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The Effects of 2 Types of Cements and 2 Types of Surface Treatments on Shear Bond Strength of Zirconia

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الخلاصة

الأهداف: تهدف الدراسة الى تقييم التأثير المشترك لأنواع مختلفة من اللواصق و معاملات مختلفة للأسطح على قوة الربط لمادة الزركونيوم. **المواد وطرائق العمل**: تم اعداد ٣٠ عينة من مادة الزركونيوم ب(قطر ٤ملم و سمك ٤ ملم) .ولقد تم صقل جميع العينات بورق الصقل ذو (٦٠٠)درجة بوجود الماء.ولقد تم تنظيف الأسطح المصقولة بمادة الأسيتون لمدة (١٥)دقيقة المجموعة الاولى:وهي المجموعة القياسية لم يتم معاملتها ، فقط الصقل. المجموعة الثانية: لقد تم معاملة اسطح العينات بـ(اوكسيد الألمنيوم) المجموعة الثالثة: لقد تم معاملة اسطح العينات بـ(اوكسيد الألمنيوم+سلاين) لقد تم تقسيم كل مجموعة الى مجموعتين تبعا لنوعية اللواصق المستخدمة.وهما نوعين من اللواصق هما اسمنت الراتنج الذاتي اللصق واسمنت الزجاج المدعوم . لقدتم استخدام هذه اللواصق للصق عينة الزركونيوم بعينة (الكومبزت)التي استخدمت لهذه الدراسة وكل زوج من قالب الزركونيوم مع قالب الكومبزت عرض لثقل (١٥)كيلو غرام لمدة (١٥) ثانية وضعت جميع العينات بالماء بدرجة٣٧ لمدة ٢٤ ساعة. اختبرت العينات بماكنة الاختبار العالمية وفَحصَ نمط الفشلِ باستخدام ال stereomicroscope **الاستنتاجات**: العينات التي تم معاملتها بـ(اسمنت الراتنج الذاتي اللصق) اظهرت نتائج افضل من العينات التي تم معالجتها (اسمنت الزجاج المدعوم)٠والعينات التي تم معالجة اسطحها (اوكسيد الالمنيوم) ومن ثم معالجتها (اسمنت الراتنج الذاتي اللصق) اظهرت نتائج افضل على قوة التلاصق من العينات التي تم معالجتها بـ(اوكسيد الالمنيوم)و(سلاين)ومن ثم (اسمنت الزجاج المدعوم)) اظهرت نتائج اقل على قوة التلاصق.

ABSTRACT

Aim: The aim of this study is to evaluate the interactive influence of different types of cement and surface treatments on bond strength of zirconia**. Materials and Methods**: Thirty cylindrical zirconia blocks (Diameter4mm& thickness4mm)were fabricated for the study. All the specimens were ground with 600 grit silicone carbide polishing paper. Zirconia specimens were randomly divided into three main groups. 1stcontrol (600grit) no surface treatment. 2ed the specimens were air-abrasion with 50um aluminum oxide particles (Al_2O_3). 3 rd the specimens were air-abrasion with 50um aluminum oxide particles (Al_2O_3)+ silane .Then ,each group was divided into two groups according to the luting cement used which are(Reinforcd glass ionomer cement and dual cure resin cement).The zirconia blocks bonded to the composite blocks using these types of cements. Then each pair of zirconia block and composite was applied to load of(15 Kg) for 15 minutes in order to standardized the applied pressure for each type of cement . The resin cement was then light polymerized for 30s with LED light at 500mW/cm2 (Ledition, Ivoclar Vivadent, Liechtenstien) at the distance of 1mm from each direction. The specimens were stored for 24 hours at 37°C. Shear bond strength was measured by using Universal Testing Machine, and mode of failure examined by a stereomicroscope. **Results**: All the zirconia blocks were bonded to the composite blocks by self adhesive resin cement revealed results more preferable than zirconia blocks were bonded to the composite blocks by Reinforced glass ionomer cement. The shear bond strength between Zirconia and self-adhesive resin cement for the surface were treated with Aluminum-oxide air abraded alone, and zirconia surface were treated with Aluminum-oxide air abraded + silane more preferable than shear bond strength cement between Zirconia and Rinforeced glass ionomer cement for the surface were treated with Aluminum-oxide air abraded alone, and zirconia surface were treated with Aluminum-oxide air abraded + silane**. Conclusion**: Zirconia was more durable with Self-adhesive resin cement than Reinforced glass ionomer cement. Zirconia Surface treatments were very critical for improvement bond strength with cement in addition to the properties of that cement .

Key words: Zirconia, bond strength ,self-adhesive resin cement, air-abrasion, silane.

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INTRODUCTION

The high mechanical properties, chemical stability, and biocompatibility make zirconia an attractive core material for fabrication of all-ceramic restorations.[1,2] The interest in using high-strength zirconium oxide ceramics for oral rehabilitation has been growing in recent years.[3,4] These high-strength ceramics offer a wide variety of clinical applications, such as fixed-partial dentures (FPDs), posts or implant abutments in prosthetic dentistry. Zirconia has the most favorable properties, with a flexural strength of 900t-1200MPa, fracture resistance of more than 2000 N, and fracture toughness of 9-10MPam0.5, which is almost twice that of alumina-based materials.[5] Recently, a new zirconia ceramic (ICE Zirconia, ZirkonZahn) has been introduced in the dental market. According to its manufacturer, this zirconia ceramic has bending strength over 1,400 MPa and could be used for fabrication of 16-unit fixed dental prosthesis. [6] It is reported that zirconium oxide restorations, due to their high fracture resistance, can be cemented using conventional luting agents.[7] However, microleakage caused by inadequate marginal seal often leads to poor prognosis including dislodgement of restorations, secondary caries, and discoloration of cervical area.[8] For restorations to function for a long time, it is very important to clarify the adhesive behaviors at the interface between the hybrid layer formed on the surface of dentin-pulp complex and resin luting agents, as well as between zirconium oxide ceramics and resin luting agents.[9]

Recently, so-called universal, all-purpose, or multipurpose self-adhesive resin cements have been available, each purportedly bonding to enamel, dentin, amalgam, metal, and porcelain.[10,11] Self-adhesive resin cements that been proposed for luting zirconia-based restorations.[12,13] The resin matrix of these self-adhesive resin cements consists of multi-functional acid methacrylates.[14] If a high content of acidic functional monomers can react with the substrate and achieve enough chemical bond strength.[14]

Several protocols to provide a predictable bond to zirconia have been tested . One technique is to sandblast the internal surface of the restoration with aluminum oxide before cementation.[13,15] The air-borne-particle abrasion is an alternative method for roughening.[15] It has been reported that applying a sandblast, combined with a phosphate success of bonding to zirconia, as it enhances the acidic functional monomers capability of chemically reacting with the substrate.[7]

In addition, it has been shown that the use of a modified silane/bonding agent used with a modified resin cement will predictably increase the bond to zirconia.[16,17] According to previous studies[18-20] the association of resin luting cements with primers promoted a better interaction with the zirconia surface due to the increase in cement wetting. This wetting favors the adhesion process and improves the chemical interaction between resin cement and the zirconia surface. These ceramic primers usually contain silane and a functional phosphated monomer. .The interaction of the primer with the substrate and resin cement is promoted, forming cross links with the OH of cement methacrylates.

The null hypotheses of the study were:

(1)) Would reinforced glass ionomer cement provide a durable bonding to this zirconia compared to self adhesive resin cement?

(2) A durable bonding to the zirconia would be achieved regardless of the surface treatments.

(3)The effect of ceramic primer(silane) on the bond between luting agent and zirconia.

Data Analysis

Data were analyzed using t–test, Fisher Freeman–Helman test, Duncan's Multiple Range analysis and Kappa test.

MATERIALS AND METHODS

Specimens preparation:

The materials used in this study are listed in (Table 1). Thirty cylindrical zirconia blocks (Diameter 4mm & thickness 4mm) 5.03% (by weight) $Y2O3$ - 94.67% (by weight) $ZrO2$, were fabricated according to the manufacture construction .Each surface of the specimen was polished manually for one minute in one direction with 600grit Silicone carbide polishing paper (AL ALamain GHALB K.S.A). Polished surface was cleaned in acetone for 15 minutes each to remove factors that inhibit adhesion, then dried naturally in the atmosphere. Composite blocks were prepared from Te-Econom plus restorative composite resin (Ivoclar Vivadent AG schan⁄ Liechtenstien) using a mold with central hole (5mm) in diameter and (5mm) in height. Composite resin was applied in increments 1-2 mm thickness and carefully condensed by instrument with plastic working end and intensity of irradiation 500mW/cm2 (Ledition, Ivoclar Vivadent , Liechtenstien) for 40 s at 5 mm distance.

Table (1): Materials used in this study.

Material	Batch No.	Manufacturer
zirconia(zircon zahn)	Certified according to ISO 9001:2008 and ISO 13485:2993	The European Medical Device
Reinforced Glass ionomer luting cement	1101121	JAPAN
self adhesive resin cement	1200000669	(Illinois,USA) Bis-cem(Bisco)
Silane coupling agent	1100010235	(USA) BISCO

Surface Treatment before Bonding:

Thirty Zirconia blocks were randomly divided into three main groups (n=10)for each group, according to the different mechanical and/or chemical treatment performed:

Group 1: 600 grit(control groups)no surface treatment.

Group 2:Sandblasting(Figure 1)(bio.art Rua Teotônio Vileia, 120- Jd. Tangará-CEP 13568-000- São Carios- SP-Brasil.) the specimens were air-abraded with alumina particles 50-µm applied perpendicular to the surface at 3.0 bar from a distance of 10 mm for 10 seconds in circling movements(Figure 2).

Group 3 : Sandblasting + silanization. After sandblasting with alumina particles 50-µm ,coupling agent was applied ,mix the BIS-SILANE by dispensing one drop from each of the two bottles (parts A&B)into a mixing well .Brush on 1-2 coats and wait for 30 s. Then, the specimens were divided into two subgroups (n=5) according to the cement types(self adhesive resin cement and Rinforced glass ionomer cement).

Figure (1): Microblast.

Figure (2):Represent surface operated with microblaster.

Bonding Procedure

After appropriate surface treatment, each cement was applied according to the manufacturers' instructions at room temperature (23.0 ± 1.0℃) and relative humidity (50% ± 5%). The zirconia block bonded to the composite block under the load of (15 kg) for 15 minutes in order to standardize the applied pressure. The excess resin cement was removed by means of a laboratory knife. The resin cement was then light polymerized for 30s with LED light at 500mW/cm2(Ledition, Ivoclar Vivadent, Liechtenstien) at the distance of 1mm from each direction. Luted specimens were stored in distilled water at 37°C for 24 hours before testing shear bond strength.

Shear Bond Test:

Figure 3 shows the shear bond testing machine, which is present in mechanic engineer collage.(Soil Test Co. Inc., USA) with a Knife edge head placed at the interface between zircon block and composite block at a cross head speed of 0.5mm/min (21).The force at separation (N) was divided by the cross section area (100mm2)to provide results in units of stress(MPa). The failure e mode examined by a stereomicroscope (Zeiss, MC 63A, Germany) at 20X magnification power.

Figure (3): Universal Testing Machine (Soil Test Co. Inc.,ILL. USA)

Statistical Analyses:

The mean values of each group were statistically analyzed using one way analysis of variance (ANOVA)) followed by Duncan's Multiple Range Test at a significance level of $p<0.001$.

RESULTS

Means and standard deviation of shear bond strength values for both luting agent and surface treatments were shown in Table (2). Analysis of variance of each cement revealed a highly statistically significant difference in shear bond strength value among the groups ($p<$ 0.001). ANOVA result between cements was shown in Table(3).

Analysis of variance within the same and between the groups revealed statistically difference among the groups($p<0.001$).ANOVA result were shown in Table (4). Duncan's Multiple

Range Test to identify statistically the effects of surface treatments and cement types on shear bond strength of zirconia ,test was indicate that no significantly different between the first three groups (a1b1,a1b2,a1b3) that to say the groups related to the reinforced glass ionomer cement. While highly significant different between second three groups (a2b1. a2b2, a2b3), that to say the groups related to the self adhesive resin cement were shown in Table(5). Figure 4: demonstrates the effects of cement types and surface treatments on bond strength of zirconia.

Table (6) represent failures mode.The failures mode were mixed and adhesive within the self adhesive resin cement for all groups, while was cohesive within the reinforced glass ionomer cement groups.

Table(2):Mean and SD for Reinforced glass ionomer cement and Self adhesive resin cement.

Materials	**control**	**Sandbast (AL2O3)**	**Sandblast(AL2O3)┼Silane**
Reinforced glass Ionomer cement	0.1820(04604) a1b1	0.2680(006535) a1b2	0.3760(.6189) a1b3
Self adhesive Resin cement	1.0320(.05404) a2b1	2.2280(.29064) a2b2	2.8920(0.36996) a2b3

Table (3): ANOVA between cement.

	SUM OFSquares	**df**	**Mean SQUARE**	**f**	**sig**
Between Groups	23.639	1	23.639	66.740	.000
Within Groups	9.917	28	.354		
Total	33.556	29			

Table(4): ANOVA within the same and between the groups.

	SUM OF Squares	**df**	**Mean SQUARE**	**f**	**sig**
Between Groups	32.618	5	6.524	166.929	.000
Within Groups	.938	24			
Total	33.556	29			

Table (5): Duncan's New Multiple Range Test for variables

V VAR00011	**N**	**Subset for alpha=.05**			
		1	2	3	4
1.00	5	.1820			
2.00	5	.2680			
3.00	5	.3760			
4.00	5		1.0320		
5.00	5			2.2280	
6.00	5				2.8920
Sig		.155	1.000	1.000	1.000

Table(6): Type of failure between cements and zirconia

Cements types	Control	Sandblast with AL2O3	Sandblast with AL2O3┼Silane
RGIC	a1b1(I)5*	a1b2(I)5*	a1b3(I)5*
Self adhesive resin cement	a2b1(II)5*	a2b2(II)2*(III)3*	a2b3(III)5*

RGIC: Reinforced glass ionomer cement.AL2O3: Aluminum oxide air abrasive.a1b1:Reinforced glass ionomer cement with surface polishing by silicon carbide paper.a1b2: Reinforced glassionome cement┼+sandblast with Aluminum oxide.a1b3: Reinforced glass ionomer cement+ sandblast with Aluminum oxide┼Silane.a2b1: Self adhesive resin cement with surface polishing by silicon carbide paper.a2b2: Self adhesive resin cement+ sandblast with Aluminum oxide.a2b3: Self adhesive resin cement+ sandblast with Aluminum oxide +Silane.I: Cohesive failure. :II: Mixed failure .III :Adhesive failure.*:Number of specimens.

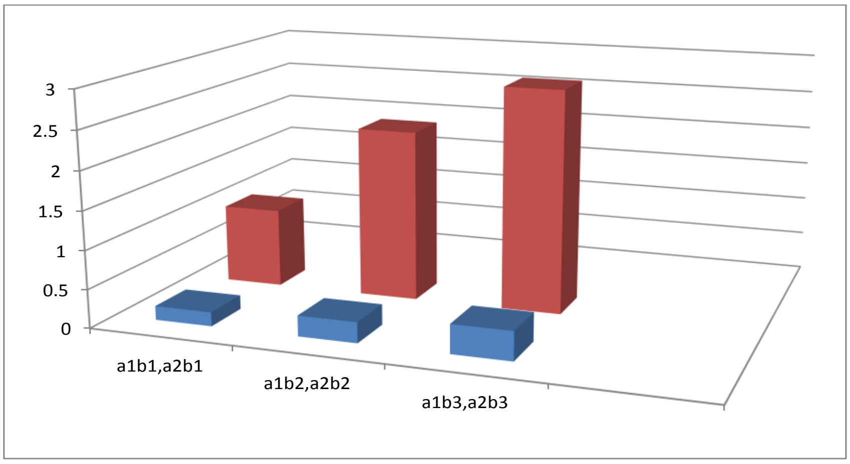

Figure 4: demonstrates the effects of cement types and surface treatments on bond strength of zirconia

a1b1:control group(polishing┼RGIC)

a2b1:control group(polishing┼Resin cement)

a1b2:RGIC┼AL2O3

a2b2:Resin cement┼AL2O3

a1b3:RGIC┼AL2O3┼SILANE

a2b3:Resin cement┼AL2O3┼SILANE

DISCUSSION

The surface hardness of zirconia is so high that is difficult to create grooves and micro retention for luting agents for that reason many studies were investigated to improve retention. (12,13,17) In this study, shear bond test has been used, a commonly used bond strength test, fast and easy to perform and also reflecting the clinical situation. However, the study did not intend to find the absolute bond strength values but to evaluate if the used pretreatments showed dramatic difference in bond strength.

When comparing the bond strengths of reinforced glass ionomer cements with self adhesive resin cement, reinforced glass ionomer cements revealed lower bond strength values than self adhesive resin cement. As seen in Fig. (4). The bond strengths of reinforced glass ionomer cements did not exceed (.3760 MPa).The cause related that reinforced glass ionomer cement was contained only 10 wt% HEMA. This was a relatively smaller HEMA content compared with 20－30 wt% in conjunction with Phosphate acidic monomer for self adhesive resin cement., the more functional monomers the more ability to form chemical bonds with the metal oxides of zirconia, secondary bonds such as van der Waals, or hydrogen bonds at the zirconia-resin interface, increasing the surface wet ability. [20] This increase in bond strength is more evident when functional monomers are used. Therefore, future studies should include these issues for investigation. So, the first hypothesis was rejected.

The failure mode for all the specimens were related to reinforced glass ionomer cement revealed cohesive failure Table, [6] while the specimens were related to self adhesive resin cement revealed mixed failures. This point explain what was mentioned previously, due to the weak bond between zirconia and Reinforced glass ionomer cement ,while more durable between zirconia and Self-adhesive resin cement.

In Table (2) Self adhesive resin cements were revealed bond strength values higher for sandblasted specimens (2.2280Mpa) than for #600-polished specimens(1.0320Mpa).On the other hand, with reinforced glass ionomer cements, no significant differences were found between sandblasted specimens and #600-polished specimens, which was consistent with the results of Uo *et al*.[22]

Sandblasting is an effective technique to improve bond strength by producing rough irregular surfaces with increased surface area necessary for micromechanical bonding. [13,15] Phase transformation of zirconia from tetragonal phase to the monoclinic phase is associated with volume expansion. This volume expansion stemming from the transformation causes a crack sealing compressive stress, which is why zirconium exhibits relatively high fracture toughness and strength, as well as the ability to resist crack propagation. On this ground, sandblasting is thought to be effective for zirconium oxide ceramics. [23] The results of this study are in agreement with findings of several studies, [18,24.25] which reported that airborne-particle abrasion produced an activated micro roughened zirconia surface, increased the bonding area and modifying the surface energy and wetability.

The failure mode as show in Table (6) more than >75 specimens surface show adhesive failure in comparing to the control groups less than< 75 specimens surface.

In this study the use of surface treatment in conjugation with primer(silane) in order to enhance bond strength between zirconia and cement. In Table (2)bond strength for sandblasting ┼silane groups reveled higher than bond strength for sandblasting groups alone. The cause related to the combined action of the two materials(silane and resin cement) would increase the presence of phosphate radicals ,and could lead to better bond between the zirconia and resin cement.

The majority of studies have demonstrated that chemical or mechanical modification was shown to positively influence bond strength to resin luting cements.[26-28] However the increase in bond strength was not always achieved, with contradictory results being shown.[29-31] The combination factors, such as the use of resin cements containing adhesive phosphate monomers, airborne particle abrasion pressure and primers , could promote a durable long-term bond to zirconia.[28,32] The use of primers without alumina airborne particle abrasion resulted in no long-term interlocking with the zirconia surface.[28,33]

CONCLUSION

Within the limitations of the present in vitro study the following conclusions can be drawn:

(1)The use of self-adhesive resin cements were mandatory with zirconia.

(2)The surface treatments improves bond strength between self-adhesive resin cement and zirconia.

(3) More improvement in bond strength between zirconia and self-adhesive resin cement will happen when using silane with surface treatment.

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