EFFECT OF THERMOCYCLING ON REPAIRED ZIRCONIA CERAMIC RESTORATIONS USING DIFFERENT BONDING AGENTS

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ABSTRACT

Statement of the Problem: The clinical success of zirconia intra-oral repair depends on the strength of the bond between the zirconia core and veneering composite.

Purpose: In the present study, the effect of aging on the shear bond strength of zirconia ceramic and nanohybrid composite resin as a repair material was evaluated using different bonding agents.

Materials and Method: Fifty six plates of zirconia ceramic (10mm×2mm) were prepared by CAD/CAM. All discs were air-abraded using 50μm Al_2O_3 particles at 2 bar pressure for 10 s, then divided into two equal groups A and B (n= 28) according to exposure to thermocycling. Each group was subdivided into four equal subgroups (n=7) according to the used bonding agent, as follows: control subgroup I, treated with Z-prime plus; subgroup II, All- Bond Universal; subgroup III, optibond; and subgroup IV, Voco-Futura bond. Surface roughness of all specimens (n=56) were evaluated immediately after bonding application using contact surf-test (Mitutoyo SJ 201, Japan). The composite resin (Ceram X nanohybrid) were then bonded and cured to each zirconia plate. The shear bond strength test was performed to group A with a universal testing machine at a crosshead speed of 1mm/ min before thermocycling. Specimens of group B were thermocycled 1000 times in water between 5°C and 55°C, then the shear bond strength test was performed. Bond strength values (MPa) were calculated and analyzed with a two-way ANOVA. The failure mode of each specimen was evaluated under a stereomicroscope.

Results: The mean shear bond strengths before thermocycling were 13.22, 13.03, 7.02, and 6.69 (MPa) in subgroups IA, IIA, IIIA and IVA, respectively. After exposure to thermocycling the mean shear bond strengths were 13.47, 11.48, 0.00 and 0.00 (MPa) in subgroups IB, IIB, IIIB and IVB, respectively. The results showed that the shear bond strength were significantly higher in subgroups I and II than subgroups III and IV (p< 0.05). Chemical pre-treatment of zirconia with Z- Prime Plus revealed significant higher bond strength than the Optibond (subgroup III) and Voco bond (subgroup IV) before and after aging conditions.

Conclusion: Treatment with both Z-prime and All- Bond Universal resulted in higher bond strength values compared to the Optibond and Voco Futurabond. The use of Z-Prime Plus treatment in combination with air-abrasion procedure resulted in the highest bond strength value that is stable on aging.

KEYWORDS : Zirconia; bond strength; Primer; Composite; Intra-oral Repair; Bonding agents.

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INTRODUCTION

Lately, more patients prefer dental restorations that syndicates high strength and good natural esthetic qualities that is why ceramic materials are replacing metallic restorations in dentistry. [1] All ceramic restorations are characterized by their excellent esthetics, soft tissue biocompatibility and acceptable mechanical properties, zirconia is generally used as a single crown core material or as a fixed bridge.[2]

Zircon material is defined as a gem since ancient times. It is a white polymorph crystalline oxide of zirconium that is consisting of tetragonal, cubic and monoclinic phases at various ranges of temperature. The unique temperature dependent transformation from tetragonal to monoclinic phase of zirconia causes strain and a volumetric increase of 3%-4%. The addition of 2-3% mol yttrium oxide, partly stabilizes the tetragonal phase of zirconia (TZP) Tetragonal Zirconia Polycrystalline to prevent the propagation of cracks. This strengthening mechanism of zirconia is known as transformation toughening and it has been sometimes referred to as 'ceramic steel' due to this strengthening and toughening mechanism. [3]

Zirconia-based ceramics have improved mechanical resistance than other types as leucite or lithium disilicate ceramics, particularly when fixing posterior teeth.[4] It was also found that zirconia is excellent in terms of mechanical performance as strength, toughness and fatigue resistance when compared to alternate materials, on the other hand, poor adhesion to substrates can be confronted in zirconia dental applications which is a problem.[5]

Loss of retention and chipping of veneering porcelain are the highest commonly identified clinical problems of zirconia-based ceramics.[6] In many cases, poor retention and failure of restoration may be related to improper tooth preparation and unacceptable cement selection or cementation technique.[7]

The accurate adhesion between veneering ceramic and zirconia core is extremely vital for the success of all restorations. Previous study reported 25% fracture between zirconia core and veneering ceramic at 31 months.[8] While another study detected 13% failure at 3 years.[9] Fractures can occur due to; improper framework support, overloading, low fracture toughness of porcelain veneers , surface defects and fatigue. [10-12]

When fracture is encountered in ceramic veneers, the suitable option for solving this problem is the repair of restoration than a complete replacement. Restoration repair can be performed intraorally because this conservative method is less traumatic to the patient and more acceptable. Veneering a composite material to the zirconia framework is economic, can overcome the problem of crown removal, and reduce time consuming of lab work. Intra-oral systems for zirconia and ceramic repair have been introduced by some manufacturer. [13]

Numerous zirconia core surface treatments have been investigated in order to increase its surface activation for chemical adhesion and improve their bond strength with veneering ceramic or composite resin. [14, 15]

Common surface treatment procedures for zirconia and ceramics surface treatment involves; diamond bur abrasion, grinding, sandblasting with Al_2O_3, laser-treatment, acid-etching, silane application, or the usage of more than one procedure.[5] The most clinically recommended surface treatment for increasing surface roughness and mechanical interlocking to zirconia, is Al_2O_3 abrasion.[16, 17] Air abrasion is used to enlarge surface area, increase surface energy, improve wettability and adhesion of zirconia-based ceramics. Many studies have shown that comparable surface roughness patterns can be produced with aluminum oxide abrasion and silica coating processes. Air abrasion displays a critical role in phase transformation of tetragonal to monoclinic phase that increases flexural strength. [18, 19]

Chemical pretreatment methods are still being investigated, a study showed some micro-morphologic changes on the surface topography of zirconia after applying 9.5% hydrofluoric acid (HF) in 25°C for 24 h, 9.5% HF in 80°C, and 48% HF in 25°C and concluded that concentration and temperature of acid could affect the reaction rate.[20]

Another convenient method used for activation of zirconia ceramic surfaces is the application of different primers that has reasonable cost and simplified application. Primers that have the composition of 10-methacryloxydecyl dihydrogen phosphate (MDP) and phosphate monomers can improve the bond strength to zirconia. [21-23] Many researches [21, 24-26] reported that the combined use of MPD primer and air-abrasion can improve the bond strength to zirconia core. The air abrasion was found to improve resin bonding to zirconia mechanically, while the adhesive monomers are necessary for chemical bonding. [27]

Many studies concluded that these primers bonding agents not only can form a favorable bond with tooth structures, but also they can bond to other different substrates such as resins, metals and other indirect restorations, including zirconia ceramics and lithium disilicate. [28-30]

Before performing clinical studies, in-vitro studies should be undertaken to prove materials' applicability and performance, however in-vitro studies should replicate clinical conditions, [31] therefore thermal-cycling and long term water storage are important factors during in-vitro studies.[32, 33]

Bond strength measurement is used to assess the efficacy of an adhesive system and to subsequently predict its clinical implementation. There are two types of bond strength tests that are most commonly used; tensile and shear bond strength. Several researches have described that cohesive fractures are more frequently occurring with shear bond strength rather than adhesive fractures. [34] In clinical situations, cohesive failures are hardly detected in bonded restorations.

This research assessed the thermal-cycling effect on the shear bond-strength of composite resins as repair material to zirconia substrate using different bonding agents as a method for chemical activation of inert zirconia surface. The null hypothesis was that, there would be no significant difference in the shear bond strength values among the various groups tested.

MATERIALS AND METHODS

Materials used in the study and their composition are shown in Table 1.

For this in-vitro study, fifty six plates of zirconia (10 mm in length X 2 mm in thickness) were produced using CAD/CAM technology (Arum x5 400, Doowon Co., Ltd. Daejeon, South Korea). Specimens are then sintered at 1550°C according to a cycle recommended by the manufacturer in a sintering furnace (Luoyang, China). All specimens were air abraded using 50 μm Al_2O_3 particles at low pressure 2 bar [35] for 10 seconds, using a sandblasting apparatus (JNBP-2, Jianian Futong Medical Equipment Co. Ltd., Tianjin, China) followed by rinsing for 60 minutes with distilled water in an ultrasonic cleaner device (MCS, CD4820, Codyson, China) and drying with an air syringe. All specimens were divided into two equal groups (n= 28) group A without thermocycling and group B with thermocycling. Each group was subdivided into four subgroups (n=7) according to the used bonding technique; control subgroup I, treated with Z-prime plus; subgroup II, All- Bond Universal; subgroup III, Optibond; and subgroup IV, Voco-Futura bond.

TABLE (1) Materials utilized in this study

Material	Batch No	Composition	Manufacturer
Zirconia ceramic	5054089	Zirconium dioxide ZrO_2 3Y-TZP-A or 3Y-TZP	Nacera, Doceram medical ceramic Gmbh-Hesslingsweg 65-67, D-44309 Dortmund, Germany
Z-Prime Plus	1800001455	BPDM, Ethanol 75-85%, HEMA 5-10%, Bis-GMA 5-10%, MDP 1-5%, Proprietary (phosphate and carboxylate functional monomer)	Bisco Inc., Schaumburg, IL 60193,USA
All-Bond Universal	1800004472	BPDM, Ethanol 30-50%, HEMA 5-25%, Bis-GMA 20-50%, MDP 5-25%, water, initiator	Bisco Inc., Schaumburg, IL 60193, USA
Kerr, **OptiBond** ALL-IN-ONE. Self-Etch Dental Adhesive	6494038	**Monomers** - GPDM Self-etching adhesive monomer - Comonomers including mono- and difunctional methacrylate monomers • **Solvents** water, acetone and ethanol •**Photo-initiator** (CQ)-based photoinitiator system • **Fillers** three nano-sized fillers • **Fluoride-releasing fillers** – sodium hexafluorosilicate and ytterbium fluoride	Kerr Itaia Sri, Via Passanti, 332, Italy
VOCO Futura-bond DC	1742689	Acidic adhesive monomer, 2-hydroxyethyl methacrylate	Voco, America, Inc
Ceram.X Sphere TEC ™ One Nano hybrid composite, Dentsply	1801000949	Non-agglomerated barium glass fillers and ytterbium fluoride (≈0.6 μm), highly dispersed, methacrylic polysiloxane nano-particles.	EENTSPLY Detrey GmbH, Germany

Specimens preparation

Subgroup I (ZP) Control group, specimens were treated with Z-Prime Plus (Bisco, USA) primer according to manufacture recommendation by applying 1-2 coats of Z-Prime plus to zirconia surface, and then drying for 3-5 s with an air syringe.

Subgroup II (AB), specimens were treated with light-cured dental adhesive All-Bond Universal (Bisco, USA) primer as follows: 2 drops of All-Bond Universal were dispensed into a clean well, 1 coat was applied to zirconia surface and air dried to get rid of excess solvent, then light cured for 10 s.

Subgroup III (OB), specimens were treated with self-etching adhesive OptiBond All-In-One (Kerr, Italia) to zirconia surface according

to manufacturer's recommendations as follows: 2-3 drops of OptiBond All-In-One adhesive were dispensed into a clean well. A disposable applicator brush was used to apply adhesive to zirconia surface. Two coats of OptiBond were applied with a brushing motion for 20 s each. The adhesive was dried with gentle air for 5 s, then light cured for 10 s.

Subgroup IV (VB), specimens were treated with dual-cure universal adhesive VOCO- Futurabond (Voco, America, Inc.) a coat was applied and air dried for 5 s then light cured for 10 s.

Surface roughness measurements

Surface roughness of all specimens (n=56) were evaluated immediately after bonding application using contact surf-test. The average surface roughness

(Ra) of each group was examined using a profilometer (Surftest SJ210, Mitutoyo Corp., Kawasaki, Japan) according to the ISO 4287-1997. For this purpose, seven specimens were measured of each group and the stylus was placed in the middle of the specimen surface. Each specimen was scanned 5 times and the mean roughness parameter (Ra) was calculated in (μm). The tracing length was 8 mm, at a scanning speed 0.5 mm/s. The resolution of the recorded data was 0.01 μm.

Shear bond strength measurements

A custom- made Teflon split mold with 6 mm diameter and 4 mm thickness, was centralized on the zirconia surface. The composite resin (Ceram X Nano hybrid, Dentsply) was dispensed, inserted into the mold, condensed and light cured according to manufacturer's recommendation.

The bond-strength test (SBS) was performed on a universal testing machine (Instron Universal testing machine, model no. 3345, England) at a crosshead speed of 0.5 mm/min, data was presented using Computer software BlueHill Instron.

A knife edge chisel apparatus (5 mm blade length) was used to direct a parallel shearing load exactly at the composite resin /zirconia interface. Specimens of group B (n=28) were thermocycled 1000 times in water between 5°C and 55°C, water storage. The dwell time at each temperature was 30 seconds with a transfer time of 15 seconds between baths (Theromocycler, Robota, Alexandria, Egypt). The thermocycling conditions were based on the calculation of that 1000 thermocycles would simulate the situation during one year in the oral cavity.[37] After thermocycling, specimens were tempered to room temperature in a water bath then shear bond strength test was performed. SBS values were recorded in mega-Pascal (MPa).

The debonded zirconia specimens were carefully examined for failure modes (adhesive, cohesive or mixed) under a stereomicroscope (BS-3060C, China) at magnification of x40. The failure modes are classified as cohesive failure; that is referred to a complete fracture within the ceramic or within the composite resin, adhesive failure; which means fracture between the ceramic (or composite resin) and bonding agent, and mixed fracture; which indicates fracture involving two materials.

Statistical analysis

The collected data were studied using one way and 2-way ANOVA (Analysis of Variance test), when significant differences were found between the groups, Bonferroni test (post-hoc test) was applied. The level of significance was set at p-value < 0.05.

RESULTS

Surface roughness were evaluated immediately after bonding application using contact surf-test device The resultant average surface roughness values (Ra) expressed in μm were recorded and tabulated in **Table 2.** It was found that, Z prime subgroup showed a high statistical significant mean roughness value (2.010 μm) among all groups p=0.000. Optibond subgroup showed a statistical significant mean roughness value among all groups (1.088 μm), except with Voco-Futurabond p=0.188. Voco-futurabond showed a statistical non-significant mean roughness value (0.738 μm) with all groups, except with Z prime where p=0.000. All-Bond Universal showed a statistical non-significant mean roughness value (0.689 μm) with all groups, except with Z prime subgroup where p=0.000 and Optibond where p=0.011.

Means and standard deviations for shear bond strength (MPa) for all tested subgroups were recorded before and after thermocycling then tabulated as in **Table 3**. Two-way ANOVA showed that the shear bond strength were significantly higher in subgroups I and II compared to the other two subgroups (p< 0.05). Chemical pre-treatment of zirconia with Z- Prime Plus revealed significantly higher bond strength than the optibond (subgroup III) and Voco bond (subgroup IV) before and after aging.

The current results also revealed that there were no significant difference between the mean shear bond strength of subgroup IA (Z-prime) and subgroup IIA (All-bond universal) before thermocycling, the same results were observed between subgroup III (Optibond) and subgroup IV (Voco-Futura bond). There were no significant difference in subgroup II before and after thermocycling. There were no significant difference between subgroup IIIB and IVB

after aging. However it was found that, there were high significant difference in the mean and standard deviations of shear bond strength values before and after thermocycling in both subgroup III and IV.

In the present study, it was noticed that specimens with lower bond strength failed adhesively; at zirconia-composite resin interface. On the other hand, specimens with higher bond strength showed mixed failure modes.

TABLE (2) Showing the average of surface roughness values (Ra) expressed in Micrometer (μm) using one way ANOVA and post hoc test.

	Non-sintered Group	Sintered Group	Sandblasted Group	Z-Prime Subgroup I	All-bond Universal Subgroup II	Optibond Subgroup III	Voco-Futrabond Subgroup IV
Roughness in (μm)± (SD)	0.414[cd]± (0.124)	0.449[cd]± (0.049)	0.614[cd] ± (0.039)	2.010[a]± (0.555)	0.689[cd] ± (0.158)	1.088[bc] ± (0.290)	0.738[bcd] ± (0.126)
(Min-max)	0.271-0.650	0.395-0.531	0.544-0.693	0.916-2.756	0.463-0.958	0.638-.1.590	0.521-0.980
95% Confidence interval for mean	0.325-0.503	0.413-0.484	0.597-0.631	1.703-2.318	0.615-0.763	0.921-1.256	0.679-0.797

Similar superscripted letters denotes statistical non-significant at p= 0.05

TABLE (3) Showing means and standard deviations of shear bond strength of the different study groups before and after thermocycling using Two Way ANOVA

	No.	Z-prime	All Bond Universal	Optibond	Voco-Futurabond
Before thermocycling Group A	28	13.22[a] (0.45)	13.03[a] (1.66)	7.02[ab](1.56)	6.69[ab] (1.69)
After thermocycling Group B	28	13.47[a] (3.77)	11.48[a] (1.24)	0.00[b]	0.00[b]

Similar superscripted small letters denote statistically non-significant difference at p= 0.05

DISCUSSION

This study appraised the effect of aging on the shear bond strength (SBS) between zirconia core and Nano hybrid composite resin as a repair material using different bonding agents.

Clinical studies of yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramic

restorations showed that the common causes of failure were chipping and delamination of veneering ceramic.[37, 38] These failures can be a serious impediment for patients and clinicians. The veneers chipping can occur due to an inadequate micro-mechanical interlocking with a deficiency of chemical bonding at the interface and tensile stress on the surface of the veneer.[39, 40] The bonding

between ceramic veneers and zirconia core was found to be weakest feature of the restoration.[41] A reasonable micromechanical interlocking at the interface of zirconia core and its veneer, attained by increasing surface roughness, can improve their bond strength.[42] Surface treatments to YSZ substrate can improve bond strength with veneer ceramic and can be done by adding or removing materials from the surface. Chipping has many proposed repair techniques, but the direct repair of this kind of failure is significant [43], many repair systems utilizes a resin composite materials.[44] After veneer chipping, when the framework is bared, bonding between the composite resin repair material and zirconia core must be done successfully.[45]

The use of CAD/CAM technology system for soft machining zirconia blocks in our study is chosen to minimize the volumetric changes of zirconia.[46] In the current study, the new nano-hybrid composite (Ceram.x, Sphere TEC™) was chosen because of its high filler load that supports mechanical strength and reduces polymerization shrinkage. Maximization of filler load was able to be done by using new type of pre-polymerized filler primary particles (Sphere TEC) which are smaller than 1 μm. The SphereTEC fillers are virtually perfectly spherical and have a distinct, micro-structured surface that reduces the amount of resin needed in ceram.x and thus the stickiness of the paste to hand instruments. Sub-micron SphereTEC particles resulted in msuperior esthetics and wear resistance, as during finishing and polishing, the fillers are removed layer-by-layer leading to an even restoration surface after polishing.[47]

The results of this study rejected the null hypothesis, since various groups tested produced significantly different SBS values. Mechanical and chemical surface treatments methods have been utilized to increase bond strength between ceramic core material and resin composite. All tested groups were mechanically treated using an Al_2O_3 abrasion procedure, as this mechanical surface treatment improves bond strength between

the resin and zirconia by increasing surface area, surface roughness and wettability, thus resulting in improving of resin flow onto the zirconia surfaces. [24, 48] In addition, this procedure removes organic contaminants from the zirconia surface for chemical bonding. [27] Other researches have reported that air-abrasion process can produce micro-cracks in zirconia surface that can result deteriorating zirconia properties; [24,49] therefore, the zirconia in this current study were abraded at a low pressure of 2 bar, using a powder with a particle size of 50μm Al_2O_3 to avoid surface damage. [49, 50]

Results of some previous studies, showed that one of the most commonly used tests for evaluation of the bond strength is the shear bond strength test.[27,51]

In this study 1000 thermocycles were performed to simulate intra-oral environmental aging conditions. Group III and IV showed failure and separation of composite resin from zirconia after thermocycling occurred, this can be due to the effect of thermal stresses that might have decline the zirconia-composite resin bond because of the difference in the coefficient of thermal expansion and bond deterioration via hydrolysis.[52] Specimens preparation with machining technique for tensile bond strength testing can cause adhesive defects and resulted in stress-concentration at the zirconia interface and affect the bond strength values [49,53] but the shear test is more simple and does not require samples trimming; yet, the shear bond strength test has been questioned for nonhomogeneous stress distribution at the interface.[53]

Many research works found that the preeminent adhesion to zirconia ceramic can be acquired by exploiting primers containing a phosphate-based functional monomer, especially MDP; [23,25,49,51] hence in this study, two types of the primers were used that contained MDP monomer, and it seems that they are both equally effective regarding aged bond-strength. According to the results of this study, both Z-Prime Plus and All-Bond Universal

primers have higher bond strength that does not deteriorate with aging, other than Optibond and Voco-Futurabond that have significantly lower bond strength that deteriorates after aging.

Z-Prime Plus treatment group showed a high statistical significant mean roughness value (2.010 μm) among all subgroups p=0.000, this might be accredited to the large molecular size and nature of this material; as it chemically bonds to zirconia structure and its way of application; as it requires two coats. Z-Prime Plus group has also significantly higher bond strength values because of its chemical structure that contains conventional MDP and carboxylic monomers which can chemically react with the zirconia oxide layer at the interface.[54] The interfacial forces might improve the wettability and chemical bonding to zirconia ceramics. [24,54] In addition, MDP has an amphiphilic construction; the vinyl group, as the hydrophobic end, can copolymerize with the resin monomer and the phosphate group as the hydrophilic end can interact with the hydroxyl groups on the zirconia surface, improving the chemical affinity. [21, 55] It seems that the synergistic effect between acidic MDP and carboxylic monomer is the most likely reason for having the maximum bond strength values. [29] The results of this current study were in accordance with Zandparsa et al. [23] and Shin et al. [25] who reported that the combined use of Z-Prime Plus primer and air abrasion improved the bond strength of zirconia ceramic to resin cements. It was also noted that Z-prime Plus group has a higher shear bond strength after thermocycling, although non-significant, than before thermocyling, this might happened due to an improved polymerization process in the Z-prime that occurred by thermal stresses of thermocyling.

In this study, All-Bond Universal group showed a high shear bond strength value that does not significantly differ than Z-prime group, although it has statically significant lower roughness value than Z-prime group, the similarity of bond strength value might be attributed to the higher percentage of MDP in All-Bond (5-25%) as compared to Z-prime

(1-5%), MDP. This higher concentration may have intensified the chemical bonding between composite and zirconia surface in the All Bond Universal group, and stabilized it over thermocycling and aging conditions. On the other hand, Optibond subgroup showed a statistical significant mean roughness value among all subgroups (1.088 μm), except with Voco-Futurabond p=0.18. However, the both subgroups showed a significantly lower bond strength value as they do not contain MDP in their composition for chemical bond formation with composite resin.

In this study, it was observed that specimens with lower bond strength failed adhesively; at zirconia-composite resin interface as in Optibond and Voco bond specimens. On the other hand, specimens with higher bond strength values due to primer treatments, mixed failure modes were observed as in Z-prime and All-bond universal specimens. These results were in agreement with the studies of Seabra et al., (2014) [29] and Xie et al., (2016). [30] Cohesive failures within the zirconia specimens did not occur, which is one of the most important advantages for zirconia restorations because the intraoral repair of zirconia restorations is difficult and adequate bond cannot be achieved to repair zirconia with composite resin. [30]

CONCLUSION

Under the limits of the current study, it could be concluded that, the bond strength between zirconia core and Ceram-x composite resin was affected by the chemical surface treatment. Treatment with both primers (Z-Prime Plus and All-Bond Universal) resulted in higher more stable bond strength than the other two bonding agents (Optibond and Voco-Futrabond). Treating the chipped zirconia surface by either Z-Prime Plus or All-Bond Universal after Al_2O_3-abrasion process can enhance composite resin adhesion to zirconia ceramic as it resulted in a higher bond strength that is not prone to aging.

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