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BASIC RESEARCH:

The Effect of Beverage Type and Exposure Duration on Bond Strength Between Zirconia-Based CAD/CAM Ceramic and Resin Cement

Efecto del tipo de bebida y la duración de la exposición sobre la resistencia de unión entre la cerámica CAD/CAM a base de zirconia y el cemento resinoso

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ABSTRACT: This *in vitro* study aimed to evaluate the effects of beverages with different pH levels and various storage durations on the shear bond strength (SBS) of CAD-CAM zirconia bonded with dual-cure resin cement. A total of ninety specimens were prepared using CAD-CAM zirconia (Katana Zirconia, Kuraray, Japan) and a dual-cure resin cement (Panavia V5, Kuraray, Japan). The specimens were randomly divided into three groups according to the storage solution: distilled water, mineral water, and cola. Each group was further subdivided into three time intervals: 24 hours, 30 days, and 90 days (n=10 per subgroup). All specimens were stored at room temperature and exposed to the respective solution for 20 minutes daily. Shear bond strength testing was performed using a universal testing machine, and the results were analyzed by one-way ANOVA at a significance level of $\alpha=0.05$. The statistical analysis revealed no significant effect of the type of beverage ($p=0.114$), storage time ($p=0.214$), or the interaction between them ($p=0.433$) on SBS values. Although the lowest bond strength was observed in specimens stored in cola for 90 days (11.95±3.32 MPa), this difference was not statistically significant. The highest value was recorded in the distilled water group at 24 hours (18.65±5.93 MPa). Within the limitations of this study, the type of beverage and exposure duration did not significantly affect the shear bond strength of the zirconia-resin cement interface. However, acidic beverages such as cola may demonstrate a trend toward decreased bond strength following prolonged exposure.

KEYWORDS: Zirconia; Resin cement; Bond strength; Beverages; Aging.

RESUMEN: Este estudio *in vitro* tuvo como objetivo evaluar los efectos de bebidas con diferentes niveles de pH y distintos tiempos de almacenamiento sobre la resistencia adhesiva al corte (SBS) de una zirconia CAD-CAM cementada con un cemento resinoso de curado dual. Se prepararon noventa muestras utilizando zirconia CAD-CAM (Katana Zirconia, Kuraray, Japón) y un cemento resinoso de curado dual (Panavia V5, Kuraray, Japón). Las muestras se dividieron aleatoriamente en tres grupos según la solución de almacenamiento: agua destilada, agua mineral y refresco de cola. Cada grupo se subdividió en tres periodos de almacenamiento: 24 horas, 30 días y 90 días (n=10 por subgrupo). Todas las muestras se almacenaron a temperatura ambiente y se expusieron diariamente durante 20 minutos a su respectiva solución. Se realizó la prueba de resistencia adhesiva al corte utilizando una máquina de ensayos universal, y los resultados se analizaron mediante ANOVA de un solo factor con un nivel de significancia de $\alpha=0.05$. El análisis estadístico no reveló efectos significativos del tipo de bebida ($p=0.114$), el tiempo de almacenamiento ($p=0.214$), ni de la interacción entre ambos factores ($p=0.433$) sobre los valores de SBS. Aunque la resistencia más baja se observó en las muestras almacenadas en cola durante 90 días (11.95±3.32 MPa), esta diferencia no fue estadísticamente significativa. El valor más alto se registró en el grupo de agua destilada a las 24 horas (18.65±5.93 MPa). Dentro de las limitaciones de este estudio, ni el tipo de bebida ni la duración de la exposición afectaron significativamente la resistencia adhesiva al corte en la interfaz entre la zirconia y el cemento. Sin embargo, bebidas ácidas como la cola pueden mostrar una tendencia a reducir la resistencia con exposiciones prolongadas.

PALABRAS CLAVE: CAD/CAM; Óxido de circonio; Cemento de resina; Fuerza de adhesión; Bebidas; Envejecimiento.

INTRODUCTION:

In recent decades, evolving concepts in restorative dentistry have intensified the pursuit of novel restorative materials to meet both functional and aesthetic demands of patients and clinicians. Alongside direct restorative techniques, indirect restorations have gained increasing popularity because they offer superior strength, durability, and esthetics compared to conventional composite restorations (1,2). The introduction of adhesive resin cements and the continuous development of adhesive systems have further enhanced the clinical success of these restorations (3).

Computer-aided design/computer-aided manufacturing (CAD/CAM) technology has become integral to modern restorative practice, providing consistent material quality, reduced laboratory time, and cost-effectiveness (4). Among CAD/CAM materials, zirconia-based ceramics are widely preferred due to their high fracture toughness, biocompatibility, wear resistance, and favorable optical properties (5,6). Unlike glass-containing ceramics, zirconia has a dense polycrystalline structure without a silica phase, which renders conventional silane coupling agents ineffective (7). Therefore, durable adhesion to zirconia depends on surface treatments such as airborne-particle abrasion and the application of primers containing phosphate monomers (e.g., 10-methacryloyloxydecyl dihydrogen phosphate, MDP) that chemically interact with the zirconia surface (8,9).

The long-term clinical performance of zirconia restorations is closely related to the stability of the zirconia-resin cement interface (10). However, intraoral conditions-particularly dietary factors-can jeopardize this interface. Acidic beverages such as cola, fruit juices, and carbonated mineral waters are widely consumed and have been shown to alter surface roughness, microhardness, and color stability of dental materials (11,12). Their erosive potential depends on factors such as pH, buffering capacity, acid type, and calcium-phosphate content (13). Although several studies have reported the impact of acidic environments on the physical properties of CAD/CAM materials (14,15), limited evidence is available regarding how such challenges affect the bond strength between resin cements and zirconia-based ceramics.

Given the increasing clinical use of zirconia and the widespread consumption of acidic beverages, investigating the durability of zirconia-resin cement bonds under such conditions is of considerable clinical importance. Therefore, the aim of this study was to evaluate the effects of beverage type (cola, mineral water, distilled water) and exposure duration (24 h, 30 d, and 90 d) on the shear bond strength between resin cement and a zirconia-based CAD/CAM ceramic. The null hypotheses were that (H1) beverage type would have no effect on bond strength, (H2) exposure duration would have no effect, and (H3) there would be no interaction between these two factors.

MATERIAL METHODS

SAMPLE PREPARATION

A total of 90 zirconia-based CAD/CAM disks (Katana Zirconia, Kuraray Noritake, Japan) were prepared. The blocks were sectioned into 2-mm-thick slices using a precision saw (Isomet Low-Speed Saw, Buehler, IL, USA) and embedded in acrylic resin blocks for stabilization. The surfaces were ground under water for 10s using 600- and 1000-grit silicon carbide papers. Airborne-particle abrasion was then performed with 50 μm Al_2O_3 at 0.25 MPa for 10s. After ultrasonic cleaning (5 min) and drying with oil-free air, Clearfil Ceramic Primer Plus (Kuraray Noritake, Tokyo, Japan) was applied and air-dried for 10s.

Resin cement (Panavia V5, Kuraray Noritake, Tokyo, Japan) was placed using a stainless-steel cylindrical mold (2.6 mm internal diameter, 3.0 mm height) positioned perpendicularly to the zirconia surface. To prevent cement extrusion beneath the mold, excess cement was carefully removed with a microspatula while maintaining firm contact between the mold and specimen. Light polymerization was performed with an LED curing unit (Elipar, 3M ESPE, Seefeld, Germany; 1470 mW/cm²) for 20 s. To minimize methodological issues such as triangular cohesive failure within the cement, specimens were visually inspected, and defective ones were excluded. All prepared specimens were stored in distilled water at 37 °C for 24 h before aging procedures.

The materials used in the study and their contents are shown in Table 1.

Table 1. Materials used in the study.

Material	Type	Composition
Katana Zirconia	Ultra Translucent Multilayer Zirconia	ZrO_2, HfO_2, Y_2O_3, pigments.
Clearfil Ceramic Primer plus	Ceramic Primer	3-Trimethoxysilylpropyl methacrylate 10-Methacryloyloxydecyl dihydrogen phosphate, Ethanol.
Panavia V5	Resin Cement	Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, Initiators, Accelerators, Silanated barium glass filler, Silanated fluoroaluminosilicate glass filler, Colloidal silica, Silanated aluminium oxide filler, dl Camphorquinone, Pigments.

AGEING OF SPECIMENS

Specimens were randomly divided into three groups according to the immersion solution:

Cola (Coca-Cola®, The Coca Cola Company, USA; pH≈2.5, containing phosphoric acid), Carbonated mineral water (Kızılay®, Turkey; pH≈6.8, containing carbonic acid), Distilled water (pH≈7.0, used as control).

Each specimen was immersed daily in the assigned beverage for 20 minutes at room temperature (23±1 °C). After immersion, the specimens were rinsed with distilled water to remove surface residues and then stored in fresh distilled water at 37 °C for the remainder of the day. The storage medium was renewed daily to prevent contamination or pH alterations.

The total immersion periods were 24 hours, 30 days, and 90 days, simulating short-, medium-, and long-term intraoral exposure to beverages with different acidities.

The pH values of the beverages are presented in Table 2.

Table 2. The pH levels and acidity of the beverages utilized in the stud.

Beverages	Acid	pH
Coca-Cola	Phosphoric Acid	2.5
Mineral water	Carbonic Acid	6.8
Distilled water	-	7

MEASUREMENT OF SHEAR BOND VALUES

Shear bond strength was measured using a universal testing machine (Bisco Shear Bond Tester, Bisco Inc., Schaumburg, IL, USA) at a crosshead speed of 0.5 mm/min. A knife-edge chisel apparatus was positioned tangentially to the resin-zirconia interface to apply load without introducing bending stresses. The maximum load at failure (N) was recorded, and SBS values (MPa) were calculated by dividing the load by the bonded area ($\pi \cdot r^2$; r=1.3 mm).

Failure types were classified under a stereomicroscope at 40× magnification as adhesive (at interface), cohesive (within resin cement), or mixed. The laboratory phase workflow is shown in Figure 1.

STATISTICAL ANALYSIS

All experimental data were subjected to statistical evaluation using SPSS software (version 27.0; IBM Corp., Armonk, NY, USA). The normality of the datasets was examined with the Shapiro-Wilk test, confirming that all variables conformed to a normal distribution. Descriptive statistics are expressed as mean±standard deviation. A two-way analysis of variance (ANOVA) was employed to assess the effects of storage medium and storage duration on shear bond strength (SBS). Subsequent pairwise comparisons were performed using Tukey's post hoc test. Differences were considered statistically significant at a threshold of $p<0.05$.

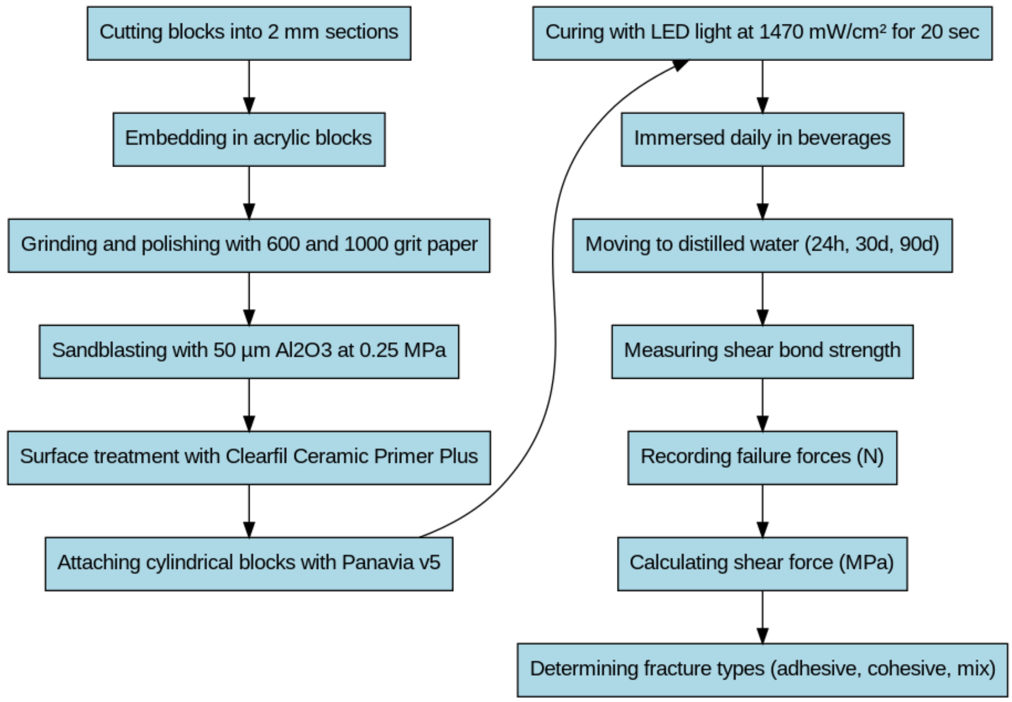

Figure 1. Workflow of the laboratory phase of the study.

RESULTS

The results of the statistical analysis revealed that the aging solution had no statistically significant effect on bond strength (p=0.114). Similarly, the storage duration in the solutions did not result in a significant difference in bond strength values (p=0.214). In addition, no significant interaction was observed between the aging solution and storage duration with respect to SBS values (p=0.433). The bond strength data obtained from the study, along with the corresponding means and standard deviations, are presented in Table 3.

The highest bond strength values were observed in the specimens stored in distilled water for 24 hours (18.65±5.93 MPa), while the lowest values were found in the specimens stored in cola for 90 days (11.95±3.32 MPa). However, the statistical analysis indicated that the type of aging solution had no significant effect on bond strength (p=0.114). Similarly, storage duration (24 hours, 30 days, and 90 days) did not significantly influence bond strength values (p=0.214). No statistically significant interaction was found between the aging solution and the storage time with respect to bond strength values (p=0.433).

The results of the failure type analyses indicated the presence of 37 adhesive fractures, 19 cohesive fractures, and 34 mixed fractures. The percentages of fracture types are shown in Table 4.

Although the bond strength appeared lower in the cola group after 90 days (11.95±3.32 MPa) compared to the mineral water and distilled water groups, the differences were not statistically significant (p>0.05). Similarly, although the highest bond strength was observed in distilled water at 24 hours (18.65±5.93 MPa), no statistically significant difference was found among the groups regarding the aging solution, storage duration, or their interaction (p=0.114, p=0.214, and p=0.433, respectively).

Table 3. Comparison of beverages according to storage time.

Beverages	Time	n	Mean + Std. Deviation
Distilled water	24 hours	10	18.65±5.93
	30 days	10	15.81±6.04
	90 days	10	16.29±6.42
Mineral water	24 hours	10	18.77±5.66
	30 days	10	15.04±5.94
	90 days	10	17.57±4.34
Cola	24 hours	10	15.38±6.22
	30 days	10	15.95±5.38
	90 days	10	11.95±3.32

Table 4. The failure patterns of bond strength of CAD/CAM materials according to beverages type.

	24 Hours	30 Days	90 Days
Distilled water	%60 Adhesive	%50 Adhesive	%50 Adhesive
	%30 Mix	%30 Mix	%50 Mix
	%10 Cohesive	%20 Cohesive	%0 Cohesive
Mineral water	%40 Adhesive	%60 Adhesive	%20 Adhesive
	%30 Mix	%30 Mix	%50 Mix
	%30 Cohesive	%10 Cohesive	%30 Cohesive
Cola	%20 Adhesive	%50 Adhesive	%20 Adhesive
	%40 Mix	%40 Mix	%40 Mix
	%40 Cohesive	%10 Cohesive	%40 Cohesive

DISCUSSION

The aim of this study was to evaluate the effect of beverages with different pH values on the bond strength between dental restorative materials and dual-cure resin cement. The results of the present study demonstrated that the beverages used for aging and the immersion periods did not create statistically significant differences in bond strength. Consequently, the null hypotheses tested in this study were accepted.

Once dental restorative materials are transferred to the oral cavity, they are continuously exposed to liquids with varying pH values (15). Although some studies have shown that beverages may affect the bond strength of restorations, research in this field remains limited (16). The key chemical parameter to be considered in determining whether a beverage is potentially erosive to dental tissues is the hydrogen ion concentration, i.e., the pH value (17,18). Throughout the day, the consumption of foods and drinks with different pH levels may have adverse effects on the tooth surface and on materials used in restorative dentistry. Previous research has indicated that beverages such as herbal teas, cola, and mineral water can influence the bonding of CAD/CAM materials (19,20,21). Khoda *et al*. (22) bonded orthodontic brackets to teeth and subsequently exposed them to acidic beverage solutions with pH values ranging from 2.95 to 3.71, after which the shear bond strength of the brackets was evaluated.

In the present study, no statistically significant differences were observed among specimens stored in different acidic beverages. This finding suggests that the pH levels of the beverages used for aging did not exert a pronounced effect on bond strength. Considering the conflicting results across previous studies and the outcomes of the current research, it is likely that these discrepancies are primarily due to methodological differences. In particular, variations in exposure times to beverages, the absence of standardized criteria for the pH levels of the tested solutions, and the diversity in the hydrophilic properties of primers and adhesive systems may account for inconsistent findings (23). Therefore, it can be concluded that pH alone is not a decisive factor in bond strength; rather, exposure duration, the chemical composition of the material, and environmental conditions must be considered collectively.

The Clearfil™ Primer Plus used in this study is a self-etch primer containing MDP (10-Methacryloyloxydecyl dihydrogen phosphate), which provides both chemical and physical interactions that enhance the bonding between CAD/CAM restorative material and dual-cure resin cement (24). The ability of MDP to chemically bond to the surface of restorative materials contributes to the durability of the cement-material bond, while the optimal viscosity and penetration capacity of the primer's solvent enhance surface contact and create a homogeneous bonding interface (25). These properties may have limited potential softening or surface dissolution effects of acidic beverages, thereby contributing to the preservation of bond strength. Furthermore, the chemical composition of Clearfil Primer Plus and its compatibility with dual-cure resin cement may have enhanced the long-term stability of the bond against mechanical stresses and environmental challenges. Thus, the absence of significant effects of pH differences on bond strength may be largely attributable to the chemical properties of the adhesive system and the cement-material interactions.

Since this study was conducted under *in vitro* conditions, the findings may not fully replicate the biological and mechanical dynamics of the oral environment. Variables such as salivary flow, thermal cycling, masticatory forces, and enzymatic activity observed in the clinical setting were beyond the scope of this study. In addition, only shear bond strength was evaluated; alternative methods such as microtensile or other mechanical tests were not employed. This limitation restricts the ability to provide a comprehensive assessment of bonding performance under different stress conditions. Nevertheless, the present findings provide valuable insights into material-cement interactions under the studied conditions and may serve as a guide for future research employing different testing methodologies and aging protocols.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn:

No statistically significant differences in bond strength were detected among specimens aged in different acidic beverages. This suggests that commonly consumed acidic beverages may not clinically compromise the bond between CAD/CAM restorations and dual-cure resin cement. The pH differences tested did not exert a decisive influence on bond strength; instead, the chemical interactions of the adhesive system and the stability of the material-cement interface appear to mitigate potential adverse effects of environmental acidity.

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ETHICAL AND CONSENT TO PARTICIPATE: No ethical requirements as human and animal samples are not used.

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