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Evaluation of repair bond strength with different methods for zirconia restorations

Rami M. Galal^{1,2*}, Ghada E. Hamza¹ and Menatallah M. ElHotieby^{1*}

Abstract

Background: The aim of the study is to assess the bonding of two repair methods for zirconia.

Null hypothesis: Repair of zirconia with Cojet (3M Cojet sand, 68,411) and clearfil (clearfil REPAIR Kuraray Japan) has the same effect on the bond strength of zirconia restorations. Zirconia blocks were sliced before sintering into 28 samples (inCorisZI mono L), surface treated with two repair methods (Cojet and clearfil) then bonded to composite. Storage and thermocycling was done then loaded under shear until failure. Shear bond strength was collected and statistically analyzed. Graph pad InStat (Graph Pad, Inc.) software for windows was used for analyzing results.

Results: It was found that COJ surface treatment recorded statistically non-significant higher shear bond strength mean value (8.45 ± 0.70 Mpa) than CLE surface treatment (7.67 ± 2.55 Mpa) as indicated by paired *t*-test.

Conclusion: It was concluded that bond strength between composite resin and inCoris ZI mono L is the same in resistance to shear stresses after Cojet surface treatment and after Clearfil surface treatment.

Keyword: Repair, Bond, Zirconia

Background

Due to limitations of metal ceramic restorations, all ceramic systems have been used Haidar and Masoud (2021).

With increasing desire about esthetics, zirconia prostheses have widely spread. These prostheses should fulfill certain mechanical needs for optimum durability comparable with traditional porcelain fused to metal prostheses with proper esthetical properties in the same time (Höland et al. 2008). However, fracture and de-bonding were two major defects with zirconia restorations especially with the cementing phase. Adhesion enhances strength with penetration of defects within the fitting surface with prevention of crack progression (White et al. 1994).

Some sort of fracturing of ceramics does not indicate full replacement of the prostheses, but there is a challenge for the operator esthetically and functionally to repair it (Sailer et al. 2006). Replacing of prostheses is not in all cases the ideal treatment option as it is expensive, there is difficulty of removing the restorations, there is more traumatic loss of hard tissues, more chair time and more patient's time loss (Sailer et al. 2006; Kelsey et al. 2000; Kim et al. 2005; Edelhoff et al. 2001). Instead of removing the restoration, the availability of an esthetic and functional intraoral repair option may provide a more practical solution, which enables the use of the restoration if it is in an acceptable condition.

Trials were done for development of easy, proper and cheap way to intra-orally solve the problem of fractures within veneering material of ceramic restorations. The way the ceramic is processed, prevents adding more material inside the patient's mouth (Ozcan 2003). Repairing methods has a classification of directly and indirectly. Indirectly, framework is modified, with cementation of new veneering part above (made by the technician). Which can be done with big fractures, at sites with great loads during function,

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and when esthetical results are very important. Nevertheless, it needs two visits; it is expensive, with possibility of inconvenience if the substructure is thin (Ozcan 2003; Hammond and Swift 2009).

So, according to how large is the fractured part, chair-side direct repairing with resins and adhesives is a simple alternative method offering several advantages as having a result immediately, less loss of time, cheaper, and being easier to apply. It was introduced in 1978 by Newburg and Pameijer, with improper results in the beginning due to instable silane agent using for preparing the site (Ozcan 2003).

For repairing using resins, a number of ceramic repair systems with a defined sequence of application for the products are commercially available. Old ceramic repairing kits depended on macro-mechanical retentive means, using a groove or undercut but results then were unsatisfactory because of esthetic and mechanical limitations. Nevertheless, recent systems mainly depend on bonding micromechanically and chemically to ceramic surface, which can be variously, treated using acid etching, sand-blasting, silica coating and silanization (Melo et al. 2007).

In this study, two intra-oral repair systems are going to be assessed with zirconia.

Methods

The Twenty-eight zirconia samples were used. Divided into two groups (I and II) according to type of repair system to be tested.

Group I: zirconia repair using Cojet intra-oral repair system (n = 14).

Group II: zirconia repair using Clearfil repair system (kit) (n = 14).

Materials used for zirconia repair are listed in Table 1.

Blocks of (inCoris ZI mono L) were used to prepare slices, which had 2 mm thickness after sintering of zirconia (Attia 2011). Slices were cut by an 0.5 mm thickness diamond disc (Buehler USA) attached to Isomet 4000 low speed precision sectioning saw (Attia 2011) at speed 2500 rpm and feeding rate 10 mm/min, under continuous water irrigation, finished using silicon carbide papers of 300 grits, cleaned for 180 s with distilled water in an ultrasonic cleaner and dried with oil-free air.

The slices of zirconia were placed on a firing tray and transferred to a special sintering furnace. The sintering was carried out in approximately 8 h as follows: the temperature

was increased from room temperature to 1500 °C within 3 h with heat rise rate of 8 °C/min; this temperature was held for 2 h, than the furnace started to cool down from 1500 °C to room temperature in 3 h. Sintered blocks were removed from the furnace. Finally, dimensions of the slices were verified to be 2 mm in thickness by digital caliper and then finished and cleaned. Before the surface treatment, the sintered inCoris ZI mono L block samples were divided into 2 groups according to the type of surface treatment and repair kit used.

Before any surface treatment was performed, each block slice was embedded in an acrylic block leaving one surface of the porcelain uncovered, for easier handling and fixation during shear bond testing as shown in Fig. 1. Surface treatment was performed according to the manufacturers' instructions for each step of repair kit. In order to standardize the shape and dimensions of the repair composite material over the ceramic samples, specially designed circular split Teflon molds are constructed as shown in Fig. 2.

Figure 1 removed and starting from this figure.



Fig. 1 Slices embedded in the acrylic blocks

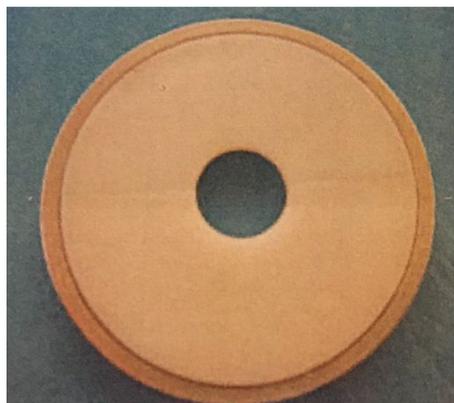


Fig. 2 Teflon mold with the outer stabilizing ring

Table 1 Materials used for zirconia repair

Group	Material used for repair
Group I (n = 14)	Cojet intraoral repair system
Group II (n = 14)	Clearfil repair system kit

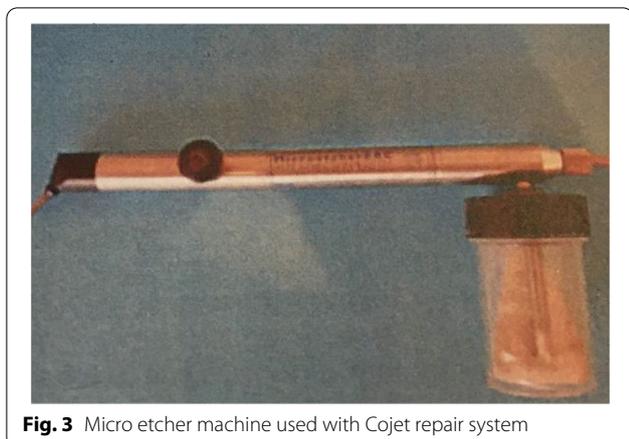


Fig. 3 Micro etcher machine used with Cojet repair system



Fig. 4 Application of composite using Teflon mold and metallic ring

It consists of an inner split Teflon mold with a central hole having an inner diameter of 5 mm and a height of 2 mm (Attia 2011) assembled by an outer stabilizing metal ring. Figure 3 shows the Teflon mold with the outer stabilizing ring.

The blast-coating agent container of the micro etcher unit was cleaned and dried. The air pressure was set to 30–45 psi to ensure that the energy of impact is sufficient for successful coating. The micro etcher unit was filled with a Cojet sand; which is a specially developed 30- μm aluminum oxide grains coated with silicone dioxide, and then tested on a working sample (Fig. 3). Micro blasting was done from a distance of 7–10 mm and perpendicular to the surface of the specimens. The entire surface of the specimen was coated evenly for 15 s. Any residual blast-coating agent was removed with a stream of dry oil-free air.

Directly after coating, ESPE Sil silane solution was placed into the dappen dish and applied using a clean brush to wet the entire surface of the ceramic blocks with the solution. The silane solution was left to dry for 5 min. Visio bond was introduced onto a mixing pad and applied using a disposable brush to form a thin layer of the material to the silanized surfaces of the blocks. The bonding agent was light-cured for 20 s. Z 100 composite resin (a visible-light activated, radiopaque composite with glass, colloidal silica, silica, Bis-GMa, TEGDMA and dicamphorquinone) was applied to ceramic surface according to manufacturer's instructions. The composite filling was placed over the specimens using a custom-made Teflon matrix of 5 mm internal diameter and 2 mm in thickness (Fig. 4). The Teflon matrix was placed on the surface of the specimens using the holding ring (Fig. 4). The composite resin was then light-cured with visible light at 500 mW/cm^2 intensity and 5 mm away from the specimen for 40 s. Figure 4 shows the application of composite using Teflon mold and metallic ring.

Airborne-particle abrasion was performed for 20 s with an airborne-particle abrasive unit of 50 μm aluminum oxide particle size at a pressure of 2.5 bars and a distance of 10 mm from specimen surface, then the specimens were rinsed for 10 s using air water spray and dried using oil/water free compressed air. Phosphoric acid etching gel (K-etchant gel) was applied on ceramic blocks surface after sandblasting and left in place for 5 s before washing and drying with oil-free air.

One drop of Clearfil SE bond primer (self-etching primer containing phosphate monomer MDP) and Clearfil porcelain bond activator (silane coupling agent) was dispensed into the well of the mixing dish supplied by the manufacturer and mixed immediately before application. The mixture was applied to the ceramic surfaces with a disposable brush tip and left in place for 5 s. After application, the volatile ingredients were evaporated using a mild oil-free air stream. Sufficient dryness of treated porcelain surface was performed to avoid impaired adhesion.

The required amount of Clearfil SE Bond; formed of phosphate monomer (MDP), Methacrylate monomer, Hydroxyethyl Methacrylate (HEMA) and small amount of inorganic filler was dispensed into the well of the mixing dish. The bonding agent was applied to the entire specimens' surface with a disposable brush tip. Light air stream was used to make the bond film as uniform as possible. The bonding agent was light cured for 10 s using a visible light curing activator.

Clearfil AP-X (light cured, radiopaque) composite resin was used. The principle ingredients of this type of composite are silanated barium glass, colloidal silica, silica, Bis-GMa, TEGDMA and di-camphorquinone. The composite filling was placed over the specimens using the previously mentioned Teflon mold and holding ring. The composite was then light-cured for 40 s at 500 mW/cm^2 intensity and at 5 mm distance away from the specimens.

All specimens were stored in distilled water for 7 days before thermocycling. Thermocycling was done between 5 and 55 °C for 7500 cycles (Blum et al. 2012) with a 30-s dwell time in a thermocycler (thermocycler mechatronic) (Fig. 5).

A circular interface shear test was designed to evaluate the bond strength. All samples were individually mounted on a computer-controlled materials testing machine with a load cell of 5 KN and data were recorded using computer software. Samples were secured to the lower fixed compartment of testing machine by tightening screws through Teflon custom made housing device with central cavity into which the ceramic plate fit (dimensions; 14 × 12 × 2 mm). Shearing test was done by compressive mode of load applied at ceramic-composite interface using a mono-beveled chisel shaped metallic rod attached to the upper movable compartment of testing machine travelling at cross-head speed of 0.5 mm/min. The load required to debonding was recorded in Newton. The load at failure was divided by bonding area to express the bond strength in Mpa: $\tau = p/\pi r^2$.

Where; τ = shear bond strength (Mpa), p = load at failure (N), $\pi = 3.14$ and r = radius of disc (mm).

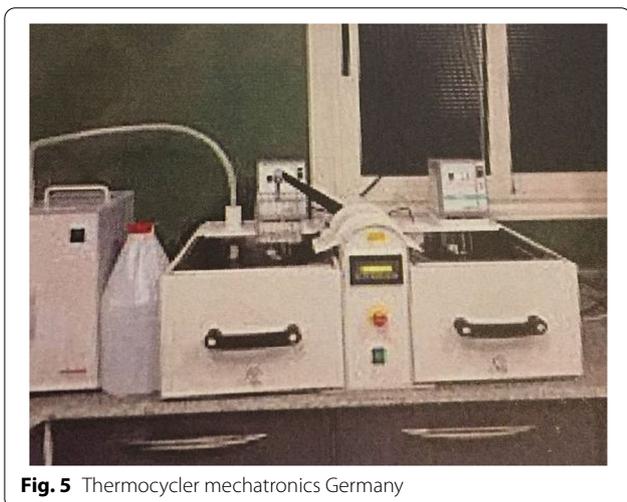


Fig. 5 Thermocycler mechatronics Germany

Results

Analysis was done using graph pad InStat (graph pad, Inc.) software. Value of $p \leq 0.05$ is considered significant. Homogeneity of variance and errors normal distribution confirmation was done; a 2-way analysis of variance (ANOVA) was performed.

Descriptive statistics of shear bond strength (Mpa) to show mean, standard deviation (SD), minimum, maximum and 95% confidence intervals (low and high) values of different surface treatments are shown in Table 2 with graph shown in Fig. 6. CO surface treatment recorded statistically non-significant higher shear bond strength mean value (8.45 ± 0.7 Mpa) than CL surface treatment (7.67 ± 2.55 Mpa) as indicated by paired t -test ($p = 0.4 > 0.05$).

Discussion

Recently, it is a point of concern to study repair of prostheses. Although there is great advances in the ceramics but fractures and chippings are common problems. Therefore, the need for repair is a demand. The choice of the repair system is a point of concern for the clinicians (Blum et al. 2012). In vitro results can give an indicator to the in vivo clinical situation regarding the performance of certain material. Zirconia is a polycrystalline dioxide of zirconium. Polycrystal containing porcelains without glass; atoms are compacted regularly (crystal arrays) so, difficultly crack could be derived than in low density and irregularly shaped networking glass materials. Polycrystal

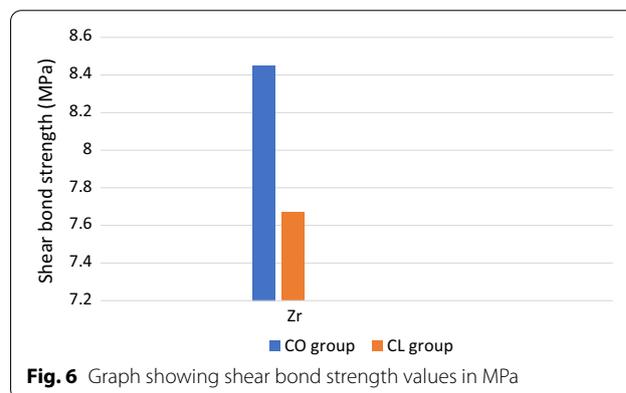


Fig. 6 Graph showing shear bond strength values in MPa

Table 2 This Descriptive statistics of shear bond strength results (mean values SDs) for both groups

Variables	Mean ± SD	Min	Max	95%		Statistics P value
				Low	High	
Zr CO group	8.45 ± 0.7	7.4	10.6	7.8	9.1	0.45 Ns
Zr CL group	7.67 ± 2.55	4.6	13.3	5.4	10.1	

Ns: Non-significant ($p > 0.05$)

reinforced materials have more toughness and strength than glass matrix materials (Kelly and Denry 2008). Adhesive cementation of ceramics makes removing them to be indirectly repaired with causing no harm for natural tissues and prosthesis uneasy. Repairing using resin materials can be applied with restoring appearance and functional needs with cheap, conservative and fast method (Hammond and Swift 2009).

Effectiveness of adhesive way depends on surface quality of the adhering site and its surface energy. Changing the surface quality by chemical preparation can change its area and surface energy and the adhesive potential, so enhances the micromechanical retention of the resin. Adhesion mechanically can be improved with site by roughening, grinding using abrasive stone, airborne-particles abrasion using aluminum oxide particles, etching with several acidic agents and combination of any of these techniques. For effective composite porcelain bonding, bonding micromechanically and chemically is necessary. Silane solutions act bifunctionally improving the wetting and the surface energy of the ceramic surface allowing a bond between ceramic silica and organic part of the resin by covalent bonds (siloxane bonds) (Thompson et al. 2011). Silica is not found in zirconia, so tribochemical application of a silica layer by means of air borne particle abrasion (Cojet system) followed by silane application was found to provide strong resin-ceramic bond (Kim et al. 2005). Also, phosphate ester group contained in some adhesives can bond in a direct way with metal oxides with possible another bond technique to zirconia (Oyague et al. 2009). In this research, two repairing methods relying on two conditioning ways have been tested.

Cojet system used air-abrasion with Cojet sands (CJ) and Clearfil system used air-abrasion with Al_2O_3 (CR). To ensure strong bond, the repair composite should have a minimal polymerization shrinkage and minimum coefficient of thermal expansion to reduce the stress that can strain the interfacial bond between the composite and the ceramic (Yoshida et al. 2007). Because of the Cojet sand blasting system sand's fine particle size (30 μ), the abrasion rate is much lower than with conventional abrasives. During the process of striking the surface with the particles, very high temperatures are produced and parts of the abrading material are incorporated within the surface down to 15 μ deep, as assumed by the manufacturer.

Phosphoric acid etching gel (K-etchant gel) was applied in the other group after air-borne abrasion with 50 μ alumina, followed by self-etching primer (Clearfil SE bond primer) containing phosphate monomer (MDP) and silane coupling agent (clearfil porcelain bond activator) mixed together. A light cured, radiopaque composite resin (clearfil AP-X) was used.

Shear bond strength testing was performed in this study because these stresses are believed to be the main reason for failure of the repaired veneer (Ozcan 2003).

Ozcan et al. (2009) found that Cojet system achieved the higher bond strength than Clearfil system both in dry and aged conditions. The superiority of the Cojet system reported by these studies might be due to silica coating leading to formation of a finer roughly formed site increasing the area via airborne particle abrasion and thus enhances the micromechanical bonding to resin. Those methods form silica particles on the site by high velocity introduction of aluminum oxide modified by silica particles (tribochemical silicoating), with formed bond chemically of the silica coated site with composite resin material, via silane coupling agent (3-methacryloxypropyl trimethoxysilane). Barutçigil et al. (2019) augmented that Cojet system had positive effect on bond strength of all ceramic systems.

While another recent study by Saleh et al. (2019), it was confirmed that adding agents containing MDP (as Clearfil) added to the formation of durable bond strength of resins to zirconia.

According to the results, it was found that for zirconia, Cojet surface treatment recorded non-significantly higher shear bond strength mean value than Clearfil surface treatment. This coincides with Lima et al. (2019) who found that control group with no chemical treatment (MDP agents) presented low surface free energy and subsequent less bond strength, this proved the positive effect of chemical treatment, while it is in contradiction with Khan et al. (Khan et al. 2017) who found in a review that tribochemical coating can be used alone and improved bonding as recommended by some studies.

The high results of Clearfil with zirconia may be explained as the special functional monomers have chemical affinity to metal oxide components with the possibility of including in resinous part of the cement and adhesive agents or put in a direct way on the ceramic surface. The phosphate ester monomers, such as 10-methacryloxydecyl-dihydrogenphosphate (MDP), makes chemical reaction with ZrO_2 , promoted a water-resistant bonding with dense sintered zirconia materials (Grasel et al. 2018), MDP-based resins are necessary to have proper adhesive bond with zirconia as stated by Kim et al. (2014), although some researches showed no bonding superiority than traditional BIS-GMA, containing resin cements (Cristoforides et al. 2012). As results are in some cases not significant, combining primers and abrasion techniques lead to formation of better bonding quality with more longevity as stated by Cheung et al. (2014).

Conclusions

So the use of the mechanical and chemical agents are the recommended for better bonding between zirconia and resins.

Abbreviations

COJ: Cojet; CLE: Clearfil; Clearfil S.E bond primer: Self etching primer containing phosphate monomer.

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Author contributions

"Conceptualization, "RG"; methodology, "GE"; validation, "ME"; formal analysis, "RG"; investigation; resources, "GE"; data curation, "ME"; writing—original draft preparation, "RG"; writing—review and editing, "GE"; supervision, "ME"; project administration, "RG"; funding acquisition. All authors have read and agreed to the published version of the manuscript.

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Availability of data and materials

Data and material are available.

Declarations

Ethics approval and consent to participate

No ethical approval was needed as the study was carried out according to the ethical guidelines of the World Medical Association (Declaration of Heliniski 1975) and was approved by the Ethics Committee at the National Research Centre (NRC) Cairo, Egypt.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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