Remove excess. ▪ Light-cure for 40 seconds.

****Information provided by the manufacturer:**
10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate.
Bis-GMA: Bisphenol A-glycidyl methacrylate.
UDMA: Urethane dimethacrylate.
HEMA: 2-hydroxyethyl methacrylate.
DDMA: Decamethylene dimethacrylate.
TEGDMA: Triethylene glycol dimethacrylate.
BPO: Benzoyl peroxide.
BTM: 2-benzotriazolyl-4-methylphenol.

After the application of the chemical conditioning agents, a cylindrical silicone matrix with four holes, each with an internal diameter of 1.4 mm, was positioned on the zirconia surface. Each hole was filled with a conventional dual-cure resin bonding agent (RelyX ARC, 3M ESPE, St. Paul, MN, USA), handled according to the manufacturer's instructions, and light-cured for 20 seconds using a light-curing unit (Optilight MAX, Gnatus; Ribeirão Preto, Brazil) at an intensity of 1000 mW/cm^2, which had been previously verified with a radiometer (Bluelight Meter, Ivoclar Vivadent, Schaan, Liechtenstein). Following light-curing, the silicone matrix was removed, revealing four resin bonding agent cylinders. Figure 1 provides a graphic depiction of the methodology steps.

MICRO-SHEAR BOND STRENGTH TEST

All samples were stored in distilled water at 37°C for 24 hours. Immediately afterward, two of the four resin cylinders from each specimen were subjected to a micro-shear bond strength test. After completing the immediate test, specimens with the remaining two cylinders were then stored in distilled water at 37°C for six months. After this delay period, the remaining two cylinders from each specimen underwent the micro-shear bond strength test.

This test was performed using a mechanical universal testing machine (CMT 2000; MTS SANS, China) at a crosshead speed of 1.0 mm/min. Bond strength (in MPa) was calculated by dividing the load (in Newtons) by the bonded interface area (mm^2).

The specimens were randomly organized into groups,

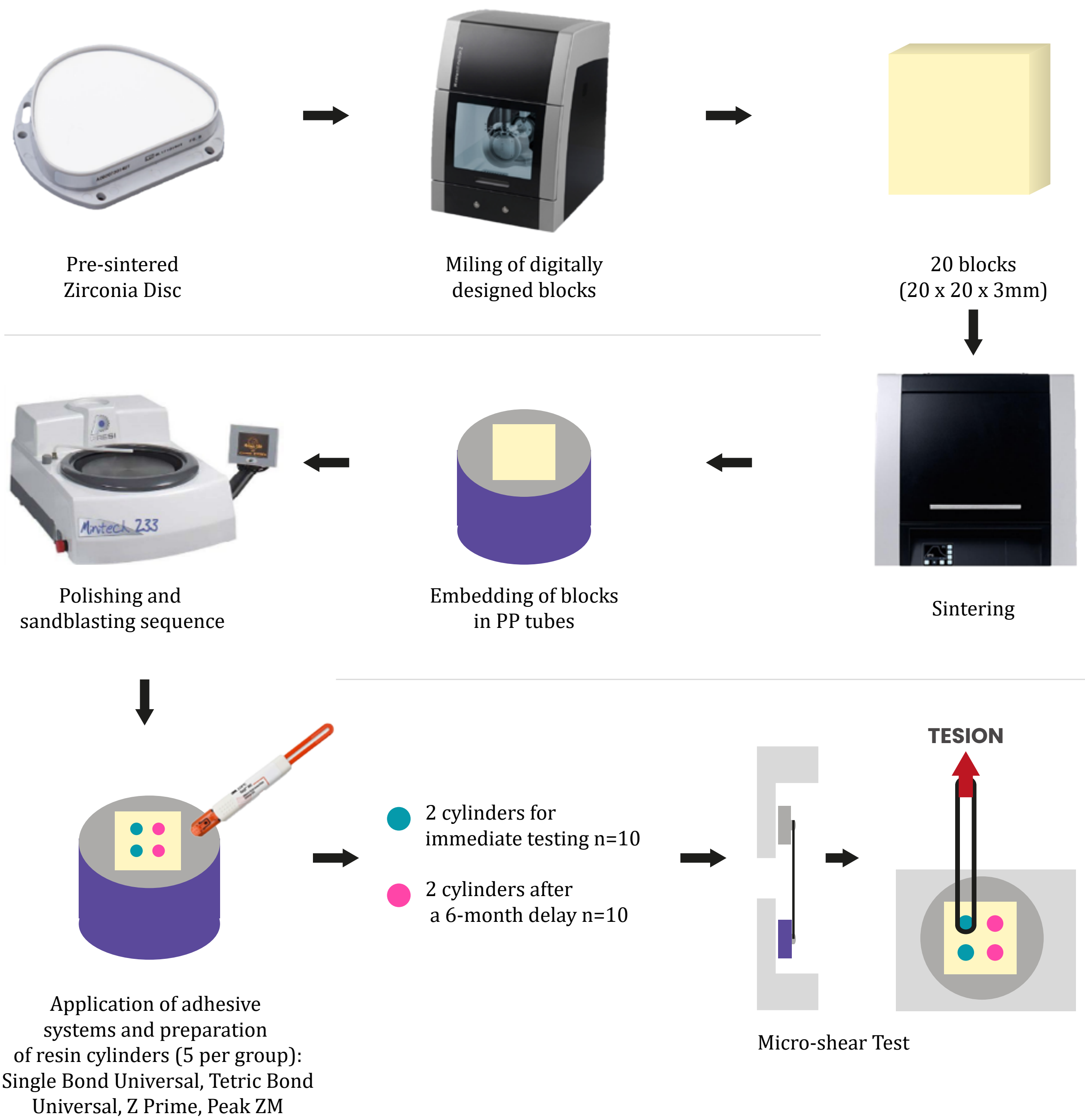

Figure 1. Methodological scheme

with each zirconia block containing two resin cylinders for immediate testing and two for testing after delay (n=10):

1. SBU-i Group: Single Bond Universal application; immediate shear test.
2. SBU-e Group: Single Bond Universal application; shear test after 6 months of delay.
3. TBU-i Group: Tetric Bond Universal application; immediate shear test.
4. TBU-e Group: Tetric Bond Universal application; shear test after 6 months of delay.
5. PZP-i Group: Peak ZM application; immediate shear test.
6. PZP-e Group: Peak ZM application; shear test after 6 months of delay.
7. ZP-i Group: Z Prime application; immediate shear test.
8. ZP-e Group: Z Prime application; shear test after 6 months of delay.

STATISTICAL ANALYSIS

Data were analyzed to ensure normality and homogeneity of variance. After verifying these assumptions, the micro-shear bond strength data were statistically assessed using a two-way ANOVA to evaluate the effect of the factors (chemical conditioning agent and delay) on the dependent variable. A post-hoc analysis was conducted using Tukey's test to compare the bond strength means between the individual groups. Statistical analyses were performed using SigmaPlot 12.0 software. A significance level of 0.05 was applied for all tests.

Results

Figure 2 shows the micro-shear bond strength results for all groups tested both immediately and after a 6 month delay.

According to the two-way ANOVA, the micro-shear bond strength was not significantly affected by the chem-

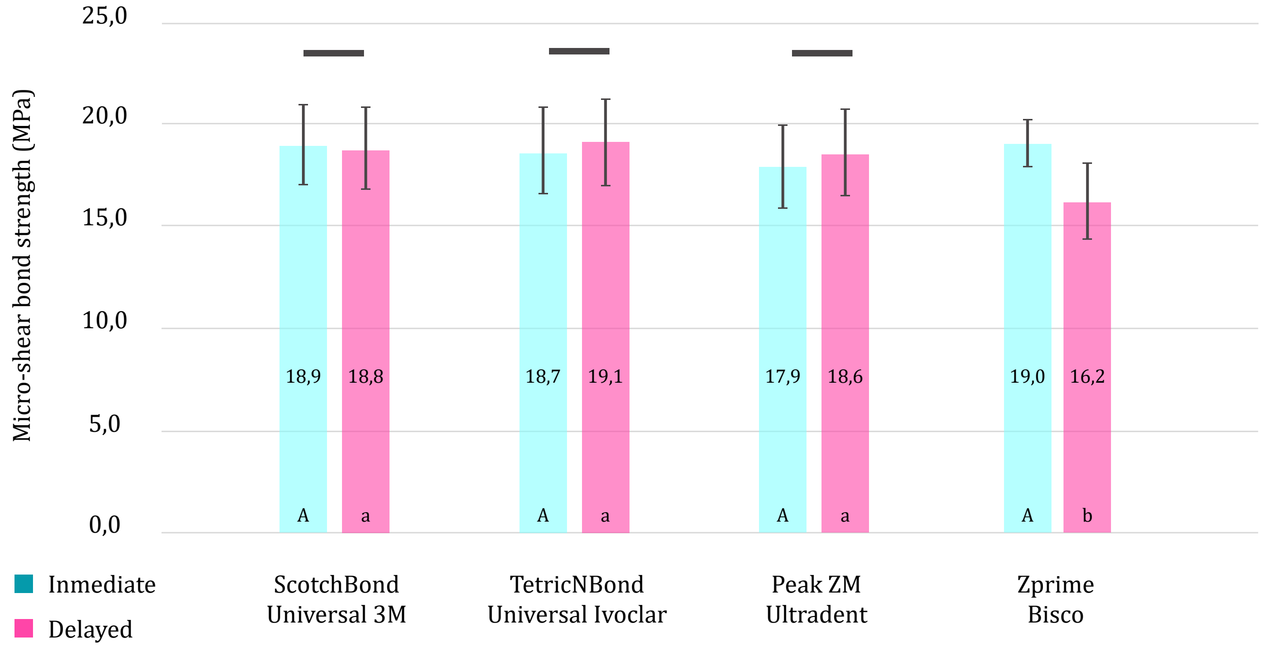

Figure 2. Micro-shear bond strength values (MPa). Different uppercase letters indicate statistically significant differences between the chemical conditioning agents evaluated at 24 hours. Different lowercase letters indicate statistically significant differences between the chemical conditioning agents evaluated after 6 months. Columns under the same horizontal line indicate no statistically significant difference between the immediate and delayed results for each chemical conditioning agent.

ical conditioning factor (p = 0.058) nor the delay factor (p = 0.216). However, the interaction between both factors was significant (p = 0.005).

When analyzing the chemical conditioning factor at 24 hours, the highest value was observed for the Z Prime group; however, there were no statistically significant differences compared to the other adhesive strategies (p ≥ 0.426). In turn, when analyzing the Group factor at 6 months of delay, the Z Prime group showed the lowest adhesion values, which were significantly lower than the rest of the groups (p ≤ 0.016). The other groups showed no significant differences among them (p ≥ 0.993).

Upon analyzing the time factor in each of the adhesive strategies used, a significant decrease was observed in the Z Prime group (p < 0.001), while the other groups did not show significant differences when comparing immediate results with those after 6 months of delay (p ≥ 0.216)

Discussion

This study aimed to evaluate the immediate and delayed micro-shear bond strength of a conventional dual-cure resin bonding agent to zirconium oxide surface using different chemical bonding promoters. The results did not show a statistically significant difference in the immediate and delayed adhesion across the different groups, with the exception of Z Prime, where a decrease in adhesive strength was observed between the immediate and delayed values. Therefore, the null hypothesis was partially accepted.

Upon evaluating the immediate bond strength values, these ranged from 17.9 MPa to 19 MPa, showing no statistically significant differences between the groups (p ≥ 0.426). These values are consistent with those reported in the literature (17,22). Furthermore, the bond strength values after the delayed period ranged from 16.2 MPa to 19.1 MPa, with no significant differences observed among the SBU, TBU, and PZM groups (p ≥ 0.993). These findings can be attributed to the similarity in the composition of the chemical bonding promoters, which are based on the phosphate monomer 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP) (18, 23–25). This molecule has the ability to form bonds between the divalent phosphoryl groups of the MDP monomer and hydroxyl groups on the zirconia surface (22,26). The literature reports various clinical protocols for treating the zirconia surface, with no clear superiority when comparing different 10-MDP-based conditioners (23,27). However, several authors conclude that mechanical treatment, combined with priming using a universal adhesive or a zirconia-specific primer, results in adequate bond strength values in in vitro studies (10,18).

It has been reported that the presence of the 10-MDP monomer is essential for providing reliable bonding, while its concentration is less important. (27) However, the results of this study showed a significant decrease in adhesion in the group where a zirconia-specific primer was used (p≤0.016). It has been demonstrated that delaying the test specimens leads to a reduction in adhesion values, (24,28) which may be attributed to the weakening of the bond due to its hydrolysis when stored in water for 6 months. (17)

In this study, a decrease in adhesive values was observed in the ZP group when subjected to a delay through water storage. Additionally, previous studies have reported inferior performance of zirconia-specific primers compared to other bonding promoters, such as universal adhesives (29,30). This could be attributed to the presence of fillers and monomers more resistant to hydrolytic degradation, such as Bis-GMA (25). Other potential contributing factors include differences in the concentrations and purity of the functional monomers used in each product (29). Furthermore, ZP contains Bis-GMA without an initiation system, which, when not polymerized, could accelerate the hydrolysis of the adhesive layer, thereby reducing bond strength values (31).

The literature establishes that values between 15 and 25 MPa in laboratory studies are considered adequate for clinical use, (32) while values below 13 MPa may be deemed critical. (19) Therefore, all the materials analyzed in this study meet these standards. However, comparing and extrapolating in vitro data to clinical performance remains challenging, as additional factors must be considered, such as cavity shape, restoration thickness, and finishing, among others, (33) as well as clinical parameters like secondary caries, material fracture, or gingival health. (34) The authors believe that potential clinical failures may result from the improper application of clinical protocols by professionals. These include overlooking contamination of the zirconia surface with saliva after try-in, (35,36) incorrect application of bonding agents (such as insufficient rubbing, evaporation, or light curing), (37) or inadequate sandblasting of the restoration's internal surface. (27)

The results of this study should be interpreted with caution due to its in vitro nature, as a clinical scenario cannot be fully replicated. The literature also highlights significant variability in in vitro methodologies, making data comparison challenging. (23) Furthermore, a single bonding agent was used, and only four bonding promoters were analyzed.

Conclusions

Despite the limitations of this study, the results suggest that universal adhesives containing 10-MDP exhibit immediate bond strength comparable to zirconia-specific chemical surface conditioners. After a period of delay, universal adhesives appear to offer greater bond strength stability.

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Data availability

The entire dataset supporting the results of this study has been published in the article itself.

Conflict of interest declaration

The authors of this work declare no potential conflict of interest.

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Authorship Contribution and Collaboration Statement

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1. Administración del proyecto
2. Adquisición de fondos
3. Análisis formal
4. Conceptualización
5. Curaduría de datos
6. Escritura - revisión y edición
7. Investigación
8. Metodología
9. Recursos
10. Redacción - borrador original
11. Software
12. Supervisión
13. Validación
14. Visualización

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