Original paper

Influence of surface treatment and simulated aging on the shear bond strength of different luting agents to zirconia

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Abstract

Background. Zirconia, or stabilized zirconium oxide (ZrO_2), is a widely used dental ceramic for full-coverage crowns owing to its favorable biological and esthetic properties. Upon cooling after high-temperature sintering, zirconia undergoes a tetragonal-to-monoclinic phase transformation, which causes volume expansion and brittleness. Adhesive cementation (AC) is crucial for the clinical performance of zirconia restorations, enhancing their retention, marginal adaptation and fracture resistance, thereby contributing to their suitability for dental restorations.

Objectives. The aim of the present study was to evaluate how different kinds of surface treatment affect the shear bond strength (SBS) of zirconia, to analyze the influence of various luting agents on the SBS of zirconia, and to investigate the effect of simulated aging through water storage on the SBS of zirconia.

Material and methods. An experimental study design was used to assess the impact of 2 luting agents (RelyX™ Unicem and Panavia™ F 2.0), different surface treatment methods (STMs) and variable water-storage durations (1day (1D), 1 month (1M) and 6 months (6M)) on the SBS of zirconia.

Results. The study revealed that RelyX consistently showed lower mean SBS values than Panavia across the different water-storage durations. For instance, in group 1 at 1D, the mean SBS for RelyX was 17.12 ±0.71 MPa as compared to 21.76 ±2.81 MPa for Panavia. ANOVA indicated significant differences in SBS between RelyX and Panavia in each group (group 1: $F = 13.892$; $p < 0.000$). Post-hoc tests confirmed significant differences in the mean SBS across the different aging durations. Overall, the findings provide comprehensive statistical evidence regarding the performance of the 2 dental luting agents under different surface treatment and aging conditions.

Conclusions. RelyX consistently demonstrated lower mean SBS values than Panavia across the different water-storage durations, indicating differences in their bonding performance. Statistical analyses confirmed significant differences in SBS between the 2 luting agents.

Keywords: dental materials, zirconia, surface treatment, shear bond strength, luting agents

Highlights

- Surface treatment significantly affected shear bond strength (SBS), as acid etching, particularly with silane, generally produced stronger and more durable zirconia–resin bonds than air abrasion alone.
- Cement selection influenced bonding performance, with Panavia™ F 2.0 generally showing higher initial SBS than RelyX™ Unicem, although performance varied with surface treatment and aging.
- Aging significantly reduced SBS across most surface treatment groups and both cements, indicating decreased bond durability over time.
- Group 6 produced the most stable bonding, as the combination of silane, air abrasion and HF treatment resulted in the highest and most stable SBS throughout storage.
- Combined surface treatment modalities improved bond durability, suggesting that integrating chemical and micromechanical treatment is more effective than using either approach alone, although further clinical validation beyond this in vitro model is needed.

Introduction

Zirconia, or stabilized zirconium oxide (ZrO_2), is a high-strength dental ceramic widely recognized for its excellent fracture toughness and biocompatibility. Its tetragonal phase, stabilized by a small addition of yttria, enhances its mechanical properties and dimensional stability, making it an ideal material for dental prosthetics.[1–3]

Although zirconia inherently possesses high fracture toughness, the manner in which the tooth is prepared remains crucial in influencing stress distribution within the crown–tooth complex. A well-performed tooth preparation reduces the risk of fracture and enhances the mechanical retention of the zirconia crown, particularly in posterior regions, where greater occlusal forces are exerted.[4] Until recently, zirconia ceramic crowns were fabricated similarly to metal-ceramic crowns, with an external layer of sintered porcelain and an internal zirconia core. This combination was intended to provide the crown with improved esthetic appearance as well as enhanced mechanical strength.[5–7]

Fractures of veneering porcelain pose significant challenges in fixed dental prostheses, primarily due to compatibility issues between the zirconia framework and the veneering material. In recent years, monolithic zirconia has emerged as an alternative to veneered zirconia restorations. This innovative approach involves the fabrication of full-contour zirconia restorations using a dental computer-aided design/computer-aided manufacturing (CAD/CAM) system, thereby eliminating the complications associated with the bonding of zirconia and veneering porcelain.

Despite this progress, it is important to recognize that monolithic zirconia may not provide the same esthetic outcomes as zirconia cores veneered with porcelain. This difference may be attributed to the relatively uniform color and limited optical properties of monolithic zirconia crowns, which can result in a less natural and esthetically pleasing appearance.[8]

The prosthetic fabrication of zirconia has evolved from traditional layering techniques to advanced CAD/CAM processes. These methods not only ensure precise anatomical conformity and improved esthetic outcomes, but also significantly reduce the potential for manual errors.[9] The present study specifically focuses on CAD/CAM-fabricated restorations, given their increasing prevalence in clinical applications and their relevance to the evaluation of surface treatment and bonding efficiency.

Cementation is crucial to the clinical effectiveness of ceramic restorations. Zirconia ceramics can benefit from adhesive cementation (AC), which may improve retention, marginal adaptation and fracture resistance. The use of zirconia and resin cements may also permit more conservative tooth preparation while reducing the risk of restoration failure. Various surface treatment and bonding strategies have been proposed to achieve a strong and durable bond between zirconia and resin cements. These include incorporating phosphate-modified monomers, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP), into resin cement; air abrasion, performed either in the laboratory or chairside, using 30- or 110-micrometer silica-coated aluminum oxide particles; the use of zirconate-coupling primers, tetraethoxysilane flame-treatment devices, organofunctional silanes, laser irradiation, silicon vapor-phase deposition, and selective infiltration etching.[7,10,11]

Despite the availability of various surface treatment methods (STMs) aimed at enhancing the strength of bond to ZrO_2, conflicting results persist, primarily due to variations in bond-strength testing methodologies.

Due to its chemical inertness and lack of a silica-rich glassy phase, establishing an effective bond between zirconia and resin cements remains challenging, rendering conventional acid-etching methods less effective. Achieving a strong and durable resin–zirconia bond generally requires a comprehensive approach combining chemical and micromechanical surface pretreatment.[12] Acid etching can increase the wettability and surface area of silica-containing ceramics by altering their surface energy and bonding potential. However, unlike silica-based ceramics, zirconia has a highly crystalline, silica-free microstructure that is resistant to conventional acid etching. Consequently, acid etching produces limited topographical changes on the zirconia surface, and is generally insufficient to achieve a strong and durable bond with a resin cement.[13]

Alternatively, acid-resistant ceramics can be air-abraded with aluminum oxide particles to produce surface irregularities and enhance micromechanical retention. However, research suggests that although air-abraded zirconia ceramics may initially improve bonding with certain luting agents, this bond may not remain stable during prolonged storage in artificial saliva or thermocycling in water. This instability may be attributed to limited chemical interaction between the treated zirconia surface and certain luting agents, as well as to the degradation of the resin–zirconia interface under aging conditions. In summary, acid etching may not produce substantial topographical alterations in highly crystalline ceramics, while air abrasion can initially enhance bonding through micromechanical retention, but may not provide sufficient long-term bond durability when used alone.[14]

To improve resin bonding to metal and highly crystalline ceramics, various silica-coating systems were initially introduced as experimental surface treatment techniques. Notable systems, such as Rocatec, CoJet and Silicoater MD, have demonstrated adequate bond strength in various studies. Monolithic zirconia crowns can benefit from resin-based cements, particularly when enhanced retention and bonding are required; however, achieving a durable bond remains challenging because of the inert, silica-free nature of zirconia. Air abrasion is commonly recommended to enhance micromechanical retention, while resin-based cements can provide additional chemical interaction when functional monomers are incorporated. Despite the high strength and fracture toughness of zirconia, the properties and durability of the resin cement are also important for maintaining adequate support and long-term stability of the ceramic restoration.[15]

The study specifically employed RelyX™ Unicem and Panavia™ F 2.0, selected for their distinct adhesive formulations and widespread clinical use. RelyX Unicem, a self-adhesive resin cement, is characterized by the ease of use and favorable initial bond strength, making it suitable for a broad range of clinical indications. Panavia F 2.0, a resin cement used in conjunction with an adhesive primer, was selected for its high bond durability and effectiveness under moist conditions. These properties make both cements suitable for evaluating the effects of different STMs and aging conditions, providing insights into their performance under varied clinical scenarios.[16]

The present study aimed to assess the effects of 2 luting agents, different STMs and variable water-storage durations on the shear bond strength (SBS) of zirconia.

Surface preparation techniques, such as air abrasion, selective etching, and the application of functional primers, can play an important role in enhancing the bond strength between zirconia and resin cements. These treatment modalities can improve micromechanical interlocking and promote chemical interaction at the zirconia–resin cement interface, thereby contributing to bond durability. The present study explores different surface treatment protocols to determine their effects on the SBS of various luting agents to zirconia, an aspect that is critical to the long-term clinical success of ceramic restorations.

Hypotheses

The following hypotheses were tested:
- null hypothesis 1: Different STMs do not significantly affect the SBS of zirconia;
- null hypothesis 2: Different types of resin cement do not significantly affect the SBS of zirconia;
- null hypothesis 3: Artificial aging through water storage does not significantly affect the SBS of zirconia.

Material and methods

Methodology

Each zirconia block was sectioned into specimens measuring 10 mm × 4 mm × 2 mm, using an EM-DC2 die-cutting machine (Aixin Medical Equipment, Tianjin, China). The specimens were then sintered and polished. This uniform rectangular shape was selected to standardize the testing area and ensure the consistent application of the surface treatment and bonding procedures across all specimens. The specimens were randomly divided into 6 groups, each of which received specific surface treatment before bonding with the luting cement (Fig. 1).

Surface treatment groups

The zirconia specimens were divided into 6 distinct groups, each subjected to a different surface treatment protocol to evaluate its effect on bond strength:
- group 1: Air abrasion using 50-micrometer alumina (Al_2O_3) particles at a pressure of 3.5 bar, a working distance of 10 mm, and a blasting time of 15 s;
- group 2: Air abrasion using 100-micrometer Al_2O_3 particles under the same conditions as group 1;
- group 3: Immersion in 10% hydrofluoric acid (HF) at 60°C for 60 min, followed by rinsing with deionized water and air-drying;
- group 4: Immersion in 20% HF under the same conditions as group 3;
- group 5: Immersion in 30% HF under the same conditions as groups 3 and 4;
- group 6: Combination treatment consisting of air abrasion with 110-micrometer Al_2O_3 particles at 3.5 bar for 10 s, followed by immersion in 30% HF at 60°C for 60 min and subsequent silanization.

A total of 360 specimens were prepared and equally distributed among the 6 surface treatment groups. Each group comprised 30 specimens for RelyX Unicem and 30 specimens for Panavia F 2.0, resulting in 180 specimens for each type of resin cement.

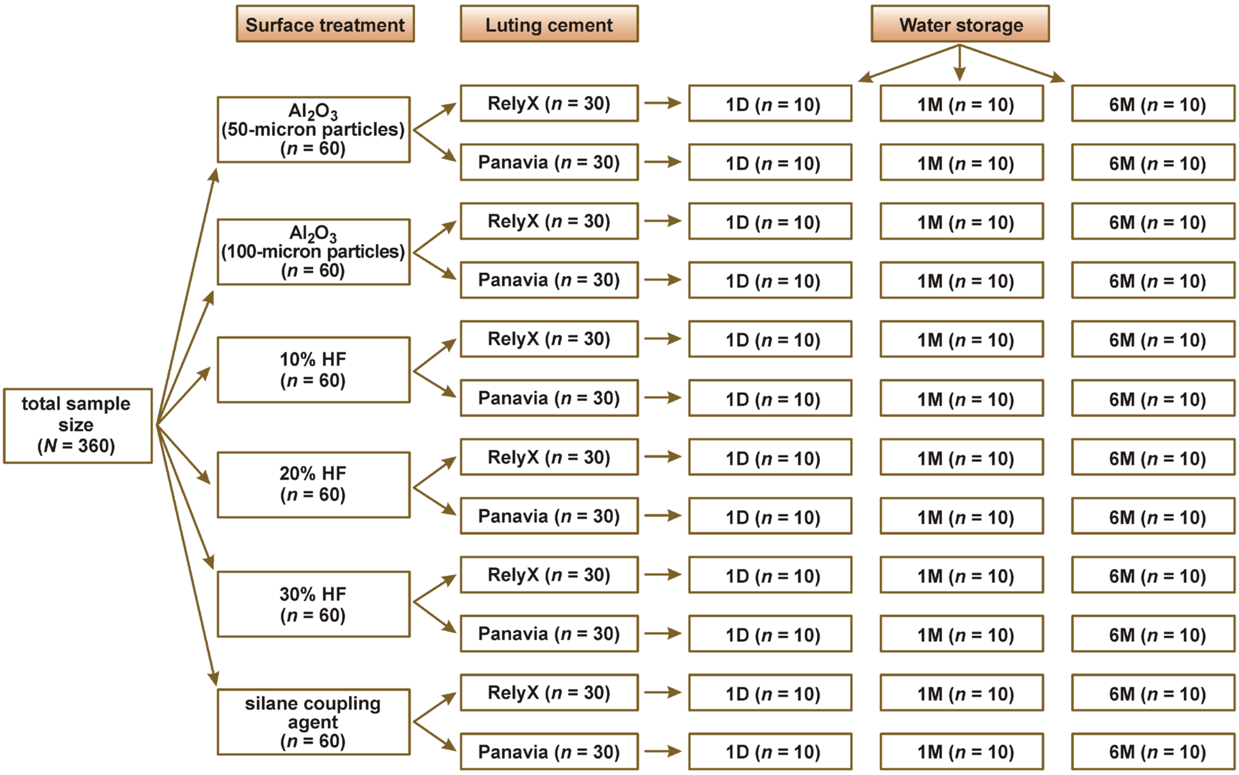

Fig. 1. Study design

Water-storage durations: 1D – 1 day (24 h); 1M – 1 month; 6M – 6 months. Al_2O_3 – aluminum oxide (alumina); HF – hydrofluoric acid.

Bonding of zirconia specimens with resin cement blocks

Of the 360 specimens prepared, 120 specimens – 60 bonded with RelyX Unicem and 60 with Panavia F 2.0 – underwent immediate testing to assess the initial bond strength. The remaining 240 specimens were allocated for extended testing following different water-storage durations to evaluate the effects of aging on bond strength. Prior to bonding, the specimens were cleaned and primed according to the respective surface treatment protocols. Bonding was performed at room temperature under a constant load of 75 N.

Water storage

Following the bonding procedure, the specimens were immersed in distilled water according to the following storage periods:

- category 1: Specimens were immersed in distilled water for 24 h (1 day –1D) to assess the initial bond strength following water exposure;
- category 2: Specimens were stored in distilled water for 1 month (1M), with weekly water changes, to assess the intermediate-term stability of the bond;
- category 3: Specimens were stored in distilled water for 6 months (6M), with weekly water changes, to assess the long-term durability and performance of the bonded interface.

Shear bond strength testing

Shear bond strength was evaluated using a universal testing machine (UTM) at a crosshead speed of 1 mm/min. The maximum load recorded at the time of debonding was measured in kilograms (kg), converted to newtons (N), and then divided by the bonding area to calculate SBS, expressed in megapascals (MPa).

Results

In a comparative analysis of the RelyX and Panavia dental cements, the mean SBS values and standard deviations (*M* ±*SD*) were assessed across 6 groups at 3 water-storage durations – 1D, 1M and 6M. Overall, the results indicated a tendency toward decreased SBS with prolonged water storage, although the pattern varied among the groups and luting agents (Table 1).

Specimens in category 1 generally showed the highest SBS values, whereas those in categories 2 and 3 exhibited greater reductions following prolonged water storage. In group 1, the mean SBS for RelyX decreased from 17.12 ±0.71 MPa at 1D to 8.54 ±6.42 MPa at 6M, while for Panavia, the values decreased from 21.76 ±2.81 MPa to 7.10 ±2.80 MPa. Group 2 showed a different pattern, with RelyX reaching its highest mean value at 1M (24.47 ±2.95 MPa), whereas Panavia exhibited its highest mean value at 1D

Table 1. Shear bond strength (SBS) values [MPa] for the RelyX and Panavia cements according to different surface treatment modalities (groups) under different water-storage (aging) conditions

Group	Water-storage duration	RelyX		Panavia	
		n	SBS [MPa]	*n*	SBS [MPa]
1	1D	10	17.12 ±0.71	10	21.76 ±2.81
	1M	10	10.76 ±1.06	10	17.61 ±3.48
	6M	10	8.54 ±6.42	10	7.10 ±2.80
	total	30	12.14 ±5.20	30	15.49 ±6.93
2	1D	10	23.89 ±10.04	10	32.41 ±10.04
	1M	10	24.47 ±2.95	10	22.96 ±15.86
	6M	10	16.84 ±10.35	10	17.38 ±6.48
	total	30	21.73 ±8.93	30	24.25 ±12.74
3	1D	10	20.45 ±3.37	10	19.01 ±4.93
	1M	10	16.91 ±1.34	10	13.03 ±3.54
	6M	10	10.81 ±1.95	10	9.62 ±2.90
	total	30	16.06 ±4.66	30	13.89 ±5.44
4	1D	10	18.58 ±3.49	10	20.52 ±2.36
	1M	10	16.05 ±2.11	10	10.31 ±2.85
	6M	10	9.92 ±5.18	10	8.06 ±4.73
	total	30	14.85 ±5.21	30	12.96 ±6.45
5	1D	10	18.31 ±2.89	10	21.37 ±0.95
	1M	10	14.53 ±4.39	10	8.65 ±3.99
	6M	10	9.73 ±4.36	10	5.44 ±2.17
	total	30	14.19 ±5.22	30	11.82 ±7.46
6	1D	10	31.07 ±3.28	10	31.94 ±3.59
	1M	10	26.47 ±2.86	10	23.97 ±3.27
	6M	10	19.98 ±3.56	10	17.94 ±3.68
	total	30	25.84 ±5.59	30	24.62 ±6.75

Data presented as mean ± standard deviation (*M* ± *SD*).
Groups: 1 – air abrasion (50-micron Al_2O_3 particles); 2 – air abrasion (100-micron Al_2O_3 particles); 3 – acid etching with 10% HF; 4 – acid etching with 20% HF; 5 – acid etching with 30% HF; 6 – combination treatment with a silane coupling agent.

Table 2. ANOVA results for the RelyX and Panavia cements under different water-storage conditions for each group

Group	Cement	Comparison between the groups (*F*; *p*-value)	Comparison within the groups (mean square)	Total (sum of squares)
1	RelyX	13.892; $p < 0.000$*	14.293	783.1
	Panavia	61.500; $p < 0.000$*	9.300	1,392.4
2	RelyX	2.499; $p = 0.101$*	72.227	2,311.1
	Panavia	4.400; $p = 0.022$*	131.500	4,706.1
3	RelyX	42.046; $p < 0.000$*	5.660	628.8
	Panavia	15.000; $p < 0.000$*	15.100	859.1
4	RelyX	13.702; $p < 0.000$*	14.485	788.0
	Panavia	36.600; $p < 0.000$*	12.000	1,206.1
5	RelyX	11.898; $p < 0.000$*	15.563	790.5
	Panavia	98.900; $p < 0.000$*	7.200	1,613.3
6	RelyX	29.449; $p < 0.000$*	10.542	905.5
	Panavia	39.800; $p < 0.000$*	12.400	1,320.6

Groups: 1 – air abrasion (50-micron Al_2O_3 particles); 2 – air abrasion (100-micron Al_2O_3 particles); 3 – acid etching with 10% HF; 4 – acid etching with 20% HF; 5 – acid etching with 30% HF; 6 – combination treatment with a silane coupling agent.
* statistically significant.

(32.41 ±10.04 MPa). Groups 3, 4, 5, and 6 generally demonstrated decreasing SBS values with prolonged water storage. For example, in Group 4, the mean SBS for RelyX decreased from 18.58 ±3.49 MPa at 1D to 9.92 ±5.18 MPa at 6M, while Panavia decreased from 20.52 ±2.36 MPa to 8.06 ±4.73 MPa). Overall, these findings suggest differences in the stability and bonding performance of the 2 luting agents under different water-storage conditions.

Table 2 summarizes differences in SBS among the 2 luting agents, STMs and aging conditions. It provides a comparative assessment of the performance of RelyX and Panavia under the various experimental conditions and illustrates the effect of prolonged water storage on bond strength.

Table 3 presents the results of the post hoc tests evaluating differences in SBS between zirconia specimens bonded

Table 3. Post hoc test results for the RelyX and Panavia cements

Group	Comparison	Cement	*MD*	*SE*	*p*-value
1	1D vs. 1M	RelyX	6.36	1.691	0.001*
		Panavia	4.15	1.360	0.005*
	1D vs. 6M	RelyX	8.58	1.691	0.000*
		Panavia	14.66	1.360	0.000*
	1M vs. 6M	RelyX	2.22	1.691	0.200
		Panavia	10.51	1.360	0.000*
2	1D vs. 1M	RelyX	−0.58	3.800	0.880
		Panavia	9.45	5.128	0.076
	1D vs. 6M	RelyX	7.05	3.800	0.074
		Panavia	15.03	5.128	0.007*
	1M vs. 6M	RelyX	7.63	3.800	0.055
		Panavia	5.58	5.128	0.286
3	1D vs. 1M	RelyX	3.54	1.064	0.003*
		Panavia	5.98	1.735	0.002*
	1D vs. 6M	RelyX	9.64	1.064	0.000*
		Panavia	9.39	1.735	0.000*
	1M vs. 6M	RelyX	6.10	1.064	0.000*
		Panavia	3.41	1.735	0.060
4	1D vs. 1M	RelyX	2.53	1.702	0.041*
		Panavia	10.21	1.551	0.000*
	1D vs. 6M	RelyX	8.66	1.702	0.000*
		Panavia	12.46	1.551	0.000*
	1M vs. 6M	RelyX	6.13	1.702	0.001*
		Panavia	2.25	1.551	0.159
5	1D vs. 1M	RelyX	3.78	1.764	0.004*
		Panavia	12.72	1.198	0.000*
	1D vs. 6M	RelyX	8.58	1.764	0.000*
		Panavia	15.93	1.198	0.000*
	1M vs. 6M	RelyX	4.80	1.764	0.011*
		Panavia	3.21	1.198	0.012*
6	1D vs. 1M	RelyX	4.60	1.452	0.004*
		Panavia	7.97	1.573	0.000*
	1D vs. 6M	RelyX	11.09	1.452	0.000*
		Panavia	14.00	1.573	0.000*
	1M vs. 6M	RelyX	6.49	1.452	0.000*
		Panavia	6.03	1.573	0.001*

Groups: 1 – air abrasion (50-micron Al_2O_3 particles); 2 – air abrasion (100-micron Al_2O_3 particles); 3 – acid etching with 10% HF; 4 – acid etching with 20% HF; 5 – acid etching with 30% HF; 6 – combination treatment with a silane coupling agent.
MD – mean difference; *SE* – standard error; * statistically significant.

with the 2 luting agents, RelyX and Panavia, across the different aging durations. In group 1, significant mean differences in SBS were observed with regard to 1D, 1M and 6M, indicating significant differences for the 2 luting agents at nearly all tested durations (except 1M vs. 6M for RelyX). In group 2, a significant difference emerged at 6M vs. 1D for Panavia, suggesting differences in bond performance following prolonged aging. In group 3, significant differences in SBS were observed with regard to all 3 aging durations (1D, 1M and 6M). Similarly, Groups 4 and 6 demonstrated significant mean differences across all tested durations. Group 5 also showed significant differences at 1D vs. 1M and 6M. Overall, these findings underscore the significant impact of simulated aging on the performance of Relyx and Panavia when applied to zirconia, providing valuable insights for their clinical application.

Table 4 presents a statistical comparison of groups 1–6 under different water-storage conditions. In group 1, a significant difference was observed at 1D ($p < 0.001$; $t = -5.063$), with a mean difference of −4.64 and a standard error (*SE*) of 0.92, a trend consistent at a duration of 1M, but not sustained at 6M ($p = 0.465$), where the direction reversed. Group 2 showed a highly significant difference at 1M ($p < 0.001$; $t = 0.295$), with a mean difference of 1.51 and

Table 4. Comparisons between the 2 cements in different groups and under different water-storage conditions

Comparison	*F*	*p*-value	*t*	df	*p*-value (2-tailed)	*MD*	*SE*
1-1D	8.099	0.011*	−5.063	18	0.000*	−4.64	0.92
			−5.063	10.141	0.000*	−4.64	0.92
1-1M	3.299	0.086	−5.955	18	0.000*	−6.85	1.15
			−5.955	10.653	0.000*	−6.85	1.15
1-6M	2.121	0.162	0.746	18	0.465	1.60	2.14
			0.746	12.560	0.469	1.60	2.14
2-1D	0.187	0.671	−1.897	18	0.074	−8.52	4.49
			−1.897	18.000	0.074	−8.52	4.49
2-1M	29.251	0.000*	0.295	18	0.771	1.51	5.10
			0.295	9.622	0.774	1.51	5.10
2-6M	3.406	0.081	−0.140	18	0.890	−0.54	3.86
			−0.140	15.123	0.891	−0.54	3.86
3-1D	1.847	0.191	0.761	18	0.457	1.44	1.89
			0.761	15.923	0.458	1.44	1.89
3-1M	10.063	0.005*	3.240	18	0.005*	3.88	1.20
			3.240	11.522	0.007*	3.88	1.20
3-6M	0.770	0.392	1.076	18	0.296	1.19	1.10
			1.076	15.763	0.298	1.19	1.10
4-1D	0.530	0.476	−1.452	18	0.164	−1.93	1.33
			−1.452	15.821	0.166	−1.93	1.33
4-1M	0.486	0.494	5.114	18	0.000*	5.74	1.12
			5.114	16.562	0.000*	5.74	1.12
4-6M	0.251	0.622	0.837	18	0.414	1.86	2.22
			0.837	17.852	0.414	1.86	2.22
5-1D	8.223	0.010*	−3.177	18	0.005*	−3.06	0.96
			−3.177	10.927	0.009*	−3.06	0.96
5-1M	0.134	0.718	3.135	18	0.006*	5.88	1.88
			3.135	17.837	0.006*	5.88	1.88
5-6M	5.171	0.035*	2.781	18	0.012*	4.28	1.54
			2.781	13.193	0.015*	4.28	1.54
6-1D	0.036	0.851	−0.568	18	0.577	−0.87	1.54
			−0.568	17.853	0.577	−0.87	1.54
6-1M	0.019	0.892	1.813	18	0.086	2.49	1.37
			1.813	17.681	0.087	2.49	1.37
6-6M	0.232	0.636	1.258	18	0.225	2.04	1.62
			1.258	17.982	0.225	2.04	1.62

Groups: 1 – air abrasion (50-micron Al_2O_3 particles); 2 – air abrasion (100-micron Al_2O_3 particles); 3 – acid etching with 10% HF; 4 – acid etching with 20% HF; 5 – acid etching with 30% HF; 6 – combination treatment with a silane coupling agent. Water storage durations: 1D – 1 day (24 h); 1M – 1 month; 6M – 6 months. df – degrees of freedom; * statistically significant.

$SE = 5.10$, whereas differences at 1D and 6M were not statistically significant. Group 3 demonstrated a significant difference at 1M ($p = 0.005$; $t = 3.240$), with a mean difference of 3.88 and $SE = 1.20$, while no significant differences were observed at 1D or 6M. In Group 4, a highly significant difference was observed at 1M ($p < 0.001$; $t = 5.114$), with a mean difference of 5.74 and $SE = 1.12$, whereas differences at 1D and 6M were not significant. Group 5 showed a significant difference at 1D ($p = 0.010$; $t = -3.177$), with a mean difference of −3.06 and $SE = 0.96$, with similar significant differences observed at 1M and 6M. Finally, Group 6 showed no statistically significant difference at 1D, 1M or 6M. Overall, these findings provide insights into the comparative performance of these dental cements across different conditions.

Discussion

The outcomes of this study provide important insights into the application of zirconia in dental prosthetics, highlighting the crucial role of surface treatment in the longevity and performance of zirconia-based restorations. Acid-etched surfaces demonstrated higher bond strength under the experimental conditions, suggesting the potential for more effective and durable adhesive techniques in clinical practice. These findings underscore the importance of appropriate surface preparation in optimizing the mechanical properties of zirconia restorations, which are often subjected to substantial mechanical stress and wear over time.

The superior bond strength observed with the acid-etching treatment compared with air abrasion in the present study is consistent with findings reported in other studies.[14,17–19] Collectively, these findings suggest that acid treatment may enhance the surface characteristics of zirconia and promote improved interaction with resin cements under certain experimental conditions. This may contribute to greater bond durability and potentially reduce the risk of restoration failure. Further research is warranted to establish standardized and clinically validated surface treatment protocols to incorporate acid etching more broadly, thus enhancing the overall success rates of zirconia restorations.

Moreover, the observed decrease in bond strength over time, particularly following prolonged water storage, highlights an important area for improvement in dental cement formulations. Both RelyX and Panavia demonstrated reductions in bond strength under the aging conditions evaluated, which may have implications for the long-term performance of zirconia restorations. This degradation presents an important challenge in adhesive dentistry and underscores the need for further research into more durable bonding systems capable of maintaining stable adhesion under prolonged exposure to the challenging conditions encountered in the oral environment.[19–24]

Furthermore, the findings regarding the differential performance of the various luting agents and surface treatment modalities under simulated aging conditions provide valuable insights into the potential long-term performance of zirconia restorations. Dental practitioners and materials scientists should consider these variables when selecting and developing restorative materials, respectively. Such considerations are important for advancing patient care through the use of materials and bonding protocols that provide greater resistance to degradation over time.[18,20,21,24,25]

Additionally, the differences observed among the tested groups warrant further investigation into how specific surface modifications influence micromechanical retention and chemical interaction at the zirconia–resin cement interface. A better understanding of these mechanisms may help optimize surface treatment protocols and improve the bonding performance and long-term stability of zirconia-based restorations.[14,15,26–28]

Our findings support further consideration of surface treatment and cementation protocols in the clinical management of zirconia restorations. By providing empirical evidence on the behavior of different luting agents and surface treatment modalities under the tested aging conditions, this study contributes to a better understanding of zirconia–resin cement interaction and may help guide the refinement of bonding protocols. Further clinical research is warranted to determine whether these findings translate into improved long-term durability and reliability of zirconia restorations.

Limitations

While this study provides valuable insights into the bond strength of zirconia following specific surface treatment and cementation protocols, its laboratory setting limits the direct extrapolation of the findings to clinical scenarios. The use of water storage to simulate aging, although useful for preliminary assessment, may not fully reproduce the complex biological, chemical, thermal, and mechanical conditions present in the oral environment or capture long-term clinical outcomes. Additionally, mechanical testing was performed under controlled uniaxial loading, which does not fully represent the complex and multidirectional stresses experienced by dental restorations in vivo. Further research incorporating longer-term clinical follow-up and more clinically representative, multidirectional loading conditions is recommended to validate and extend these findings.

Conclusions

In conclusion, the study rejects the 3 null hypotheses, demonstrating that surface treatment protocols significantly affect the SBS of zirconia, that different luting agents, particularly RelyX and Panavia, exhibit different bonding performance, and that artificial aging through water storage significantly reduces bond strength. These findings highlight the importance of both surface treatment and aging conditions in determining the performance of luting agents used with zirconia restorations. Additionally, the study found that the application of a silane coupling agent enhanced the bond strength of RelyX Unicem to yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) before aging, whereas HF treatment reduced the surface roughness of Y-TZP. Overall, water storage generally resulted in decreased bond strength for both luting agents, emphasizing the importance of long-term bond durability in zirconia-based restorations.

Ethics approval and consent to participate

Not applicable.

Data availability

The datasets supporting the findings of the current study are available from the corresponding author on reasonable request.

Consent for publication

Not applicable.

Use of AI and AI-assisted technologies

During the preparation of this manuscript, the authors used DeepL AI platform for language checking and grammar correction.

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References

1. Nistor L, Grădinaru M, Rîcă R, et al. Zirconia use in dentistry – manufacturing and properties. *Curr Health Sci J*. 2019;45(1):28–35. doi:10.12865/CHSJ.45.01.03
2. Elissalde C, Chung UC, Josse M, et al. Single-step sintering of zirconia ceramics using hydroxide precursors and spark plasma sintering below 400°C. *Scr Mater*. 2019;168:134–138. doi:10.1016/j.scriptamat.2019.04.037
3. Deng B, Luo J, Harris JT, Smith CM. Critical stress map for ZrO_2 tetragonal to monoclinic phase transformation in ZrO_2-toughened glass-ceramics. *Materialia*. 2020;9:100548. doi:10.1016/j.mtla.2019.100548
4. Soleimani F, Jalali H, Mostafavi AS, Zeighami S, Memarian M. Retention and clinical performance of zirconia crowns: A comprehensive review. *Int J Dent*. 2020;2020:8846534. doi:10.1155/2020/8846534
5. Raigrodski AJ. Contemporary materials and technologies for all-ceramic fixed partial dentures: A review of the literature. *J Prosthet Dent*. 2004;92(6):557–562. doi:10.1016/j.prosdent.2004.09.015
6. Abdulazeez MI, Majeed MA. Fracture strength of monolithic zirconia crowns with modified vertical preparation: A comparative in vitro study. *Eur J Dent*. 2022;16(1):209–214. doi:10.1055/s-0041-1735427
7. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. *J Prosthodont Res*. 2013;57(4):236–261. doi:10.1016/j.jpor.2013.09.001
8. Ezzat Y, Sharka R, Rayyan M, Al-Rafee M. Fracture resistance of monolithic high-translucency crowns versus porcelain-veneered zirconia crowns after artificial aging: An in vitro study. *Cureus*. 2021;13(12):e20640. doi:10.7759/cureus.20640
9. Vaid NR, Adel SM. Contemporary orthodontic workflows: A panacea for efficiency? *Semin Orthod*. 2023;29(1):1–3. doi:10.1053/j.sodo.2023.02.002
10. Russo DS, Cinelli F, Sarti C, Giachetti L. Adhesion to zirconia: A systematic review of current conditioning methods and bonding materials. *Dent J (Basel)*. 2019;7(3):74. doi:10.3390/dj7030074
11. Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: A systematic review and meta-analysis. *J Adhes Dent*. 2015;17(1):7–26. doi:10.3290/j.jad.a33525
12. Snyder T. Keys to bonding esthetic, durable zirconia restorations: Two case reports. *Compend Contin Educ Dent*. 2023;44(8):e1–e4. PMID:37708046.
13. Rapani A, Berton F, Tramontin A, et al. Surface roughness of enamel and dentin after preparation finishing with rotary burs or piezoelectric instruments. *Prosthesis*. 2023;5(3):711–720. doi:10.3390/prosthesis5030050
14. Kim SH, Cho SC, Lee MH, Kim HJ, Oh NS. Effect of 9% hydrofluoric acid gel hot-etching surface treatment on shear bond strength of resin cements to zirconia ceramics. *Medicina (Kaunas)*. 2022;58(10):1469. doi:10.3390/medicina58101469
15. Su Z, Li M, Zhang L, et al. A novel porous silica-zirconia coating for improving bond performance of dental zirconia. *J Zhejiang Univ Sci B*. 2021;22(3):214–222. doi:10.1631/jzus.B2000448
16. Fidalgo-Pereira R, Torres O, Carvalho Ó, et al. A scoping review on the polymerization of resin-matrix cements used in restorative dentistry. *Materials (Basel)*. 2023;16(4):1560. doi:10.3390/ma16041560
17. Roy AK, Prasad GN, Bhagat TV, et al. Analysis of surface conditioning methods on core–veneer bond strength of CAD/CAM zirconia restorations. *Technol Health Care*. 2021;29(3):467–477. doi:10.3233/THC-202539
18. Muhammed HA, Mahmoud EM, Fahmy AE, Nasr DM. The effect of sandblasting versus acid etching on the surface roughness and biaxial flexural strength of CAD/CAM resin-matrix ceramics (in vitro study). *BMC Oral Health*. 2023;23(1):169. doi:10.1186/s12903-023-02883-6
19. Amaral R, Ozcan M, Bottino MA, Valandro LF. Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning. *Dent Mater*. 2006;22(3):283–290. doi:10.1016/j.dental.2005.04.021
20. Rigos AE, Sarafidou K, Kontonasaki E. Zirconia bond strength durability following artificial aging: A systematic review and meta-analysis of in vitro studies. *Jpn Dent Sci Rev*. 2023;59:138–159. doi:10.1016/j.jdsr.2023.04.002
21. Badarneh A, Choi JJE, Lyons K, Porter G, Waddell N, Li KC. The effect of aging on the wear performance of monolithic zirconia. *Dent Mater*. 2022;38(5):e136–e146. doi:10.1016/j.dental.2022.04.018
22. Levartovsky S, Cartier L, Brand M, Blasbalg JJ, Pilo R. The retentive strength of zirconium oxide crowns cemented by self-adhesive resin cements before and after 6 months of aging. *Materials (Basel)*. 2020;13(18):3998. doi:10.3390/ma13183998
23. Vaddamanu SK, Vyas R, Bavabeedu SS, Arora S, Badiyani BK, Kumar A. In vitro results of scanning technique on assessing cement thickness and interfacial nanoleakage of luted CAD/CAM-fabricated fiber posts. *J Pharm Bioallied Sci*. 2021;13(Suppl 1):S676–S678. doi:10.4103/jpbs.JPBS_761_20

24. Alrabeah G, Alomar S, Almutairi A, Alali H, ArRejaie A. Analysis of the effect of thermocycling on bonding cements to zirconia. *Saudi Dent J*. 2023;35(6):734–740. doi:10.1016/j.sdentj.2023.07.009
25. Sandes Farias DC, Gonçalves LM, Walter R, Chung Y, Blatz MB. Bond strengths of various resin cements to different ceramics. *Braz Oral Res*. 2019;33:e095. doi:10.1590/1807-3107bor-2019.vol33.0095
26. Heboyan A, Vardanyan A, Karobari MI, et al. Dental luting cements: An updated comprehensive review. *Molecules*. 2023;28(4):1619. doi:10.3390/molecules28041619
27. Leung GKH, Wong AWY, Chu CH, Yu OY. Update on dental luting materials. *Dent J (Basel)*. 2022;10(11):208. doi:10.3390/dj10110208
28. Giti R, Zarkari R. The effect of a zirconia primer on the shear bond strength of Y-TZP ceramic to three different core materials by using a self-adhesive resin cement. *J Indian Prosthodont Soc*. 2019;19(2):134–140. doi:10.4103/jips.jips_348_18