

## Bonding Stability of a Novel Nano-Zirconia and Commercial Veneering Ceramics

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### ABSTRACT

Nano-zirconia is commonly used in dentistry as a framework material over which feldspathic porcelain is layered to form an aesthetic and strong bi-layered restoration. Sufficient bonding strength between zirconia frameworks and veneering porcelains is crucial for the longevity of zirconia-based dental restorations. This study aims to determine the shear bond strength (SBS) of a colloidal-processed slip-casted novel nano-zirconia (NZ) and veneering ceramics, namely Cercon® Ceram Kiss (CK), VITA VM®9 (VM9), and IPS e.max® Ceram (e. max). Twenty-four cylindrical samples of each NZ and CZ were prepared and veneered with the veneering ceramics. Six groups of core-veneer assemblies, which are denoted as NZ-CK, NZ-VM9, NZ-e. max, CZ-CK, CZ-VM9, and CZ-e. max, were prepared for shear bond testing using the universal testing machine. Results of the shear test showed that NZ-VM9 recorded the highest shear strength value of 149.48 MPa, whereas NZ e. max showed the lowest SBS value of 71.56 MPa. A similar trend was also observed in the CZ groups, with the highest mean bonding strength (123.38 MPa) when bonded to VM9 (CZ-VM9), while the lowest mean value (44.07 MPa) was registered by the CZ-e. max groups. Scanning electron micrographs revealed that groups with high core-veneer bond strength values mostly displayed cohesive failure. By contrast, a mixed-mode of adhesive and cohesive failures was observed in the low SBS groups. The NZ showed similar performance based on the bonding property analysis. Therefore, NZ could potentially be used as an alternative to CZ.

*Keywords:* Nano-zirconia; veneering porcelain; slip-casting; colloidal processing; bond strength

### INTRODUCTION

The use of zirconia-based materials has become popular over recent years due to their excellent biocompatibility, exceptional mechanical properties, and acceptable optical qualities (Y. Zhang & Lawn 2018). Using the computer-aided design and manufacturing (CAD/CAM) technology, zirconia is usually milled into a framework, which is then veneered with glass ceramics to achieve uniquely strong and aesthetic bi-layered restorations (De Freitas et al. 2020; Kern et al. 2019; Shahmiri et al. 2018; Tanaka et al. 2019). Evidence from the literature suggests good clinical outcomes of zirconia-supported restorations. For example, in a 12-year retrospective clinical study, (Miura et al. 2018) estimated the survival of approximately 98.5% dental zirconia crowns at five years. However, the success rates

drastically dropped to approximately 62.1% after 10 years, with chipping of veneering porcelain as the most common complication reported. Similar findings have also been observed by several other authors (De Lima et al. 2015; Pjetursson et al. 2018; Shi et al. 2017; Triwatana et al. 2012). Premature fracture of veneer laminates occurs as frequently as 25% over three years (Ioannidis & Bindl 2016) and as early as six months post-cementation (Tsanova et al. 2018). Compared with metal-ceramic restorations, the occurrence of porcelain chipping was three times more frequent in veneered zirconia restorations (Sailer et al. 2018). Therefore, the core-veneer interface is the weakest link in the zirconia-supported all-ceramic systems. Although this technical complication can be temporarily remedied by a resin repair, replacement of the entire restoration is often indicated as a long-term solution.

The latter procedure requires tedious removal of the entire prosthesis with potential iatrogenic damage to the natural tooth structure as well as financial implications to the patient. (Ioannidis & Bindl, 2016).

Investigators believe that the cause of veneering porcelain chipping or delamination is multifactorial. Factors, such as inherent mechanical strength of porcelain veneers and zirconia core (Ghaffari et al. 2019; Vidotti et al. 2017), mismatch of thermal properties of ceramics (Jikihara et al. 2019; Mainjot et al. 2015), geometric dimension of the zirconia framework or veneers (Alothman & Bamasoud 2018; Yu Zhang & Kelly 2017), manufacturing parameters of the restorations (Methani et al. 2020), including surface treatments (Ozer et al. 2017), were found to affect the core–veneer bond strength. Nevertheless, the consensus in the literature is that a zirconia core material must form a strong bond with its porcelain laminates to withstand functional stresses during mastication.

One of the primary factors that have been identified to influence the bonding capability between the core and veneering layers is the intrinsic properties of the zirconia core. Zirconias with high susceptibility to the undesirable auto-catalytic transformation during porcelain firing possess low bond strength to their veneering ceramics (Tholey et al. 2011). Zirconia with excellent resistance to low-temperature degradation can be developed from zirconia blocks with almost 99.9% homogenous nano-zirconia particles (Amat et al. 2014). The tendency of nanoparticle agglomeration is prevented by using a special controlled colloidal processing (Aboras et al. 2016; Amat et al. 2014, 2018b; Chin et al. 2018). The high homogeneity of nanoparticles enables slip casting of zirconia powder into the required shapes without expensive equipment (Yu et al. 2011). Preliminary findings revealed that the slip-casted colloidal-processed nano-zirconia displayed comparable mechanical and aesthetic qualities to the commercial zirconias (Aboras et al. 2019; Amat et al. 2018a; Chuin Hao et al. 2016; Daud et al. 2017; Theng et al. 2015). Nonetheless, information on whether zirconia with good aging resistance can withstand porcelain chipping is limited. Thus, this study aimed to investigate the bond strength of slip-casted novel nano-zirconia with different commercially available veneering ceramics using shear bond test and observe the mode of bonding failure.

## METHODOLOGY

The present study utilized two core materials, namely a novel nano-zirconia (NZ) and a commercial zirconia Cercon® (CZ), along with three veneering ceramics of Cercon® Ceram Kiss (CK), VITA VM®9 (VM9), and IPS

e.max® Ceram (e. max). The chemical composition of each material is shown in TABLE 1.

### MEASUREMENT OF CORE–VENEER SHEAR BOND STRENGTH

#### Preparation of Zirconia Core Materials

Twenty-four cylindrical-shaped samples of 10 mm diameter and 3 mm thickness were fabricated from each zirconia used in this study. A colloidal processing technique using special dispersing parameters (Amat et al. 2018b) was employed for the novel nano-zirconia (NZ). Nano-zirconia powder was mixed with distilled water at 1:1.25 weight ratio to produce the nano-zirconia slip. Polyethyleneimine was added at 0.5 wt% to prevent the agglomeration of nanoparticles. pH adjustment was performed by titrating the hydrochloric solution (FiveEasy™ F20 pH, Mettler Toledo, Switzerland). The suspension was stirred on a magnetic vibrator for 45 min and then transferred to an ultrasonic chamber (Ultrasonik 28X, NDI, California USA) for vibration at 50 cycles per second for 15 min. Using the slip-casting approach, the dispersed zirconia slurry was poured into a porous cylindrical plaster of Paris mold and left for three days for complete hardening. The “green body” of zirconia samples was then fired at 150°C for 2 h (Chin et al. 2018).

CZ samples were prepared by milling Cercon® zirconia blocks (Cer, Brain Expert, DeguDent GmbH, Germany). The pre-sintered blanks were sintered using a furnace (Cer, Heat Plus, DeguDent GmbH, Germany) at 1350 °C for 90 min according to the manufacturer’s recommendation (Daud et al. 2017).

#### Preparation of Veneering Ceramics

Sintered core materials discs (NZ and CZ) were sandblasted with 50 µm Al<sub>2</sub>O<sub>3</sub> at 0.2 MPa at a distance of 1.0 cm for 20 s and then cleaned with acetone for 10 min in an ultrasonic bath. A piece of plastic tape was placed on each disc before the positioning of the veneering ceramics to define the bonding area. The powder and liquid veneering ceramics were mixed and packed onto the defined bonding area of the core discs by using a metal split mold. Packing was conducted incrementally, and manual vibration was performed to remove entrapped air bubbles. Residual water particles were removed through blotting with thin facial tissues before sintering in a furnace. All core samples received a layer of veneering ceramic and sintered to obtain 3 mm thick veneers as shown in FIGURE 1 All zirconia–veneer ceramic assemblies were steam- and ultrasonically-cleaned for 30 min and then stored for 24 h at 37°C (Saito et al. 2010).

Combinations of zirconia–veneering ceramic samples were divided into the following six groups: NZ-CK, NZ-VM9, NZ-e. max, CZ-CK, CZ-VM9, and CZ-e. max. The universal testing machine (AGS-1000D, Shimadzu® Co., Japan) was used to perform the shear bond strength (SBS) test with a 5 kN load cell as shown in FIGURE 2. All samples were loaded to fracture at the interface at a crosshead speed of 0.5 mm/min. The bonding strength for each sample was calculated and recorded in megapascals (MPa) using the stress formula [load (N)/area (mm<sup>2</sup>)]. Calibration was performed before each procedure, and the shear blade was set to zero position.

The debonded sample surfaces of cores and veneers were sputter-coated with an 8 µm layer of platinum (Quorum, Q150R S, UK) and examined by scanning electron microscope (FESEM, ZEISS, SUPRA 55VP, German) with secondary electron imaging and acceleration voltage of 10 kV. The photographs were taken at a magnification of x30 at the center of the debonded area of core–veneer assemblies to determine the bonding failure mode (adhesive, cohesive, and mixed adhesive/cohesive) and observe the fractured surface.

The collected data were analyzed with Statistical Package for Social Science Software version 24 (IBM, USA). Statistically significant differences were determined among and between groups by using one-way Analysis of Variance (ANOVA) and post-hoc test (Tukey's test). The level of significance for all statistical testing was predetermined at a p-value of 0.05 or less.

## RESULTS AND DISCUSSION

Table 2 shows the mean SBS values of six core–veneer groups. The SBS of six core–veneer assemblies, namely NZ-CK, NZ-VM9, NZ-e. max, CZ-CK, CZ-VM9, and CZ-e. max, were evaluated by using the universal testing machine. One-way ANOVA test with post-hoc Tukey's test was used to determine the significant differences between the six core–veneer groups. Statistically significant differences were observed when different veneers were used ( $p < 0.05$ ), while no significant distinction was found between the zirconia cores. CK and VM9 veneering ceramics achieved the highest SBS values when layered on novel and commercial zirconia cores. Consistent with other studies (Domingues et al. 2013; Juntavee & Dangsuan 2018; Mosharraf et al. 2012; Saito et al. 2010), the bonding capabilities of CK to Cercon® are expected because CK belongs to the same manufacturer and specifically intended for use with Cercon®. The similarity in the chemical composition between VM9 and CK may explain the likeness in their SBS values (Juntavee &

Dangsuan, 2018). Meanwhile, the SBS of both zirconia cores veneered with e. max was comparatively lower than the other veneering porcelains tested. The result may be due to the design of IPS e.max® Ceram for use with lithium disilicate (Elrashid et al. 2019). To date, no study has specifically examined the SBS of slip-casted zirconia; thus, the findings in this study are only preliminary. Therefore, further in vitro and in vivo studies are necessary to confirm the aforementioned results.

In the literature, veneering methods and different veneer materials may show variable SBS values. Press-one and CAD-CAM veneering methods have been shown to possess better bonding properties compared to the traditional powder-liquid layering technique (Çakırbay Tanış et al. 2020; Moses et al. 2020). TABLE 3 shows some literature studies on SBS. However, in the current study, the zirconia cores were veneered using only one technique; the conventional method. Therefore, it could be speculated that it is the difference in the veneering materials that influence the present results.

The difference in the bonding performances of the veneering ceramics to zirconia may be attributed to the variation in the coefficient of thermal expansion (CTE) values between the bilayered materials (Fischer et al. 2007; Komine et al. 2012). The core and layering materials will expand and shrink with thermal changes during the firing and cooling of porcelain laminates. Ideally, a positive CTE deviation (CTE of core materials higher than overlaying ceramics) is desirable to produce compressive stress on the veneers at room temperature (De Kler et al. 2007; Coffey et al. 1988; Fischer et al. 2009). Nevertheless, additional studies are required to corroborate the cause–effect of CTE on the SBS values obtained in this study.

The fracture interfaces were observed under the scanning electron microscope (FESEM, Zeiss, Supra 55vp, German) with secondary electron imaging and acceleration voltage of 10 kV to gain an effective explanation of the mechanism leading to ceramic/veneer bonding failure. Three forms of fracture patterns (cohesive, adhesive, or mixed mode) were generally observed after the shear testing as shown in Figure 3 and Figure 4. Cohesive failure occurs when the fractures appear predominantly within the ceramics, whereas the disintegration happens at the veneer and zirconia interface considering adhesive failure (Shilpa et al. 2019). The combinations of core and veneer with superior SBS displayed cohesive failures in the veneering porcelain, whereas mixed cohesive and adhesive failures occurred in inferior SBS groups. These findings correspond to observations published by Daud et al. (2017) (Daud et al. 2017).

The cohesive failure occurred in groups of CZ-CK and CZ-VM9, in which the majority area of core surfaces was covered by thick layers of residual veneering ceramics

(Figure 3 (a) and (e)), and pores were observed within the veneers (FIGURE 3 (b) and (f)). The voids may be caused by contamination and the inclusion of bubbles during the manual layering of the veneering ceramics. Internal stresses within the laminates may be developed in the course of veneer sintering due to differences in thermal shrinkages between the core and veneering material (Holden et al. 2009). Thus, these internal impurities propagate the crack that may be formed from residual firing stresses, leading to a cohesive failure (Abdulmajeed et al. 2017; Guo et al. 2018). The group of CZ-e. max with the lowest mean shear strength value produced an adhesive bond failure as shown in Figure 3 (c). The veneer was separated from the core, demonstrating irregularities on the entire surface of the veneer after the shear test as shown in Figure 3 (d).

Meanwhile, in the NZ groups, the combination of NZ-e. max with the lowest mean shear strength value (71.56 MPa) produced a mixed-mode (adhesive/cohesive) bond failure, as depicted in Figure 4 (c). Notably, unlike Cercon®, some areas of adhesion were formed between the NZ and e. max. However, there were corner fractures where tension probably accumulates, indicating the initiation of adhesive failure. Similar to the CZ, NZ veneered to VM9 and CK with high mean shear strength values also exhibited cohesive bond failure. Adhesive failure with generalized residual veneers observed at the periphery of the shearing zone in Figure 4 (a), (c), and (e) was absent. The micrograph results suggest that the novel nano-zirconia achieved good bonding with all veneer groups. However, further evaluation is needed to determine whether these bonding capabilities were afforded by high resistance to phase transformation characteristics of the NZ.

## CONCLUSION

Within the limitations of this study, the novel nano-zirconia achieved similar bonding capabilities to that of its commercial counterpart, when veneered with the tested ceramics. Cohesive failure was observed in the groups with high bond strength value, while the adhesive and mixed mode failure appeared in groups with lower bond strength value. Based on these results, it can be concluded that the slip casted-colloidal processed novel nano-zirconia appears compatible with the available veneering ceramics designed for use with zirconia.

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## DECLARATION OF COMPETING INTEREST

None

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