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Effect of decontamination methods on shear bond strength of resin cement to translucent monolithic zirconia

Efeito de métodos de descontaminação na resistência ao cisalhamento entre cimento resinoso e zircônia monolítica translúcida

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ABSTRACT

Objective: The aim of this study was to evaluate the effect of decontamination methods on shear bond strength of resin cement to translucent monolithic zirconia. Material and Methods: Eighty 4-mol yttriastabilized tetragonal zirconia polycrystal specimens were fabricated. All specimens were subjected to saliva contamination and divided into four groups (n = 20) according to the decontamination methods: 70% isopropanol (AL); zirclean (ZC); 5.25% sodium hypochlorite (NaOCL); and no cleaning (NC). All specimens were bonded by using self-adhesive resin cement. Half of the specimens in each group (n =10) were subjected to aging. All specimens were subjected to shear bond strength test in a universal testing machine. Failure modes were evaluated by using a scanning electron microscope (SEM). The data obtained were statistically analyzed by using one-way ANOVA followed by the HSD Tukey test (p < 0.05). Results: ANOVA test revealed a significant difference among the different decontamination methods (p < 0.05). No significant differences were found among NaOCL, ZC, and AL groups as revealed by the HSD Tukey test (p > 0.05). The effect of aging was significantly different in all groups (p < 0.05); whereas, no significant difference was found in group ZC (p > 0.05). The (SEM) analysis showed mixed failures in all groups, except in group NC where only adhesive failure was observed. Conclusion: Saliva contamination during clinical evaluation significantly decreases the shear bond strength between selfadhesive resin cement and translucent monolithic zirconia. The cleaning methods have an impact on improving the bond strength between resin cement and translucent monolithic zirconia.

KEYWORDS

Monolithic zirconia; Resin cement; Shear bond strength test; Translucent zirconia; Zirclean.

RESUMO

Objetivo: O objetivo deste estudo foi avaliar o efeito dos métodos de descontaminação na resistência ao cisalhamento entre cimento resinoso e zircônia monolítica translúcida. Material e métodos: Foram fabricados oitenta espécimes policristalinos de zircônia tetragonal estabilizada com ítria a 4 mol. Todas as amostras foram submetidas à contaminação salivar e divididas em quatro grupos (n 20) de acordo com os métodos de descontaminação: isopropanol a 70% (AL); zircleano (ZC); Hipoclorito de sódio a 5,25% (NaOCL); e sem limpeza (NC). Todas as amostras foram cimentadas utilizando um cimento resinoso autoadesivo. Metade das amostras de cada grupo (n = 10)foram submetidas ao envelhecimento. Todas as amostras foram submetidas ao teste de resistência ao cisalhamento em uma máquina de teste universal. Os módulos de falha foram avaliados usando um microscópio eletrônico de varredura (MEV). Os dados obtidos foram analisados estatisticamente utilizando ANOVA de uma via, seguida pelo teste de HSD Tukey (p <0,05). Resultados: O teste ANOVA revelou diferença significativa entre os diferentes métodos de descontaminação (p < 0.05). Não foram encontradas diferenças significativas entre os grupos NaOCl, ZC e AL, conforme revelado pelo teste HSD Tukey (p > 0.05). O efeito do envelhecimento foi significativamente diferente em todos os grupos (p <0,05); enquanto nenhuma diferença significativa foi encontrada no grupo ZC (p > 0,05). A análise (SEM) mostrou falhas mistas em todos os grupos, exceto no grupo NC, onde apenas foi observada falha adesiva. Conclusão: A contaminação por saliva durante a avaliação clínica diminui significativamente a resistência ao cisalhamento entre o cimento resinoso autoadesivo e a zircônia monolítica translúcida. Os métodos de limpeza têm um impacto na melhoria da resistência da união entre o cimento resinoso e a zircônia monolítica translúcida.

PALAVRAS-CHAVE

Zircônia monolítica; Cimento resinoso; Teste de resistência ao cisalhamento; Zircônia translúcida; Zircônia.

INTRODUCTION

T raditional 3-mol yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) restorations have been widely used in prosthetic dentistry due to their acceptable optical properties, superior mechanical properties, excellent biocompatibility, and less wear to the opposing dentition [1,2]. In addition, more conservative tooth preparation can be obtained particularly in patients with limited interocclusal space and parafunctional habits; however, the lack of translucency is considered their main drawback [3].

To improve translucency, super translucent 4-mol vttria-stabilized tetragonal zirconia materials polycrystal (4Y-TZP) containing tetragonal and cubic grains have been recently introduced [4]. Zirconia manufacturers increased the vttria content from 3 to 5 mol% enabling low phase transformation toughening where the tetragonal phase can be retained at room temperature; however, accomplishing high translucency resulted in diminished fracture toughness of 2.5 to 3.5 MPa\m1/2 and flexural strength of approximately 700 to 800 MPa [5-7]. Another noticeable reason for introducing these restorations is the high incidence of porcelain chippings (6% to 25% after 3 years) in bilavered zirconia ceramic restorations, which is significantly higher than the rates reported for metal-ceramic restorations [8-10].

The generation of (4-YTZP)new restorations is milled from pre-shaded super translucent multilayered (STML) blanks by using computer-aided design and computeraided manufacturing (CAD-CAM) technology [1]. The restoration color is homogeneous and can be easily polished by using special polishing materials [11]. Monolithic zirconia restorations can be luted by using conventional cement [12] or bonded by using adhesive resin cement which are considered the cement of choice for many clinical applications [13,14].

However, zirconia-resin bond strength is influenced by many factors, such as the ceramic wettability by adhesive resin cement, surface treatment of zirconia, the chemical composition of adhesive resins and probable contamination during bonding [15-19]. Several methods have been proposed to enhance zirconia-resin bond strength, such as acid etching, tribochemical silica coating, airborne-particle abrasion by using 50 μ m aluminum oxide particles (Al₂O₂), laser irradiation, and selective infiltration etching [20-24]. In addition; the application of 10-methacryloyloxydecyldihydrogenphosphate (10-MDP) or ester phosphoric methacrylate monomers after airborne-particle abrasion has been recommended to enhance zirconia-resin bond strength [25,26]. The enhanced bonding resulted from a chemical reaction that occurs between the phosphate ester monomer of the containing cement and the hydroxyl groups of the zirconia ceramic [27,28].

The zirconia treated surface can easily become contaminated with saliva during the clinical evaluation [29]. The deposition of a thin film of organic phosphates on the zirconia surface after a few s of saliva contamination is difficult to be removed with water resulting in reduced zirconia-resin bond strength [30]. The decontamination of the intaglio surface of zirconia restorations has been proposed in different studies, such as water rinse, 37% phosphoric acid, 70% isopropanol, hydrogen peroxide, and sodium hypochlorite (NaOCL) [31-37]. In addition, a new cleaning agent has been developed to absorb the phosphate contaminants from the intaglio surface of zirconia restorations to improve zirconia- resin bond strength [38,39].

Saliva contamination during the clinical evaluation of (4-YTZP) restorations can reduce their bond strength to resin cement. Limited data are available on the bonding performance of zirconia to resin cement after saliva decontamination; thus, the shear bond test is employed to evaluate the adhesive interfaces [40,41]. The aim of this study was to evaluate the effect of decontamination methods on shear bond strength of resin cement to translucent monolithic zirconia. The null hypothesis was that different decontamination methods would not affect the bond strength between adhesive resin cement and translucent monolithic zirconia.

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MATERIAL AND METHODS

A power analysis was performed to estimate the sample size to apply a statistical test of the research null hypothesis that there would be no difference in the effect of different decontamination methods on the shear bond strength of resin cement to translucent monolithic zirconia. Based on Tunc et al [34], a sample size of 24 specimens had a 95% power to detect a difference between means as received from the study with a significant level ($\alpha = .05$), and it was increased to 80 specimens (n = 20) per group to achieve more reliable results. Sample size calculation was performed using a statistical software program (G*Power v3.1.9.22).

Specimens preparation

The materials used in the present study are listed in Table I. A total of 80 specimens (14×14 \times 2.5 mm) were milled from a partially sintered (4Y-TZP) blank by using a precision saw machine (Isomet 4000; Buehler, Lake Bluff, IL, USA) under constant irrigation. The specimens were sintered in a special sintering furnace (5 motions furnace; Bredent, Senden, Germany) at 1550 °C according to the manufacturer instructions. Anticipating 20% volumetric shrinkage, the final dimension of the specimens was approximately $(12 \times 12 \times 2 \text{ mm})$. All specimens were sequentially wet polished with 600-grit rotating silicon carbide paper (Buehler, Lake Bluff, IL, USA) and air-abraded by using 50 μ m AL2O3 particles (Protechno, Girona, Spain) at 0.25 MPa pressure in an airborne-particle abrasion unit (ESB 2; Eurocem Srl, Milanese, Italy) for 15 s at a distance of 10 mm.

Table I - Materials used in this stu	dy.
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Material	Manufacturer	Lot No.	Composition
Katana Zirconia STML	Kuraray Noritake Dental Inc, Aichi, Japan	DYBIR	$\begin{array}{l} \text{4Y-TZP}; (\text{ZrO}_2 + \text{HfO}_2 + \text{Y}_2 \text{O}_3) \\ > 99\%, \text{yttrium oxide} \\ (\text{Y}_2 \text{O}_3) > 4\%, \text{hafnium oxide} \\ (\text{HfO}_2) \leq 5\%, \text{other oxides} \\ \leq 1\% \end{array}$
Zirclean	Bisco, Schaum- burg, IL, USA	1800007913	Zirconium oxide, water, polyethylene glycol, potassium hydroxide, pigments, additives
RelyX Unicem	3M ESPE, Seefeld, Germany	4348049	Powder: glass powder, silica, cal- cium hydroxide, initiator Liquid: methacrylated phosphoric acid ester, dimethacrylate, initiator

Surface decontamination methods

The specimens were steam cleaned (EGV 18; Eurocem Srl, Milanese, Italy), air-dried, then contaminated with saliva. Saliva was obtained from one healthy male donor who had refrained from eating or drinking for 2 h before the collection process. The saliva was left undisturbed for 10 min and then rinsed with water for 15 s and air-dried for 15 s at a 10 mm distance. This procedure was performed following the ethical regulations of Faculty of Dentistry of Beni-Suef university (approval# FDBSUREC/13032019/ FA).

Samples were assigned into 4 groups (n = 20) according to the decontamination methods: Group AL: saliva contamination and immersion in 70% isopropanol for 2 min; Group ZC: saliva contamination and application of cleaning gel (Zirclean) by using a microbrush for 20 s; Group NaOCL: saliva contamination and immersion in 5.25% percent sodium hypochlorite for 2 min; and Group NC: saliva contamination only. Subsequently, all specimens were rinsed with distilled water for 15 s and air-dried for 10 s.

Bonding procedures

For standardization of the bonding surface area, tygon tubes (2 mm diameter, 2 mm height) were placed in the center of (4Y-TZP) specimens. The self-adhesive resin cement was mixed and pressed through the tubes against the (4Y-TZP) surfaces and light-cured from all directions for 40 s at 1250 mW\cm2 (Mini LED; Satelec Acteon Group, Mérignac Cedex, France). The tubes were carefully detached, and the resin cement cylinders were light-cured for an extra 40 s. All specimens were stored in distilled water for 24 h at 37 °C. Half of the specimens in each group (n = 10) were subjected to aging in distilled water between 5 °C and 55 °C for 5000 cycles (Thermocycler-1100; SD Mechatronik, Feldkirchen-W., Germany) before testing [42,43]. The dwell time was 20 s and the transfer time was 2 s.

Shear bond strength test

The shear bond strength test was performed in a universal testing machine (Instron 3345; Instron Corp., Norwood, MA, USA) at a crosshead speed of 1.0 mm\min and a load cell of 5 KN until failure occurred (Figure. 1). The data were recorded using computer software (Bluehill Lite; Instron Corp., Norwood, MA, USA) and the values were reported in MPa.

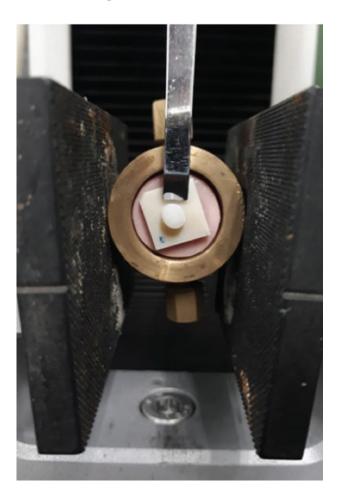


Figure 1 - Specimen placed in universal testing machine for the shear strength test.

SEM analysis

Representative debonded specimen from each group was examined under a scanning electron microscope (Quanta 250 FEG; FEI, Netherlands) with an accelerating voltage of 30 kV at a magnification of $2500 \times$ to define the failure mode which was classified as adhesive failure including complete debonding between the resin cement and the (4Y-TZP) surface, cohesive failure within the resin cement, and mixed failure including retained cement on the (4Y-TZP) surface.

Statistical analysis

Numerical data were explored for normality by checking the distribution of data and using the Kolmogorov-Smirnov and Shapiro-Wilk tests. Data showed normal (parametric) distribution were presented as mean, standard deviation (SD). One-way ANOVA followed by HSD Tukey tests were used to study the effect of the decontamination methods (p < 0.05). T-test was used to study the effect of aging. Statistical analysis was performed by using a statistical software program (IBM SPSS Statistics v.20; SPSS Inc., Chicago, IL, USA).

RESULTS

The means and standard deviations of the shear bond strength values in MPa for all nonaged and aged groups are presented in Table II. One-way ANOVA test revealed that different decontamination methods had a statistically significant effect on the shear bond strength between (4Y-TZP) and the resin cement (p < 0.05); however, no statistically significant difference was found among NaOCL, ZC, and AL groups as revealed by the HSD Tukey test (p > 0.05). Within each of the decontamination methods, the mean shear bond strength values decreased for all aged groups. The effect of aging was significantly different in group NaOCL (p =0.012), group AL (p = 0.004), and group NC (p= 0.006; however, no significant difference was observed in group ZC (p = 0.402).

Representative SEM images with $2500 \times$ magnification of debonded specimens after shear bond strength test demonstrated residual resin cement-retained on the (4Y-TZP) surfaces in non-aged and aged AL, ZC, and NaOCL groups. This observation indicated mixed failures with reasonable bond strength values; however, no residual resin cement remained on the (4Y-TZP) surface in group NC indicating adhesive failures between the resin cement and the (4Y-TZP) surface with reduced bond strength values (Figure. 2).

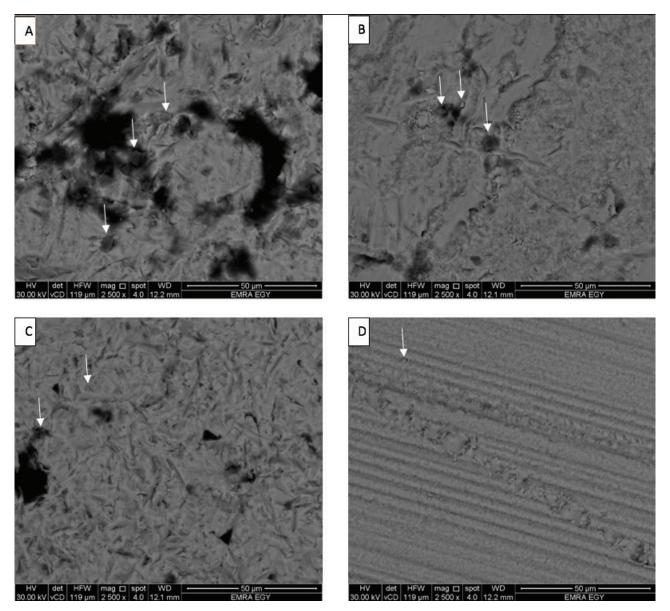


Figure 2 - Representative SEM image of debonded specimen from group ZC showing mixed failure with remnants of resin cement on the zirconia substrate. Original magnification 2500× (A).; Representative SEM image of debonded specimen from group NaOCL showing mixed failure with small traces of resin cement on the zirconia substrate. Original magnification 2500× (B); Representative SEM image of debonded specimen from group AL showing mixed failure with small traces of resin cement on the zirconia substrate. Original magnification 2500× (C); Representative SEM image of debonded specimen from group NC showing adhesive failure with almost no remnants of resin cement on the zirconia substrate. Original magnification 2500× (D).

Table II - Descriptive statistics of shear bond strength values (MPa), comparison between decontamination methods (ANOVA test)
and the effect of aging (t-test)

		Group (ZC)	Group (NaOCL)	Group (Al)	Group (NC)	F	р
Without	Mean	21.62ª	20.02ª	19.12 ^a	8.85 ^b	14.11	0.000*
Aging	SD 5.9	5.91	1.99	6.97	2.78		0.000
After Aging	Mean	16.99 ^A	15.75 ^A	14.75 ^A	5.38 ^B	23.72	0.000*
	SD	3.44	2.94	4.72	2.15		0.000*
Effect of	t	0.87	2.81	3.28	3.12		
Aging	р	0.402 ^{ns}	0.012	0.004*	0.006*		

Significance level p < 0.05; *significant, ns = non-significant; Tukey's post hoc test: within the same comparison of different decontamination methods, means sharing the same superscript letter are not significantly different.

DISCUSSION

The null hypothesis that different decontamination methods would not affect the bond strength between adhesive resin cement and translucent monolithic zirconia was rejected. The results showed that different decontamination methods had a significant effect on zirconia-resin bond strength after saliva contamination.

The success of zirconia restorations depends on micro-mechanical retention and chemical adhesion of the resin cement to the zirconia surface, which necessitates efficient decontamination to ensure surface activation [18]. It has been reported that contamination with blood or saliva after clinical evaluation of zirconia restorations resulted in lower bond strength between resin cement and zirconia [29-31].

In the present study, 70% alcohol was used as a decontamination agent and the results showed lower shear bond strength values than those of groups ZC and NaOCL; however, this difference was not significant. The obtained results were consistent with those of Yang et al [29] and Quaas et al [35] who found that alcohol cleaning was not effective in removing the phosphate contaminants.

In addition, an extraoral cleaning gel (Zirclean) was used to decontaminate the translucent monolithic (4-YTZP) surface; however, information is lacking about the effect

of this agent. Zirclean is an alkaline suspension of zirconium oxide particles, water, potassium hydroxide, and other additives. The high concentration and larger size of zirconium oxide particles act as a sponge that adsorbs the saliva phosphate contaminants rather than adherence to the zirconia surface, leaving behind a clean zirconia surface; therefore, enhancing zirconiaresin bond strength [32]. The results obtained in this study were consistent with those of Kim et al [40] who found that a reliable shear bond strength value could be obtained after Ivoclean application when compared to uncontaminated zirconia. In contrast, contradictory results have been reported by Aladag et al [38] and Ishii et al [39] who compared the effect of various surface decontamination methods, including Ivoclean cleaning paste on zirconia-resin bond strength and concluded no significant differences in the bond strength values.

Moreover, 5.25% sodium hypochlorite was used in this study as a decontamination agent because of its capability to remove the salivary phosphates from the (4-YTZP) surface. The obtained bond strength values were found to be acceptable and consistent with those previously reported [36]. In contrast, it has been reported that excess sodium hypochlorite results in oxygen generation affecting the resin polymerization [37].

The shear bond strength values for nonaged and aged groups, showed statistically significant differences; however, these values exceed the minimum clinically acceptable bond strength value which is approximately 13 MPa [23,24]. These results emphasize that the decontamination methods used in this study were effective to achieve reliable zirconia-resin bond strength [31,40].

To maximize the clinical relevance of the present study; all (4-YTZP) specimens were air-abraded with 50 μ m Al2O3 to simulate the final restoration as delivered from the dental laboratory [17]. In addition, all specimens were subjected to saliva contamination to mimic the clinical evaluation of the restoration before the final cementation followed by application of the decontamination methods, and this has been confirmed by previous in vitro studies [30-32].

Based on previous studies [12,14,22], resin cement containing ester phosphoric methacrylate monomer was used in this study to enhance the zirconia-resin bond strength. It has been reported that the ester phosphoric methacrylate monomer present in the Relyx unicem was more efficient than MDP monomer for efficient zirconia bonding [26].

To evaluate the adhesive interfaces several tests have been used, such as the microshear, shear, microtensile, and tensile tests [41]. Although the microshear bond strength tests have been used in different studies [43-45], filling a tygon tube with the resin cement is very difficult resulting in many failures during specimen preparation, such as bubble inclusion and interfacial gap defect [44]. In addition, the pressure applied on the scalpel blade by the operator to remove the tygon tubes may be transferred to the resin cement cylinder. This resulted in the introduction of flaws, stress concentrations at the adhesive interface. and crack formation along the specimen. It was assumed that microshear specimens may fail prematurely due to the propagation of these cracks [45]. Therefore, the shear bond strength test was used in this study to evaluate the zirconia-resin bond strength after saliva contamination because of its simplicity, possibility of standardization, and simulating the clinical situation.

According the International to Organization for Standardization (ISO), all specimens in this study were subjected to aging protocol including 5000 cycles (equivalent to 6 months aging) in water between 5 °C and 55 °C to simulate artificial aging in the wet oral cavity environment [46,47]. Based on the results of this study, a significant decrease in shear bond strength values was recorded in all groups subjected to aging, except in group ZC whereas no significant difference was found. This could be attributed to the successful removal of salivary contaminates by the decontamination gel; thus, improving the zirconia-resin bond strength.

The failure modes were evaluated using SEM and supported the bond strength results. The main failure mode of group NC was an adhesive failure in non-aged and aged specimens, showing that the durability of zirconia-resin bond strength was not satisfactory.

These findings were consistent with the results obtained by the authors in previous studies who reported lowest bond strength values when no cleaning was performed after contamination [17,35]. On the other hand, the failure mode in all non-aged and aged groups, was found to be a mixed failure indicating а reliable zirconia-resin bond strength: nevertheless, large remnants of resin cement were observed in group ZC indicating that it was the most efficient decontamination method. A possible explanation for the incidence of the adhesive and mixed failures could be attributed to the adhesive interface between the resin cement and the zirconia surface which was decontaminated with different decontamination methods.

limitations of this study included that the specimens were stored in water for 24 h which is not enough for water absorption by resin cements; thus, the present study provides information about the initial zirconia-resin bond strength. In addition, this study evaluated only the bond strength of zirconia to resin cement. Further studies to evaluate the resin cement shear bond strength to tooth structure including long-term water storage are required.

CONCLUSION

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

1. Saliva contamination during clinical evaluation significantly decreases the shear bond strength between adhesive resin cement and translucent monolithic zirconia.

2. The decontamination methods improved the bond strength between adhesive resin cement and translucent monolithic zirconia, though not statistically significant.

3. Aging adversely affected the shear bond strength between adhesive resin cement and translucent monolithic zirconia.

4. Owing to their acceptable bond strength, translucent monolithic zirconia appears to be a promising monolithic restoration.

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