Effect of Surface Treatments on Fracture Strength of Zirconia Core

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Abstract
Background The increasing of demand for all-ceramic restorations has led to the development of ceramic materials with optimized mechanical properties, such as densely sintered aluminum oxide and zirconium oxide (zirconia) ceramics. **Objectives** The purpose of this in vitro study was to evaluate the effect of etching with hydrofluoric acid or phosphoric acid and the effect of packable P60 composite veneer material on fracture strength of zirconia cores. **Materials and Methods** Twenty-four zirconium cores (Vita, Germany) with 0.7mm thickness were fabricated by CAD-CAM technology and sintered at 1450 ^oC for 60min according to the manufacturer's instructions. All zirconium cores were subjected to air abrasion with 50μm of Al_2O_3. These were randomly divided into three groups, (n=8). Group A: control group, veneered with IPS E-max Ceram porcelain; group B: etching with 37% of phosphoric acid and veneered with packable composite resin; and group C: etching with 9.5% of hydrofluoric acid and veneered with packable composite resin. All specimens were subjected to fracture strength test in an universal testing machine, a load was applied with 6mm diameter steel ball indenter at a crosshead speed of 0.5mm/min. **Results** One-way ANOVA test showed that there were statistically highly significant differences among all studiedgroups. LSD testwas performed to show thesource of significancy it showed a highly significant difference among all testedgroups. **Conclusion** A combination of surface treatment of 50μm Al_2O_3 and 37% concentration of phosphoric acid and adhesive agent could enhance the fracture strength of composite veneered crowns.

Keywords: Composite resin, fracture strength, surface treatment, sandblast, zirconia core.

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Introduction

Prosthodontic treatments aim to replace lost function speech, chewing, swallowing and aesthetic (Agustin et al, 2014). The use of metal-ceramic restorations in the fabrication of fixed partial dentures was for more than 40 years (Casson et al, 200; Tarib et al, 2016).

Because of the esthetic demand for dental restorations, metal systems were replaced by the all-ceramic system (Chintapalli et al., 2013). Hence zirconia ceramic has ideal properties, it has been recently introduced to restorative dentistry as a metal-free alternative (Kitayama et al, 2010; Korkmaz et al, 2015). With the development of CAD-CAM technology, the design and production of zirconia frameworks could be achieved using the digital process (Phark et al, 2009). Therefore, the all-ceramic restorations by the use of a zirconia core become more practical applications (Aboushelib et al, 2007; Ural et al, 2011). Such restorations have ceramic core replaced metal alloy and was veneered with ceramic (Guazzato et al, 2004). Zirconia core has the highest opacity; it requires veneering materials like feldspathic porcelain and these veneering materials mask its opacity and give it aesthetically appearance (Chen et al, 2008).

Fractured of veneering porcelain is the most common complication in Y-TZP based restorations (Rinke et al, 2011). It occurs during mastication as the fragments of porcelain mass was chipped (Fischer et al, 2008; Stawarczyk et al, 2011), ceramics under tensile strain are fragile (Borges et al, 2003).

Several factors could influence fractures of veneering ceramic-like overload at the premature contacts, ceramic strength and the adhesive bonding abilities; inappropriate framework design (Estevam et al, 2010; Fushiki et al, 2012); miss-match coefficient of thermal expansion; and excessive wear of the opposing teeth (Guazzato et al., 2005; Park et al, 2014).

When the zirconia core was veneered with a high strength composite resin material, it has been proposed as an alternative veneering method (Dhawan et al, 2003). Composite resins are widely used for direct restorations because they exhibited excellent, physical, optical, mechanical properties, and ease of handling (Hervas-Garciaet al, 2006). They exhibit viscoelastic effects, as well as susceptibility to creep and recovery (Komine et al, 2012). These features can provide advantages, especially in the areas of high occlusal stress, like implant-supported fixed restorations (Çiftci and Canay, 2000). The procedure of treating ceramic surfaces is required to ensure a long-term bond between composite and ceramic material like sandblasting, etching with different acids (Nagayassu et al, 2006; Bajraktarova-Valjakova et al, 2018).

Air abrasion with Al2O3 particle was recommended to provide the required micro roughening on the bonding surface (Kern, 2015). In addition, the adhesive bonding was used to improve the bonding of resin to zirconia. Such bonding is dependent on the surface energy and wettability of the adherent by the adhesive (Pisani-Proenca et al, 2006; Kobes and Vandewalle, 2013).

Material and Methods

A total of twenty-four zirconia cores were prepared from pre-sintered zirconia blank. They were designed and cut by the CAD-CAM system. Based on the surface treatment and veneering materials zirconium cores were divided after standard surface sandblasting procedure into three groups (n=8) as shown in figure (1).

An ideal pre-prepared plastic right maxillary first molar (Nissin Dental Products, Kyoto Japan) with deep chamfer finishing line (0.8mm), (2mm) reduction occlusally was utilized for the construction of a master metal die as shown in figure (2).

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The plastic die was sprayed with dental scan spray to inhibit reflection of light during the scanning process and the plastic die was placed inside the dental scanner unit (DOF, full HD, 5M pixel; Korea) and fixed on scan stage by special clay as shown in figure (3).

A three-dimensional image was taken so that all surfaces and finishing lines of the plastic die appeared clearly. The metal die was fabricated by using the CAD-CAM system to simulate the shape of ideal pre-prepared plastic die to receive all crowns (Hamza and Sherif, 2017).

The digital model of the die transferred to the CAM software to start the dry milling process of the metal die by using the milling unit which was loaded with the cobalt-chromium disc (10mm) [Interdent, Travagliato (BS) Italy]. The base for the metal die was constructed from dental stone type IV (Azarbal et al, 2018), this base allows the proper position of the metal die during scanning. Then the dental stone was mixed according to manufacturer instruction, vibrated, and poured to 4mm below the cementoenamel junction (Abdulkareem and Ibraheem, 2016) as shown in figure (4).

The metal die was surface scanned and by the same procedure used previously in the scanning of the plastic die. A zirconia core with (0.7mm) thickness was designed to fit on the digital die using CAD-CAM technology (Alsadon et al, 2017).

The cores were designed through the software, Yttria-stabilized zirconia blank was positioned in the blank holder into the milling machine and fixed with the screwdriver, at that point, milling procedure began. When the milling procedure was finished, the sintering procedure was established for all cores at a high temperature according to the manufacturer's guidelines. Following sintering, the surface of each zirconium core was subjected to an air abrasion procedure with (50µm) Al203 particles using a sandblasting machine at a pressure of 1.5 bar for 10 seconds, and at a fixed distance of 10mm between the nozzle head and the core surface. The study groups were prepared as follows:

Group (A) the control group, specimens veneered with IPS E.max Ceram porcelain.

Group B, specimens surface treated with Phosphoric acid 37% and bonding agent, veneered with 3M ESPE Filtek (Packable P60 Composite resin).

Group C, specimens treated with Hydrofluoric acid of 9.5% and the application of bonding agent, then veneered with 3M ESPE Filtek (Packable P60 Composite Resin). In group (A) a silicon index was fabricated, a putty condensation silicone impression material (Zhermack, Italy) was used to fabricate a silicon index, an impression was taken to the previously veneered composite crown to control the thickness of veneering ceramic as shown in figure (5). While for the application of veneering ceramic on zirconium core, the layering technique was used by mixing ceramic powder (50 mg) of IPS Ceram, dentin A3 powder with a special liquid to produce the desired creamy consistency of ceramic, then the ceramic was applied to the prepared core surface by a brush, then sintering process was achieved in the ceramic furnace according to the manufacturer instructions.

The procedure was repeated for the 2nd layer of dentin and enamel porcelain were condensed and fired also according to the manufacturer's instructions. After complete firing, the dimensions of veneering ceramic were checked byindex and Vernier. In group (B) a phosphoric acid gel (37% concentration) was applied to the sandblasted core surface for 60sec and then rinsed with water spray for 30sec to remove all residual acid. Single Bond Universal Adhesive (3M ESPE, USA) was used before the application of composite resin. A drop of bond applied to the etched surface and rubbed using a disposable

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brush and lightly air-dried for 2sec, then the bond was light-cured for 20sec, and all the five surfaces of each crown were light-cured. For the application of composite resin (veneering material) and to obtain the desired and uniform composite veneering thickness to all composites groups, a disposable celluloid crown was used as a mold for veneering material. The Packable Filtek P60 composite resin was applied inside the celluloid crown using the Ash instrument (no. 6) to add the composite increment and the material adapted and compacted to ensure a procedure free of bubbles or gap between the material and celluloid crown. Also to obtain a full contouring crown, then the celluloid crown containing the veneering material was placed over the zirconium core and applied a slight pressure for adjusting it. All excess material from the margin was removed using a lacron carver instrument. Then light-activated using a light-cure unit with a power intensity of 600mw/cm2 for 20sec for each occluded, buccal, palatal, mesial and distal surfaces according to manufacturer's instructions. After the curing procedure was completed, the celluloid crown was removed from the restoration. The glaze-on technique was established for the group (C), the surface of zirconium cores was coated with a very thin layer of glazing porcelain and sintered with glaze firing protocol according to manufacturer's instructions, this to obtain a glazed surface for zirconium cores (Peampring et al, 2017). For etching, a glazed surface was etched with 9.5% of hydrofluoric acid for 30sec, then rinsed with water spray for 60sec to remove all residual acid, and dried with oil-free compressed air.

The single bond universal adhesive material (3M ESPE, USA) was used before the application of composite resin. The bonding procedure was accomplished in the same manner as in the group (B). Then the veneering material applied as in group

(B) using Filtek P60 composite resin III. Fracture resistance testing procedure takes place using a universal Instron testing machine of 6mm diameter steel ball indenter (Lloyd LRX-Plus, Lloyd Instruments Ltd. Fareham Hants, UK), and a crosshead speed of 0.5mm/min at the center of each crown. The fracture at maximum load was then recorded (Peampring et al, 2017). One-way analysis of variance (ANOVA) and Least Significant Difference test (LSD) tests were applied at a confidence level of 95% and significant P-value of ($p \leq 0.05$).

Results

Table (1) showed the highest mean of fracture resistance value was in the group (B), while the lowest mean of fracture resistance value was in the group (C). In table (2), One-way ANOVA revealed that there was a statistically highly significant difference in fracture resistance between both group A and C, and B and C. While there was statistically non-significant difference effect between both group A and B. LSD test also in table (3) determined the source of variance among all tested groups.
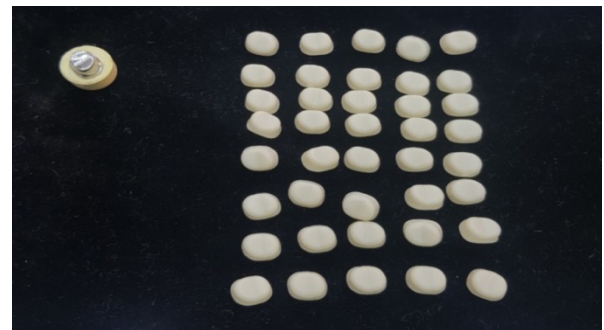

Figure 1: Zirconium cores.

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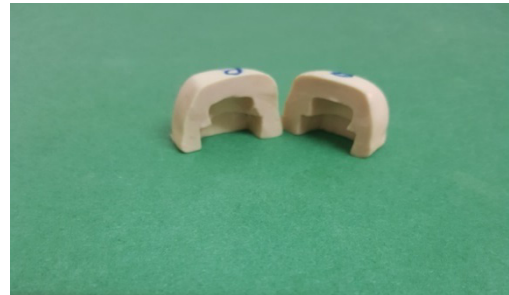**Figure 2: An ideal pre-prepared plastic right maxillary first molar.**

Figure 5: Silicone index.

Figure 3: The plastic die was placed on the stable scan stage.

Figure 4: The final metal die with stone base.

Table (1): Descriptive statistics of fracture resistance in Newton for the study groups A, B, and C.

	N	Minimum	Maximum	Mean	Std. Error	SD
Group A	8	1721.65	1978.92	1826.4575	38.59839	109.17273
Group B	8	1819.76	2133.67	1935.6350	39.77285	112.49461
Group C	8	1331.95	1716.75	1582.4713	41.56172	117.55428

Table (2): One-way ANOVA test for the study groups A, B, and C.

Groups	F	P-value	Sig
Groups (A,B,C)	20.439	.000	*** HS

*P≤0.05 Significant
**P>0.05 Non-significant
*** P≤0.001 Highly significant

Table (3): Least Significant Difference test (LSD) for the study groups A, B, and C.

Groups			Mean Difference	Std. Error	P-value
LSD	Group A	Group B	109.17750	56.56319	.155
		Group C	243.98625	56.56319	.001
	Group B	Group C	353.16375	56.56319	.000

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Discussion

To control some of the ceramic veneered zirconia lowering properties regarding fracture resistance, veneering zirconia-based crown with composite was suggested. Such crowns are fabricated with a light-activated composite. The advantages could incorporate strength, less abrasive and biocompatibility of zirconia frameworks permit simplicity of treating and repair intraorally (Guazzato et al, 2004; Agustin et al, 2014). In the present study, the load to failure test is the method to examine the structural safety of such structures, which takes into account different component layers of the crown and anatomy complexity (Casson et al, 2001), also the crosshead speed of 0.5mm/min with the load that applied at the crown occluded center using a 6mm stainless steel ball diameter (Peampring et al, 2017). The selection of (Al2O3) air-brone of 50µm particle size was highly recommended for achieving strong adhesion of veneering ceramic due to increasing surface roughness and providing undercuts (Guazzato et al, 2005). Also, sandblasting the zirconia surface before porcelain veneer or resin bonding appears to be the most popular method to promote mechanical interlocking when moderate pressure applied with small particle size (Kern, 2015). In term of sandblasting effect, the results of the present study showed a high fracture strength value was in group B, while the lowest was in group C. This could be explained by that the sandblasting which may not only bring about morphological changes of the material surface but also increase adhesion efficiency. Also, one possible factor that could be referred to is the loss of primary stability of zirconia by its transformation from the tetragonal into the monoclinic crystallographic phase as a result of elevated temperature and the presence of moisture (Korkmaz et al, 2015). The abrasion with air-brone particles probably enhances the formation of ceramic-resin micromechanical interlocking by expanding the bonding region, resulting in an activated micro-roughened zirconia surface, modifying surface energy and wettability of the ceramic surface (Chintapalli et al, 2013). The result of the present study comes in agreement with Phark et al., 2009 which concluded that abrasion with Al2O3 abrasive particle has been distinguished as an effective factor in obtaining a durable and stable bond for aluminum and zirconia-based ceramics. Also, the result of the present study comes in agreement with Su et al, 2015 who state that sandblasting procedure is an important surface treatment method that could enhance the bonding strength between veneer material and zirconia, this fine powder particle was more abrasive and significantly more zirconia was removed when using 50µm of powder particles.

On the other hand, some findings may disagree with Inokoshi et al, 2014, a study suggested that air-borne particles abrasion with alumina could produce micro-cracks and damage the surface integrity which influences the properties of zirconia. This difference in results may be due to the differences in some study variables such as applied pressure. In terms of phosphoric acid and bonding agent effect, the highest mean value of the fracture strength test was noticed when the zirconium core was abraded and chemically managed with phosphoric acid treatment. This could be explained by the interference of efficient and functional monomers within the zirconium surface (Kobes and Vandewalle, 2013). The phosphate monomer that found in the primer may have bonded to the zirconia ceramic and form chemical bonds at the zirconia resin interface through either covalent bonds, hydrogen bonds or van der Waals forces. The bond adhesive primer contains silane monomers and this silane may increase the bond strength by producing a chemical bond between res-

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in composite and silica-based surfaces. This increase in wettability could improve the flow of resin into the ceramic surface, causing a strong micromechanical bond (Kobes and Vandewalle, 2013). These results probably in agreement with Komine et al, 2009 who conducted that obtaining a durable bond strength could be obtained by utilizing an acidic functional monomer containing phosphate monomer, carboxylic anhydride, or phosphoric acid. Also, Derand and Derand, 2000, reported that the phosphoric-acid groups of 10-methacryloxydecyl dihydrogen phosphate (MDP) can react with the oxide layer on the surface of the ceramic material. This leads to sufficient adhesion between these two materials. Ural et al, 2011, also found that Methyl methacrylate in the primer can be polymerized with the monomers of a resin composite systemso that bonding between the resin and the ceramic

surface might have been increased. To fuse a glaze material to the zirconia substructure and then apply composite material with the specific bonding with silanization protocols may be advice favorably for the durable bonds of the composite to zirconia ceramics (Kitayama et al, 2010). HF acid is broadly utilized in dental laboratories in traditional fixed prosthodontics and adhesive all-ceramic applications (Guazzato et al, 2004). The hydrofluoric acid is used when the matrix contains silica or silicates. At first, silicon tetrafluoride is formed. This combines with hydrofluoric acid to form soluble complex ion (hexafluorosilicate) which in turn reacts with hydrogen protons to form tetrafluorosilisic acid, a product that can be selectively removed with water, and the crystalline structure is uncovered, the outcome surface of the ceramic becomes rough (Tarib et al, 2016). Thus, the HF acid treatment is widely used on silica-based ceramic to react with, and exclude the glassy matrix that contains silica. This leaves the crystalline phase exposed,

generating surface roughness as a result of the formation of numerous porosities and grooves due to the acid action on the matrix and the crystal structure; initiating the extreme bonding (Bajraktarova-valjakova et al, 2018). Also this study findings come in agreement with a study by Guazzato et al, 2004 who reported that the adhesion of all-ceramic restorations, surface morphological changes, just like pores and grooves, are considered important. Both chemical bonding and micromechanical interlocking to the surface of ceramic could increase the fracture resistance of the restored tooth restoration. Also, provide high retention; improve marginal adaptation; and prevente microleakage. Moreover, Borges et al, 2003 and Pisani-Proenca et al, 2006 stated that chemically a hydrofluoric acid may dissolve glass by reacting with silicon oxide which is the main ingredient in glass ceramic. Furthermore, the glaze coating of zirconia framework is an effective method to obtain a clinically acceptable bond strength of composite material to a zirconia substructure (Fushiki et al, 2012). Nagayassu et al, 2006 claimed that etching of dental porcelain with hydrofluoric acid was reported to give higher bond strength of resin composite to porcelain. The differences in such results may be explained by using silane coupling agents following etching with hydrofluoric acid.

Conclusion This study concluded that the use of different surface treatment for zirconia cores was generally more effective in increasing fracture strength. The use of a bonding agent of 37% phosphoric acid and composite resin material had a significant increase in fracture strength and bonding strength between zirconia and the composite resin.

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Conflict of interest We are the author's (Yusra A. Ibraheem, and Dr. Zahraa N. Alwahab) state that the manuscript for this paper is original, it has not been published previously; it is part of my MSc. Dissertation; is not under consideration for publication elsewhere and that the final version has been seen and approved by all authors.

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