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Abstract: Background: A variety of veneering options to zirconia frameworks are now available. The purpose of this study is to evaluate the effect of veneer materials, veneering methods, cement materials, and aging on the failure load of bilayered veneer zirconia. Material and methods: Zirconia bars ($20 \times 4 \times 1$ mm) were veneered to 2 mm total thickness (n = 10/group). Veneering method groups included: 1. Hand-layered feldsparthic porcelain (VM = Vita VM9, Vident) and fluorapatite glass-ceramic (CR = IPS e.max Ceram, IvoclarVivadent); 2. Pressed feldspathic porcelain (PM = Vita PM9, Vident) and fluorapatite glass-ceramic (ZP = IPS e.max ZirPress, IvoclarVivadent); 3. CAD-/CAM-milled feldspathic ceramic (TF = Vitablocs Triluxe Forte, Vident) and lithium-disilicate glass-ceramic (CAD = IPS e.max CAD, IvoclarVivadent). CAD/CAM veneers were either cemented with resin cements (P = Panavia21, KurarayDental), (R = RelyX Ultimate, 3M ESPE), (M = Multilink Automix, Ivoclar Vivadent) or fused with fusion glass-ceramic (C = CrystalConnect, IvoclarVivadent). A three-point bending test (15 mm span, zirconia on tension side) was performed on Instron universal testing machine (ISO 6872) recording load-to-failure (LTF) of first veneer cracks or catastrophic failure. For group VM, PM, TF-M, TF-C, CAD-M, CAD-C, ten more bars were prepared and aged with cyclic loading (100,000 cycles, 50% LTF) and thermocycling (2000 cycles) before testing. Data were analyzed by ANOVA, Tukey HSD post hoc tests, and *t*-test ($\alpha = 0.05$). Zirconia veneered with IPS e.max CAD by fusing had significantly higher failure load compared with zirconia veneered with other veneering materials ($p \le 0.05$). For cemented veneers, the cement type had a significant effect on the failure load of the veneer zirconia specimens. Specimens cemented with Panavia 21 had a lower resistance to loading than other cements. The aging experiment revealed a significant difference in failure load between non-aged and aged bars in groups VM and PM, but not in the groups with CAD-/CAMmilled veneers. In conclusion, veneer materials, veneering methods, and cement materials have a significant effect on the failure load of bilayered veneer zirconia. CAD-/CAM-milled veneer zirconia is not susceptible to aging performed in this study.

Keywords: zirconia; CAD/CAM; crowns; prosthodontics; ceramics

1. Introduction

Advances in dentistry have increased the use of computer-aided design/computeraided manufacturing (CAD/CAM) systems in fabricating ceramic restorations [1]. These machinable ceramic restorations are made from highly uniform crystalline materials as compared to conventional fabricated restorations [2]. As a result, the Weibull modulus of oxide ceramics and thus the reliability of the restorations was significantly increased [3]. New aesthetic materials have been made available over time, showing increased aesthetic and mechanical properties [4–8].

Current processing technologies unfortunately cannot make zirconia frameworks as translucent as natural teeth, so they are veneered with weaker porcelain to achieve acceptable esthetics. This veneer material is usually hand layered onto the zirconia core and fused onto the ceramic core by sintering [9].



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As veneer material is weaker compared to the core, it fails at low loads when it is placed under tension [10]. Cracks may originate from the interface between the core and the veneer, from the free surface of the veneer and from the inner surface of the core [11]. The placement of the veneer material may critically affect the fatigue properties of the layered restoration. The typical failure pattern of a veneering material in the daily clinical practice is known as ceramic chipping [12–14]. This fracture pattern is associated with a thin layer of glass ceramic that remains on the zirconia framework [12,13,15,16]. This indicates a reliable bond of veneering ceramics to the framework, but also reveals a weakness of the veneering porcelain [17]. Heat pressing of the veneer porcelain onto the zirconia core was introduced as an alternative veneering technique to overcome cohesive failure [18]. However, in vitro studies revealed no differences in failure modes and reliability of standardized trilayer configuration [19] but also in load bearing capacity of crown systems with press veneering ceramics compared to hand-layered veneering [9,17].

Recently, CAD-/CAM-milled veneering material was introduced, the zirconia core and veneering materials were milled separately. Both corresponding parts of the restorations can be joined together by two techniques. The first one is sintering technique, which is to sinter them by means of a glass ceramic powder [17]. Another technique is to bond veneering and core parts together with resin cements. In vitro studies demonstrated that crowns made with sintering technique of a CAD-/CAM-generated glass–ceramics for veneering materials to zirconia coping had higher failure load compared to crowns made with other veneering techniques [10,20]. The objective of this study is to evaluate the effect of veneering materials, veneering methods, interface materials and aging on the failure load of bilayered veneer zirconia.

2. Materials and Methods

A total of 10 rectangular specimens of zirconia for each group were prepared with dimension of $1.25 \text{ mm} \times 5 \text{ mm} \times 25 \text{ mm}$ using a 15LC diamond-wafering blade mounted on an Isomet 2000 Precision Saw (Buehler, Lake Bluff, IL, USA). The sample size for each group was determined from standard deviation data from pilot study with the following formula.

Sample size =
$$2 \times (Z_{(1-\alpha/2)} + Z_{\beta})^2 \times \delta^2 / \Delta^2$$

where α is significance level; β is power, Δ is standard deviation of pilot study; Δ is expected size of difference.

The specimen was polished using a Buehler Ecomet 250 Grinder Polisher (Buehler, Lake Bluff, IL, USA). The polishing was done with 15-micro-grit diamond polishing pad. The sectioned bars were sintered according to manufacturer's instruction. The dimension of the zirconia specimens after being sintered was $20 \text{ mm} \times 4 \text{ mm} \times 1 \text{ mm}$. The specimens were then assigned for three different veneering protocols (Table 1).

2.1. Hand-Layered Porcelain Groups

2.1.1. Group 1: Vita In-Ceram YZ + Vita VM9

Zirconia specimen was cleaned and positioned in the silicone mold. Base dentin wash bake was made by mixing VM9 Base dentin powder (Vita Zahnfabrik, Germany) with modeling liquid to obtain a thin aqueous mixture, applied very thinly to the zirconia sample and then fired in Vita Vacumat 6000 M furnace (Vita Zahnfabrik, Germany) following manufacturer's instructions. Porcelain powder was mixed with modeling liquid and packed onto the mold. Porcelain was then fired according to manufacturer's instruction. The samples were finished with the same protocol when polishing the zirconia specimens as previously mentioned, then glazed using Akzent glaze powder and liquid (Vita Zahnfabrik, Germany).

Group	Zirconia Materials	Veneering Materials	Interface Materials
1	Vita In-Ceram YZ	Vita VM9	
2	IPS e.max ZirCAD	IPS e.max Ceram	
3	Vita In-Ceram YZ	Vita PM9	
4	IPS e.max ZirCAD	IPS e.max Zirpress	
5	Vita In-Ceram YZ	Vitablocs Triluxe Forte	Panavia 21
6	Vita In-Ceram YZ	Vitablocs Triluxe Forte	Multilink Automix
7	Vita In-Ceram YZ	Vitablocs Triluxe Forte	RelyX Ultimate
8	IPS e.max ZirCAD	IPS e.max CAD	Panavia 21
9	IPS e.max ZirCAD	IPS e.max CAD	Multilink Automix
10	IPS e.max ZirCAD	IPS e.max CAD	RelyX Ultimate
11	Vita In-Ceram YZ	Vitablocs Triluxe Forte	IPS e.max CAD Crystall/Connect
12	IPS e.max ZirCAD	IPS e.max CAD	IPS e.max CAD Crystall/Connect

Table 1.	Experimental	groups
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2.1.2. Group 2: IPS e.max ZirCAD +IPS e.max Ceram

IPS e.max Ceram ZirLiner (Ivoclar Vivadent, Liechtenstein) was mixed with the ZirLiner liquid to a creamy consistency and then applied on each zirconia specimens. ZirLiner was fired in Programat EP 5000 furnace (Ivoclar Vivadent, Liechtenstein) following manufacturer's instructions. Base dentin was made by mixing IPS e.max Ceram Base dentin powder (Ivoclar Vivadent, Liechtenstein) with modeling liquid to obtain a thin aqueous mixture, applied on the fired ZirLiner and then fired following manufacturer's instructions. Porcelain powder was then mixed with modeling liquid and packed onto the specimen inside the mold using a vibrator. Porcelain was then fired according to manufacturer's instructions.

2.2. Pressed-on Ceramics Groups

Group 3: Vita In-Ceram YZ + Vita PM9 and Group 4: IPS e.max ZirCAD + IPS e.max Zirpress

The zirconia specimens were positioned into the silicone mold as previously mentioned in group 1. For group 4, IPS e.max Ceram ZirLiner was mixed with the ZirLiner liquid to a creamy consistency and then applied on each zirconia specimens. ZirLiner was fired in Programat EP 5000 furnace following manufacturer's instructions. Blue-inlay casting wax (Kerr, Switzerland) was melted and placed over the zirconia specimen. The specimens were smoothened, sprued and invested into IPS PressVEST speed investment material. The wax was burned out and the muffle was heated. The specimens were over pressed with either Vita PM9 (Vita Zahnfabrik, Germany) or IPS e.max Zirpress (Ivoclar vivadent, Liechtenstein) ingots using Programat EP 5000 furnace following manufacturer's instructions. After cooling, the investment material was removed using separating disc. The specimens were divested by sandblast with 30µm Aluminum oxide particle at 2 bars. The samples were finished with the same protocol when polishing the zirconia specimens as previously mentioned, then glazed using Akzent glaze powder and liquid.

2.3. Cemented Milled Ceramics Groups

2.3.1. Groups 5, 6, and 7: Vita In-Ceram YZ + Vitablocs Triluxe Forte + Resin Cement

Vita Triluxe Forte CAD/CAM ceramic blocks (Vita Zahnfabrik, Germany) were sectioned into ceramic bars with a dimension of $1 \text{ mm} \times 4 \text{ mm} \times 20 \text{ mm}$. The ceramic bars were polished using a Buehler Ecomet 250 (Buehler, Lake Bluff, Illinois). The polishing was done with 15-micro-grit diamond polishing pad at 30 rpm with water irrigation for 90 seconds for each side, and then thoroughly rinsed. The bar was glazed using Akzent glaze powder and liquid. Veneering bars and zirconia bars were cemented together with resin cements according to assigned groups (Group 5: Panavia 21, Group 6: Multilink Automix, and Group 7: RelyX Ultimate). The cementation process followed manufacturer's instructions.

2.3.2. Groups 8, 9, and 10: IPS e.max ZirCAD + IPS e.max CAD + Resin Cement

IPS e.max CAD blocks (Ivoclar Vivadent, Liechtenstein) were sectioned into ceramic bars with a dimension of 1 mm × 4 mm × 20 mm using a 15LC diamond-wafering blade mounted on an Isomet 2000 Precision Saw (Buehler, Lake Bluff, Illinois). The cuts were made at 800 rpm with 300 g of load with cooling provided by a dual-nozzle water irrigation system. The ceramic bars were polished using a Buehler Ecomet 250 Grinder Polisher. The polishing was done with 15-micro-grit diamond polishing pad at 30 rpm with water irrigation for 90 seconds for each side, and then thoroughly rinsed. The veneer bar was subjected to crystallization firing using Programat EP5000 furnace (Ivoclar Vivadent, Liechtenstein) and glazed simultaneously using IPS e.max CAD Crystall glaze paste (Ivoclar Vivadent, Liechtenstein). Veneering bars and zirconia bars were cemented together with resin cements according to assigned groups (Group 8: Panavia 21, Group 9: Multilink Automix, Group 10: RelyX Ultimate). The cementation process followed manufacturer's instructions.

2.4. Fused Milled Ceramics Groups

2.4.1. Group 11: Vita In-Ceram YZ + Vita Triluxe Forte + IPS e.max CAD Crystall/Connect

Vita Triluxe Forte veneer bars were prepared as previously mentioned in group 5. Zirconia and veneer bars were joined using special fusing glass–ceramic IPS e.max CAD Crystall/Connect which was applied to both bars and evenly distributed using Ivomix vibrator (Ivoclar vivadent, Liechtenstein). Then, the bars were fitted together with slight pressure and excess fusing glass–ceramic was removed. Then, they were subjected to sintering using Programat EP5000 furnace. The bars were then glazed using Akzent glaze powder and liquid.

2.4.2. Group 12: IPS e.max ZirCAD + IPS e.max CAD + IPS e.max CAD Crystall/Connect

IPS e.max CAD bars were prepared as previously mentioned in group 8. Zirconia and veneer bars were joined using special fusing glass–ceramic IPS e.max CAD Crystall/Connect which was applied to both bars and evenly distributed using Ivomix vibrator (Ivoclar Vivadent, Liechtenstein). Then, the bars were fitted together with slight pressure and excess fusing glass–ceramic was removed. Then, they were subjected to crystallization firing using Programat EP5000 furnace and glazed simultaneously using IPS e.max CAD Crystall glaze paste (Ivoclar Vivadent, Liechtenstein).

A three-point flexural test was conducted on the specimens using Instron 5566A Universal Testing Frame (Intron, Norwood, Massachusetts, USA) with 1kN load cell. Ten specimens from each group were positioned on the flexure and centered under the loading apparatus with perpendicular alignments and zirconia under tension side (Figure 1). Three-point bending test was conducted on a 15mm span, at a crosshead speed of 0.5mm/min. Each specimen was loaded with the force until the failure of the specimen occurs. Fractured pieces of the specimen are retrieved and stored for future uses. The fracture patterns were also observed and marked as catastrophic failure or veneer delamination/chipping.



Figure 1. Three point bending test of bilayered specimen.

A total of ten rectangular specimens for each group were prepared as mentioned previously. The samples were subjected to 100,000 cycled cyclic loads at 1 Hz using 50% load of the failure load data from the control groups. All the specimens were then subjected to the same temperature changes for the same time period by repetitive immersion into cold 5 °C and subsequently hot 55 °C water baths for 2000 cycles. Movement of the samples between hot and cold tanks took 20 seconds. After simulated aging, the specimens were subsequently subjected to the three-point bending test as mentioned previously.

The Statistical Package for the Social Science Version 23 (IBM SPSS Statistics, New York, USA) was used for statistical analysis. Mean, standard deviation, and coefficient of variation for each group were calculated. Kolmogorov–Smirnov Test of Normality, which confirmed the normal distribution of our data (p > 0.05), was used to determine performed statistics. The failure load was analyzed with one-way ANOVA, followed by a post hoc Tukey test. Two sample Student's *t*-test were done to evaluate the effect of simulated aging on the failure load of each veneering techniques. Two-way ANOVA were done to evaluate the effect of veneering materials, veneering techniques, and cement for cemented milled ceramics groups. A significance level of 0.05 was used.

3. Results

The mean failure load \pm standard deviation values for each group before simulated aging are shown in Figure 2. There was a significant difference of failure load of different groups (p < 0.05). Two-way ANOVA test showed that the effect of veneering materials and veneering techniques had a statistically significant effect on the failure load of veneered zirconia (p < 0.05). When considering only cemented CAD/CAM veneering, two-way ANOVA showed the effect of veneering materials and cements on the failure load (p < 0.05). Post hoc test showed that bilayered specimens cemented with Panavia 21 cement had significantly lower failure load than those cemented with Multilink Automix and RelyX Ultimate (p < 0.05). The null hypotheses that different veneering techniques, veneering materials and cements have no effect on the failure load of bilayered veneer zirconia were rejected.

Regarding modes of failure of specimens. Hand-layered and press-on ceramics failed predominantly catastrophically except IPS e.max ZirPress. For CAD-/CAM-milled veneer, fused e.max CAD failed exclusively catastrophically while cemented e.max CAD with Panavia 21 failed mostly with porcelain chipping/delamination. The rest of CAD-/CAM-milled veneer failed relatively equal between catastrophic failure and porcelain chipping/delamination. The mean failure load \pm standard deviation values for each group after simulated aging was shown in Figure 3. The specimens veneered with hand-layered and pressed veneer had significant lower failure load after the accelerated aging process (p < 0.05), while the failure load of all the specimens veneered with CAD-/CAM-milled veneer remain no different after aging (p > 0.05).



Figure 2. Failure load of bilayered veneer zirconia after 24 h in water. All groups under the same horizontal line are not significantly different to each other (p < 0.05).



Figure 3. Failure load of bilayered veneer zirconia in control group (before the simulated aging) compared with after simulated aging.

4. Discussion

Strength of dental ceramic can be influenced by many factors such as size, numbers and distribution of flaws, thickness of the core, presence of the veneering material in the system, loading conditions, etc., and is usually measured in flexure (bending) because this test is generally easier to perform than a pure tensile test. In bending test, tensile stress reaches a maximum on one surface and compressive stress reaches the maximum on the opposite side.

In dental applications, ceramic copings are usually covered with an esthetic layer of feldspathic porcelain. Such a combination forms a layered structure with different elastic moduli and thermal expansion coefficients. It is thus important to evaluate the failure

stresses of such materials as layered composites. In the present experiment, bilayered bar-shaped specimens were used. This allowed the study to be reproducible and easier to standardize the specimens. Another advantage is that bar-shaped specimens required less materials compared with crown-shaped specimens. For example, one standard size (14 mm \times 14 mm) CAD/CAM block can be sectioned into 9 to 12 bars.

In the present study, there was no statistically significant difference between the failure load of bilayered hand-layered veneered and pressed veneered zirconia. The flexural strength of both hand-layered and press-on porcelain, which are around 70–90 MPa, are relatively low compared to CAD-/CAM-milled ceramics [21,22]. The hand layering technique is a very technique sensitive method and require many factors such as mixing quality of porcelain liquid, dental technician experience, cooling parameter, and ceramic shrinkage whereas the press-on technique minimize the firing shrinkage and can give good marginal adaptation [23,24]. Others have reported that divesting procedure of pressed-on ceramics involves immersion in hydrofluoric acid solution and sandblasting, to remove the reaction layer, which significantly increases the surface roughness of the ceramics [25]. These imperfections on the outer surface of ceramics are prone to crack formation and propagation [25].

CAD-/CAM-milled veneer enables stronger veneering materials to be used and high-quality standards through prefabricated ceramic blocks manufactured by industrial pressing without any porosities to be achieved [26,27]. Results from the present study showed that there are differences in the failure load between bilayered bars with different resin cement. These indicated that cement materials might play a significant role to determine the failure load of the bilayered system. The flexural strength of the interface materials and the bond strength between the interface materials and ceramic are the two properties that might be related to the failure load of the bilayered system. Low failure load of the bilayered bars cemented with Panavia 21 might be explained by the absence of zirconia primer of this system, thereby lowering the bond strength of the resin cement and zirconia.

In the present study, the zirconia bars veneered with IPS e.max CAD have generally high failure load compared to other combination of zirconia and veneer materials. One of the reasons is the flexural strength of the veneer material itself, which is about 3–4 times higher compared with the others. IPS e.max CAD and IPS e.max ZirCAD fused by fusion glass ceramic possessed highest failure load. These can be explained as an effect of the high flexural strength of lithium disilicate veneer (360 MPa) combined with high flexural strength (160 MPa) and elastic modulus of the fusing porcelain. The crystallization and fusion at the same cycle may allow the fusion glass–ceramic to flow and adhere to the IPS e.max CAD better.

Specimens veneered with hand-layered Vita VM9 or press-on Vita PM9 showed significant reduction of their failure load after simulated aging (Figure 3). This could be due to the inherent weakness of the veneering porcelains, processing flaws (voids, inclusion porosities or incomplete burn-out of the wax). All of these led to cracks and flaws that can propagate through the veneering ceramic [15,28,29]. The low thermal diffusivity of the zirconia results in the highest temperature difference and very high residual stresses for zirconia veneer specimens, which generates high tensile subsurface residual stresses, and may result in unstable cracking or chipping [30]. CAD-/CAM-milled veneer, on the other hand, retained same failure load even after simulated aging. This could be explained by the absence of porosities inside the veneering materials which reduces the possibility of crack propagation to occur.

Although bar specimen can identify important trends and has relevance to more complex clinical situations, it does have some disadvantages. For example, it has a much simpler geometry than FPD and it lacks thinner stress-concentrating connectors. Moreover, it is not supported by flexible dentin, a flexible periodontal ligament, or flexible bone. However, the same mechanical principles do apply to crowns and FPDs, and it should

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be noted that only one ceramic core/veneering porcelain thickness ratio was tested in this study.

5. Conclusions

Within the limitation of the study, the following conclusions can be drawn:

Veneering materials, veneering methods, and interface materials had a significant effect on the failure load of bilayered veneer zirconia (p < 0.05).

Zirconia veneered with IPS e.max CAD by fusing had significantly higher failure load compared with zirconia veneered with other veneering materials (p < 0.05).

For cemented veneers, the cement type had a significant effect on the failure load of the veneer zirconia specimens. Specimens cemented with Panavia 21 had a lower resistance to loading than the other cements studied (p < 0.05).

CAD-/CAM-milled veneer zirconia specimens were not susceptible to aging (cyclic loading and thermal cycling) performed in the present study (p > 0.05).

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Abbreviations

CAD/CAM: computer-aided design and computer-aided manufacturing.

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