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Evaluation of Different Priming Agents with Conventional and Bioactive Self-Adhesive Resin Cements on Shear Bond Strength to Zirconia

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Abstract: The aim of this study was to evaluate the effect of different priming agents on the shear bond strength (SBS) of conventional and bioactive self-adhesive resin cements to zirconia. One hundred and twenty zirconia discs were randomly divided into four main groups according to the priming agents used ($n = 30$): no priming agent (control), zirconia primer (Z-PRIME Plus), universal adhesive (All-Bond Universal), and universal ceramic primer (Monobond N). Then, each group was subdivided into three subgroups according to the type of self-adhesive resin cement used: TheraCem, Activa BioActive, and RelyX U200 Automix ($n = 10$). All specimens were subjected to thermocycling. The mean SBS data were analyzed using One-Way ANOVA, followed by multiple comparison Bonferroni test. Without the application of priming agents (control), most of the specimens failed during thermocycling. The priming agent, cement type, and their interaction had a significant effect on the SBS to zirconia ($p < 0.001$). Only the type of priming agent showed a significant effect on the mode of failure ($p < 0.001$), resulting in mainly mixed failure with Monobond N and adhesive failure with other primers. Regardless of the type of primer, Bioactive resin cements did not improve the SBS to zirconia compared to conventional cements.

Keywords: dentistry; prosthodontics; zirconia; bonding; bioactive cement; shear bond strength; functional monomer; bonding; ceramic; durability; adhesion; resin cement

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1. Introduction

Zirconia restorations have become increasingly popular amongst clinicians due to their excellent mechanical properties and acceptable esthetics, with evidence of long-term clinical success [1,2]. A successful fixed dental prosthesis requires a durable bond to the teeth, especially with restorations that lack retention form [3]. Silica-based ceramic materials have established bonding protocols that involve the etching of the glassy phase of the matrix [4]. However, it is difficult to etch zirconia owing to its highly crystalline structure [5]. Therefore, different methods are used to create micromechanical retention needed for better bonding [6].

Mechano-chemical surface treatment consisting of air abrasion and phosphoric monomers is the most acceptable technique for bonding to zirconia [7,8]. Air abrasion has been the superior mechanical surface treatment technique since it increases surface roughness and energy [7]. Chemical bonding to zirconia was possible with the use of primers containing a phosphate monomer, specifically in the form of 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). This monomer has a phosphate group and methacrylate group, which bond to the zirconia and resin cement, respectively. The chemical bond of the 10-MDP primer to the zirconia surface is formed through ionic and hydrogen bonds [9]. Several reports have shown that higher bond strength was achieved with MDP-primed zirconia when compared to other priming agents or non-treated zirconia [10–14]. To simplify the bonding process, some bonding agents were modified with the addition of 10-MDP to their composition in a single bottle to promote bonding to zirconia and different indirect restorations [15–20]. In a systematic review, 169 surface treatment methods performed on zirconia were investigated [21]. The authors found that 10-MDP-based resin cement provided higher bond strength to zirconia, and they concluded that the surface treatment, cement type, testing methods, and aging condition influenced the bond strength.

The interface between the tooth structure and restoration is a crucial factor for the longevity of the restoration [22,23]. One of the most common reasons for indirect restoration is secondary caries [24]. Although available self-adhesive resin cements have shown good mechanical, esthetic, and bond strength properties [5,25], a new generation of cements given the description "bioactive" were introduced. The main objective of bioactive resin cement is to minimize the chance of caries with different mechanisms and chemical compositions [26]. TheraCem (BISCO Inc., Schaumburg, IL, USA) is promoted as a calcium-fluoride-releasing self-adhesive bioactive resin cement that contains 10-MDP monomer which may allow bonding to zirconia and metal substrates without the use of an additional primer [27–30]. Activa BioActive (Pulpdent, Watertown, MA, USA) cement is promoted as a hybrid self-adhesive resin cement, chemically bonds to the tooth structure, provides a tight marginal seal against bacteria, reduces secondary caries, and aids in the remineralization of tooth structure [31–33]. It showed significantly higher flexural strength and calcium ion release compared to resin-modified glass ionomer [34]. Also, the manufacturer of this bioactive cement claims that this cement can bond to zirconia without the application of a ceramic primer [35].

With the frequent introduction of self-adhesive resin cement and priming agents, it is difficult for clinicians to choose the best combination to achieve a good bond strength. In addition, the recently introduced bioactive cements remain not fully investigated. Thus, the aim of the present study was to evaluate the effect of different priming agents (Zirconia primer, universal adhesive, or universal ceramic primer) on the shear bond strength of conventional and bioactive self-adhesive resin cements to zirconia. The null hypothesis was that different priming agents, resin cements or their combinations have no significant difference in the shear bond strength to zirconia.

2. Materials and Methods

2.1. Experimental Specimens

One hundred and twenty zirconia specimens (10 × 7 × 2 mm) were sectioned from 12 blocks of IPS e. max ZirCAD (Ivoclar Vivadent; Schaan, Liechtenstein) using a low-speed diamond blade (Allied High Tech, Compton, CA, USA) [2]. To achieve a flat standardized surface, the cementation surfaces were polished using 220, 320, 600, and 1200-grit silicon carbide abrasive papers (MetaServ 250, Buehler; Lake Bluff, IL, USA) [36]. All specimens underwent sintering firing according to the manufacturer's instructions. Specimens were then embedded in self-cured acrylic resin (15 mm in diameter and 10 mm in height) with the cementation surface exposed. Specimens were then subjected to air-particle abrasion (Duostar Z2, BEGO; Bremen, Germany) with 50-µm Al_2O_3 particles (Korox, BEGO; Bremen, Germany), at a pressure of 2 bars and distance of 10 mm, for 15 s [1].

All specimens were ultrasonically cleaned (PowerSonic 405, Hwashin; Seoul, Republic of Korea) for 5 min in distilled water of 37 °C and air dried. Specimens were randomly divided according to the priming agent used (n = 30): no conditioning agent (control), zirconia primer Z-PRIME Plus (ZP), universal adhesive All-Bond Universal (AB), and universal ceramic primer Monobond N (MN). Then, each group was subdivided into three subgroups according to the type of resin cement used: TheraCem (T), Activa BioActive (A), and RelyX U200 (Rx) Automix (n = 10/sub-group), resulting in 12 different combination groups (Figure 1).

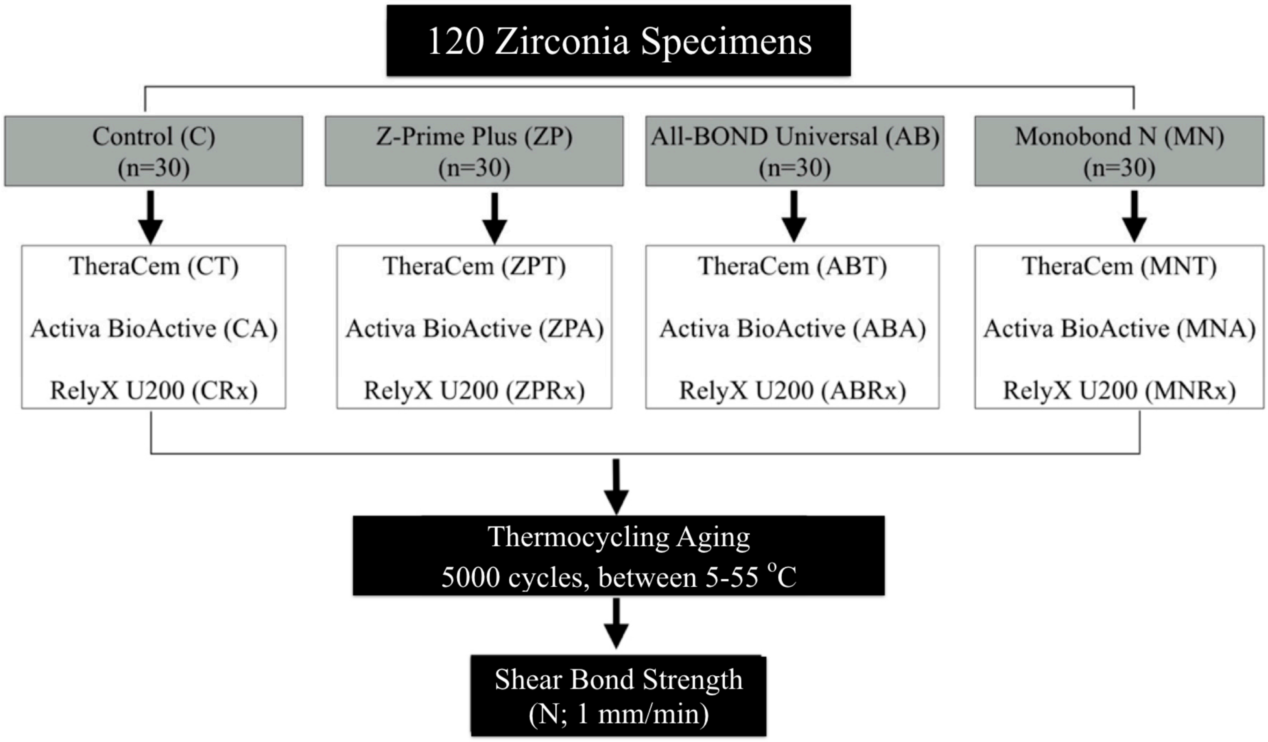

Figure 1. Flow chart of the experiment and groups distribution.

Table 1 summarizes the compositions and application of materials used in this study. A split Teflon mold (4.25 mm diameter and 4 mm height) was used to bond cement cylinders to the treated zirconia surface and light cured for 40 s using an E-Morlit curing light (Apoza, NewTaipei, Taiwan) delivering a power of 1200 mW/cm^2 [2]. All specimens underwent an artificial aging process (thermocycling of 5000 cycles), between 5–55 °C (SD Mechatronik Thermocycler, JULABO GmbH; Seelbach, Germany) and each cycle takes 1 min to be completed [4]. Any specimen that failed during or after the thermocycling process, or before the shear bond testing was considered a pre-test failure, recorded as zero, and was not included in the statistical analyses [21].

Table 1. Compositions and manufacturers of the materials used in the present study.

Materials	Manufacturer	Composition
IPS e.max ZirCAD MO 0	Ivoclar Vivadent [a]	ZrO_2, Y_2O_3, HfO_2, Al_2O_3, and other oxides.
Korox 50	BEGO [b]	Al_2O_3 (50 µm).
Z-PRIME Plus (ZP)	BISCO [c]	10-MDP, carboxylic acid monomer, BPDM, and ethanol.
All-Bond Universal (AB)	BISCO [c]	10-MDP, BPDM, Bis-GMA, HEMA, water, ethanol, and photoinitiator.
Monobond N (MN)	Ivoclar Vivadent [a]	Alcohol solution of silane methacrylate, phoshphoric acid methacrylate, and sulphide methacrylate.
TheraCem (T)	BISCO [c]	Base: Calcium base filler, glass filler, dimethacrylate, ytterbium fluoride, initiator, and amorphous silica. Catalyst: Glass filler, MDP, and amorphous silica.

Table 1. *Cont.*

Materials	Manufacturer	Composition
ACTIVA BioACTIVE (A)	Pulpdent [d]	Base: Diurethane dimethacrylate and other methacrylate-based monomers and oligomers, polyacrylic acid/maleic acid copolymer, water, barium borosilicate glass, silica, reducing agents, photoinitiators, and colorants. Catalyst: Diurethane dimethacrylate and other methacrylate-based monomers and oligomers, aluminoflurosilicate ionomer glass, silica, and oxidizing agents.
RelyX U200 Automix (Rx)	3M ESPE [e]	Base: Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizer, and Rheological additives. Catalyst: Methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizer, pigments, and rheological additives.

[a] Schaan, Liechtenstein; [b] Bremen, Germany; [c] Schaumburg, USA; [d] Watertown, USA; [e] Seefeld, Germany. 10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate; BPDM: Biphenyl dimethacrylate. Bis-GMA: bisphenol A-glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate.

2.2. Shear Bond Strength Test

The acrylic molds were then fixed and secured in position into a universal testing machine (INSTRON; Norwood, MA, USA). Compressive load was applied on the zirconia-cement cylinders interface by a knife edge chisel with a cross head speed of 1 mm/min until failure occurred. The Instron machine was connected to a computer with a specifically designed program (BlueHill 3 software, Version 3.24.1496, Instron Worldwide Headquarters, Norwood, MA, USA). This software controlled the testing machine and recorded the applied load. The shear bond strength was calculated by dividing the applied load (N) at the time of fracture by the resin-zirconia interface area (mm^2) ($MPa = N/mm^2$).

2.3. Failure Evaluation

The fracture interfaces of all zirconia specimens were inspected under a light stereomicroscope (MX 7520, Meiji Techno, Hicksville, NY, USA) at 20× magnification and the failure modes were recorded. One of two modes of failure was recorded: adhesive (completely exposed zirconia surface) and mixed failure (exposed zirconia surface with remnants of cement).

2.4. Statistical Analysis

Statistical analysis was performed utilizing SPSS software (Version 20 IBM Corp, Armonk, NY, USA). The Shapiro–Wilk test of normality was used to test the normality hypothesis of all quantitative variables. Most of the variables were found normally distributed allowing the use of parametric tests. One-Way ANOVA was used for comparing the SBS means of all groups and Bonferroni method was used for pairwise comparison. A General Linear Model (GLM) was applied to study the effect of the independent factors (priming agent and cement type) and their interaction. Chi-squared test was used for fracture mode analysis. Significance level of ($\alpha = 0.05$) and two-tailed tests were assumed throughout the analysis for all statistical tests.

3. Results

One control group, three priming agents (Z-PRIME Plus, ALL BOND UNIVERSAL and Monobond N) and three self-adhesive resin cements (TheraCem, Activa BioActive, and RelyX U200) were tested in this study to evaluate the shear bond strength to zirconia. Most of the control specimens failed during thermocycling, and those specimens which survived (6/30 specimens) were the weakest among all tested groups. Due to the limited number of control specimens, they were excluded from the statistical analysis. The descriptive analysis is presented in Table 2.

Table 2. Descriptive analysis of bond strength and failure modes.

Group	Mean (MPa) ± SD	Coefficient of Variation (CV)	Standard Error of Mean (SEM)	95% Confidence Interval for Mean		Failure Mode (N)	
				Lower Bound	Upper Bound	Adhesive Failure	Mixed Failure
ZPT	5.39 ± 2.60 ^{C,D}	48.24%	0.92	3.21	7.56	6	4
ZPA	4.89 ± 1.29 ^{D}	26.30%	0.41	3.97	5.81	8	2
ZPRx	7.07 ± 1.53 ^{C,D}	21.61%	0.51	5.89	8.24	5	5
ABT	5.60 ± 2.45 ^{C,D}	43.82%	0.87	3.55	7.65	9	1
ABA	6.19 ± 2.63 ^{C,D}	42.46%	0.83	4.31	8.07	7	3
ABRx	13.41 ± 2.27 ^{A,B}	16.90%	0.72	11.79	15.03	9	1
MNT	10.30 ± 3.14 ^{B,C}	30.52%	1.05	7.88	12.71	2	8
MNA	13.04 ± 4.34 ^{A,B}	33.25%	1.45	9.71	16.37	0	10
MNRx	16.46 ± 5.97 ^{A}	36.26%	1.99	11.88	21.05	0	10

Similar superscript letters indicate no statistically significant difference ($p < 0.05$).

One-Way ANOVA showed statistically significant differences between the test groups ($p < 0.0001$) (Table 3). Bonferroni Pairwise comparisons showed that MNRx had the highest shear bond strength to zirconia (16.46 ± 5.97 MPa), followed by ABRx (13.42 ± 2.28 MPa) and MNA (13.03 ± 4.32) which were significantly higher than other combinations. General Linear Model (GLM) revealed that the priming agent ($p < 0.001$), cement type ($p < 0.001$), and their interaction ($p = 0.027$) had a significant effect on shear bond strength to zirconia. Table 4 and Figure 2.

Table 3. One-way ANOVA result of the shear bond strength.

		df	Mean Square	F	*p* Value
Between Groups	1332.86	8	166.61	16.30	0.00000 *
Within Groups	745.97	73	10.22		
Total	2078.83	81			

df, degree of freedom (n − 1); * Significant at $p < 0.05$.

Table 4. General Linear Model: Univarinate Analysis of Variance.

Source	Type III Sum of Square	df	Mean Square	F	*p* *
Corrected Model	1332.86	8	166.61	16.30	<0.001 *
Intercept	6818.81	1	6818.81	667.28	<0.001 *
Priming agent	777.11	2	388.56	38.02	<0.001 *
Cement type	420.03	2	210.02	20.55	<0.001 *
Interaction	119.14	4	29.78	2.91	0.027 *
Error	745.97	73	10.22		
Total	9023.36	82			
Corrected Total	2078.83	81			

df, degree of freedom (n − 1); * Significant at $p < 0.05$.

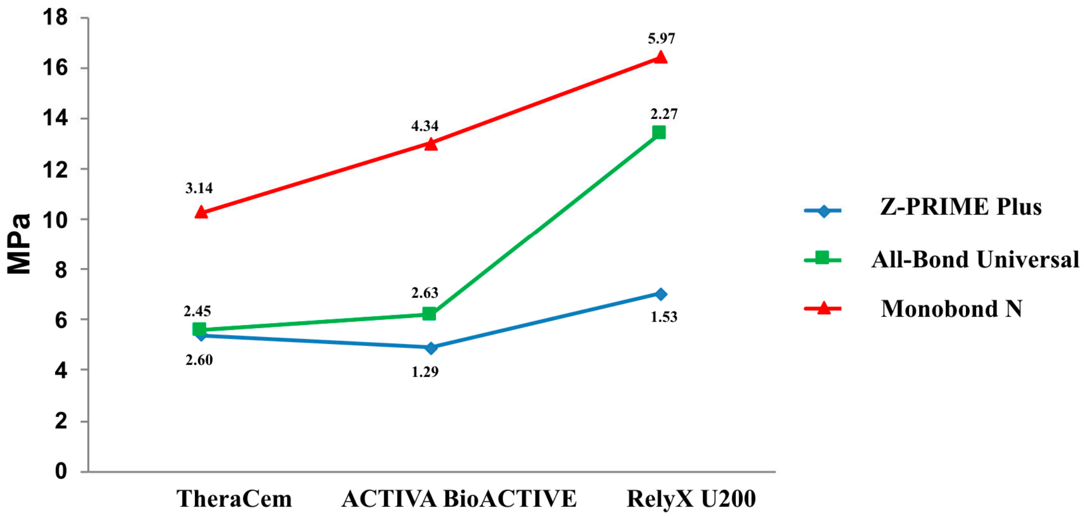

Figure 2. Mean Values of Shear Bond Strength with standard deviations illustrating the interaction between the two factors (Priming agent and cement).

Estimated marginal means of the shear bond strength data by independent variable "priming agent" are summarized in Table 5. Pairwise comparisons showed that there was a significant difference between all the groups ($p < 0.001$). Estimated marginal means of the SBS data by the independent variable "Cement type" are summarized in Table 6. Pairwise comparisons showed that there was a significant difference between the groups ($p < 0.001$)

Table 5. Estimated shear bond strength mean by independent variable "Priming Agent".

Priming Agent.	Mean (MPa) ± SD	95% Confidence Interval	
		Lower Bound	Upper Bound
MN	13.27 ± 5.14 ^{A}	12.04	14.49
AB	8.40 ± 4.36 ^{B}	7.19	9.61
ZP	5.78 ± 2.01 ^{C}	4.55	7.01

Similar superscript letters indicate no statistically significant difference ($p < 0.05$).

Table 6. Estimated shear bond strength means by independent variable "Cement Type".

Cement Type	Mean (Mpa) ± SD	95% Confidence Interval	
		Lower Bound	Upper Bound
T	7.10 ± 3.54 ^{B}	5.82	8.37
A	8.04 ± 4.50 ^{B}	6.86	9.22
Rx	12.3 ± 5.32 ^{A}	11.11	13.52

Similar superscript letters indicate no statistically significant difference ($p < 0.05$).

For the analysis of the distribution of failure modes, the Chi-square test showed that the priming agent had a significant effect on the fracture mode ($p < 0.001$) (Table 7). Monobond N had the largest mixed failure mode (93.3%). Regarding other conditioning agents, mostly adhesive failures were noted at 63.3% for ZP and 83.3% for AB. On the other hand, the cement type had no significant effect on the failure mode ($p = 0.733$) (Table 8) (Figure 3).

Table 7. Chi-square test, effect of priming agent on the fracture mode.

Priming Agent	Fracture Mode				Chi-Squared	p Value
	Adhesive		Mixed			
ZP	19	63.33%	11	36.67%	37.97	<0.001 *
AB	25	83.33%	5	16.67%		
MN	2	6.67%	28	93.33%		

* Significant at $p < 0.05$.

Table 8. Chi-square test, effect of cement type on the fracture mode.

Cement Type	Fracture Mode				Chi-Squared	p Value
	Adhesive		Mixed			
T	17	56.67%	13	43.33%	0.62	0.732520
A	15	50.00%	15	50.00%		
Rx	14	46.67%	16	53.33%		

* Significant at $p < 0.05$.

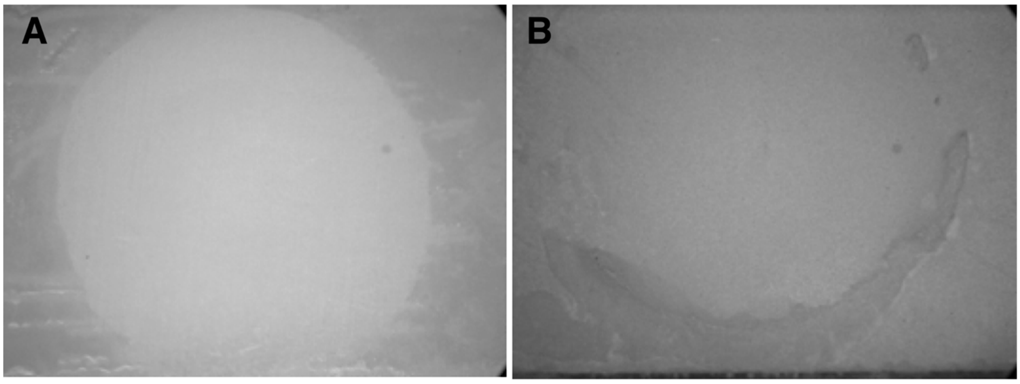

Figure 3. Failure mode inspected under a light stereomicroscope at 20× magnification. (**A**) Classic presentation of adhesive failure specimen of ABT. (**B**) Mixed failure specimen of MNRx.

4. Discussion

We aimed to evaluate the shear bond strength of three different self-adhesive resin cements to zirconia using different priming agents. The results showed that the priming agent, cement type, and their interaction had a significant effect on shear bond strength to zirconia. Therefore, the null hypothesis that different priming agents have no significant effect on the shear bond strength of self-adhesive resin cements to zirconia was rejected.

In the control group (no priming agent), most of the specimens (24/30) failed during the artificial aging, and the surviving specimens (6/30) showed the lowest bond strength among all groups. Our findings supported the concept that air-abrasion (mechanical adhesion) without a priming agent (chemical adhesion) is not enough to achieve adequate bond to zirconia [12]. Therefore, the claim that these cements are able to bond to zirconia without priming was also rejected.

To achieve an adequate bond strength to zirconia, a phosphate functional monomer is essential [3,11], which is present in all priming agents used. However, it appears that they are not equally effective. Regardless of the type of self-adhesive resin cement used, the universal ceramic primer MN achieved significantly higher bond strength values (10.30–16.46 MPa) compared to AB (5.6–13.41 MPa) and ZP (5.39–7.07 MPa). One possible explanation is that different functional phosphoric acid and methacrylate groups have different resistance to hydrolysis, which resulted in variable bond strength to zirconia [13]. Another reason could be attributed to the presence of silane in the composition (as the case for MN), which improves the wettability of zirconia and bonding to the resin cement [14]. These properties of MN agent were reflected in the failure mode where most of the samples showed mixed failures.

The findings of this study regarding (ZP) were contrary to the expectation. Although zirconia primer ZP has 10-MDP, the bond strength of zirconia primer ZP showed the lowest values with all cements. It has been reported that the carboxylic acid group could destabilize the bond between 10-MDP and the methacrylate monomers of the resin cement leading to a weak bond [14]. Similar results were reported by Amaral et al. [15] who found that AB had higher bond strength to zirconia than ZP using multilink universal adhesive resin cement. Also, the differences in 10-MDP concentrations and primer initiation systems might influence the quality and durability of the bond. The failure mode of both AB and ZP were primarily adhesive which coincides with low bond strength adhesion.

Recently, multipurpose (Universal) adhesives were introduced to enhance bonding to zirconia, glass ceramic, and metals [17,18]. Tayal et al. [19] tested bonding to zirconia using two different universal adhesive systems. The findings indicated increased bond strength in comparison to control groups (no priming agent). Amaral et al. [15] reported that AB had better performance than ZP and the failure mode was predominantly adhesive failures. In the presented study, it was only successful to achieve better bond strength to zirconia when combined with conventional self-adhesive resin cement. However, failure mode was predominantly adhesive failures. In composition of this bonding agent (AB), presence of hydroxyethyl methacrylate (HEMA) which has high water sorption might increase the bonding degradation [20].

The current results showed that the bond strength of the conventional cement Relyx was superior to the bioactive cements. In disagreement, Mahrous et al. [28] reported that TheraCem showed higher bond strength to enamel, dentin, and zirconia than conventional self-adhesive resin cement. Chen et al. [29] found that TheraCem had a higher bond strength to zirconia than a resin-modified glass-ionomer cement and conventional self-adhesive resin cement. In addition, Akay et al. [5] showed that the bond strength of self-adhesive resin cement containing MDP had higher values than conventional self-adhesive resin cement. These contradictory results may be attributed to the different materials used in these studies, which may have affected the quality of the covalent bond between zirconia and the monomer of the different self-adhesive resin cement [37]. In addition, some studies have not subjected the samples to thermocycling. The degree of polymerization of the resin cements and their composition directly influence the ability of moisture tolerance after thermocycling [38].

The control group showed adhesive failures. Hence, the application of the ceramic primer or bonding agent containing functional monomer in the composition is recommended to achieve a high bond strength [9]. AB and ZP groups showed both mixed and adhesive failures. However, the performance of MN was reflected in the failure mode. Most of the failures were mixed failures except two specimens with showed adhesive failures [14].

However, the difference between the studies might be related to multiple factors. First, the exact composition and percentage of each material are not declared completely by the provider which might influence the bond strength between different MDP containing cement, ceramic primer, or universal adhesive agent. Second, the use of artificial aging techniques, such as thermocycling, can replicate the oral environment and stress the resin-zirconia contact; however, few studies employ this step.

This study included multiple Limitations. First, using shear bond strength instead of microtensile bonds strength which allow to perform uniform stress distribution on the bonding surface. However, shear bond strength is most common to assess ceramic bonding, easier to perform, and provide the researcher with a group ranking [2]. Second, thermocycling is used for aging, but other factors such as mastication load and fluctuation of the pH level might considerably increase the degradation of the bond. Therefore, the authors recommend further studies to evaluate other important mechanical and physical properties in order to complete the knowledge about various zirconia-based materials [39–42].

5. Conclusions

Based on this in vitro study, the following conclusion were drawn:

1. Self-adhesive bioactive resin cements did not provide sufficient bond strength to zirconia without the application of priming agents.
2. Both the priming agent and the cement type have a significant effect on the shear bond strength to zirconia.
3. The application of the ceramic primer or bonding agent containing phosphate functional monomer is recommended to achieve a high bond strength.
4. Regardless of the cement type, universal ceramic primer MN provided the highest shear bond strength to zirconia. Moreover, regardless of the priming agent used, conventional self-adhesive resin cement Rx provided higher bond strength to zirconia compared to bioactive cements.
5. Not all phosphate functional monomers containing primers are effective in providing a reliable bond to zirconia. It is the clinician responsibility to evaluate and select the best cement–primer combination.

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