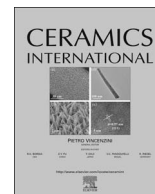




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Evaluation of shear bond strength of a novel nano-zirconia and veneering ceramics

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ABSTRACT

The core–veneer bond strength of zirconia is believed to be influenced by various factors, including zirconia types. To date, studies on bond strength between veneering porcelain and zirconia core fabricated by slip-cast technique are few. The aim of this study was to investigate and compare the shear bond strength (SBS) of three core materials with two veneering porcelains. Sixty cylindrical discs (11 mm in diameter and 3 mm in thickness) were fabricated and divided into Experimental zirconia (Zir), Cercon® (Cer) and metal (Met) core group. Ceramkiss® (Ck) and InLine® (IL) veneering porcelains were fired to each core at a thickness of 3 mm. Before veneering, all core materials were sandblasted with 100 μm Al₂O₃, and a layer of opaque liner was applied in the first firing. The bonded samples were subjected to shear bond test following storage in distilled water (37 °C) for 24 h. Scanning electron micrograph (SEM) was used to identify the mode of bonding failure. The results were analyzed with One-way ANOVA and Independent *t*-test with Tukey post-hoc test. Interaction effect analysis was performed using two-way ANOVA. Met-IL showed the highest mean SBS value (28.9 ± 3.8 MPa), whereas Cer-IL group obtained the lowest value (2.8 ± 0.7 MPa). The mean SBS values of Zir-Ck and Zir-IL were found to be 23.1 ± 2.4 MPa and 4.1 ± 0.9 MPa respectively. When veneered with the same veneering porcelain, both experimental zirconia and Cercon® were comparable, whereas significant differences were observed between metal and zirconia groups. All core materials exhibited significant differences in terms of shear bond strength when veneered with different veneering ceramics. Cohesive failure occurred in the high SBS group, whereas mixed cohesive-adhesive failures were seen in the other groups. The core–veneer SBS is material dependent. The types of core and veneering materials significantly affected the SBS. Slip casted experimental zirconia and commercial zirconia demonstrated similar bonding capabilities with the existing veneering porcelain designed for zirconia core.

1. Introduction

In recent years, zirconia has become widely accepted as the restorative framework material of choice because of its excellent mechanical properties. Zirconia is usually milled into a framework using CAD/CAM (Computer Aided Design/Computer Assisted Manufacturing) technology and is further layered with veneering feldspathic porcelain to form a uniquely strong and aesthetic dental restoration. Comparable survival rate of this restoration to metal-based restoration has been found; approximately 85–100% of the CAD/CAM milled zirconia prosthesis remains functional for 3–7 years [1–5].

Despite the excellent mechanical properties of zirconia framework, technical complication such as veneering porcelain chipping is still continuously reported after the zirconia-ceramic restoration is subjected to loading [2,4,6–8]. A recent study has shown that almost 15% clinical veneer chipping occurred after 7 years [4]; 32% of zirconia-based restorations chipped compared with only 10% of metal-ceramic restorations after 10 years [8]. The causes of clinical chipping are multifactorial, as follows: flaws during fabrication, a low bond strength, excessive occlusal load, and inadequate framework design [2,7,9].

Low bond strength was recorded as the main cause for veneer chipping [9]. Coefficient of Thermal Expansion (CTE) mismatch

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[10,11], veneering porcelain thickness [12], and different types of zirconia core and veneering ceramics, as well as different manufacturers [13] and poor wetting properties of zirconia core [9] played an important role in development of residual stresses and weakening of the bond strength at core–veneering porcelain interface. Bond strength between zirconia and veneering porcelain was between 11 MPa [14] and 34.3 MPa [15], as recorded in several studies for the last 10 years. This wide range of shear values shows that zirconia-ceramic restoration does not have any conclusive minimum bond strength and a standard protocol for the test to be conducted in vitro. Most of the authors have referred to the standard guideline available for metal-ceramic restorations [16–19]. Many authors have tried several methods and materials combinations to produce better core–veneer bonding. In addition, they proposed a better formulation for zirconia manufacturing that could achieve a satisfactory outcome and may reduce the chipping potential.

Recently, a novel nano-zirconia material has been developed by means of controlled colloidal processing and slip cast technique [20]. This is the first time this technique has been used to fabricate 100 wt% nano-zirconia material. This material claimed to produce denser, more homogenous slurry with minimal porosity [20], which is difficult to achieve during commercial zirconia fabrication. Furthermore, mechanical properties of nano-zirconia fabricated by both slip cast and commercial dry-press techniques have shown comparable findings [20–23].

Therefore, there is a great potential for this novel zirconia to be used as an alternative to the currently available dental zirconia and to subsequently reduce the clinical failure. However, a limited study specifically examined the core–veneer bond strength of zirconia fabricated by colloidal process and slip cast technique. The evidence to compare the core–veneer bond strength produced by this technique and other manufacturing methods are also lacking. Thus, the aims of this study were to investigate the bond strength of slip casted experimental nano-zirconia with different commercially available veneering ceramics by shear bond test and to observe the mode of bonding failure.

2. Experimental procedure

2.1. Materials

The study utilized 3 core systems (Metal, Cercon and Experimental zirconia) and 2 veneering materials (InLine and Ceramkiss) with six core–veneer combinations (Fig. 1) where 10 samples fabricated for each combination group. Samples included in the study were free from macroscopic cutting defects after the sintering process, and the desired geometry was achieved (11 mm diameter and 3 mm thickness). Deformed or fractured samples during manufacturing or sintering process with poor attachment to lateral arms of the jig at universal testing machine were excluded from the study.

2.2. Core preparation

The experimental zirconia was fabricated via colloidal process and slip-cast technique. Powder at 40 g (50 nm) was ultrasonically mixed with 45 ml distilled water to form an aqueous solution. Two molar of nitric acid (HNO₃) were added in the mixture to get a more stable pH. Then, 100 ml (0.5 wt%) polyethyleneimine (PEI) were pipetted in the mixture to disperse the agglomerated zirconia particles and stirred under magnetic vibrator (IKA Color Squid Magnetic®) for 45 min. The homogenous aqueous solution was poured into 12 teflon molds (15 mm diameter and 4 mm thickness) and left in situ for 24 h. The samples were eventually sintered at 1400 °C for 2 h to obtain the final geometry [20].

Commercial zirconia discs were prepared by milling 20 Cercon® blocks in CAD/CAM machine (Cercon Brain®). Afterward, all samples were sintered at 1350 °C for 1.5 h to obtain a standardized geometry of

samples. Metal-ceramic combination was used as a control group. The lost wax technique was used to fabricate the metal cores. Wax patterns (20 discs) corresponding to final geometry of samples were fabricated and sprued onto cylindrical holder. De-waxing process was carried out, and the created channel was filled with semiprecious noble metal alloy (Lodestar, Ivoclar Vivadent) during casting. Afterward, the sprued metal was de-invested, ground, and polished with diamond coated polishing burs [18,24,25].

All samples were sandblasted with 110 µm aluminium oxide particles at 4 bars and at 10 mm distance from nozzle to the core surface at a perpendicular position and immediately steam-cleaned to create adequate surface roughness [26–28].

2.3. Veneering process

Prior to veneering the porcelain, a thin layer of veneering opaque was painted on the bonding core surface area and sintered. Then, powder and liquid of the veneering ceramics (InLine® and Ceramkiss®) were mixed using a ratio recommended by the manufacturer to form a slurry mixture. The mixture was then manually packed onto a core surface by using a metal split mold. The air bubbles and water particles were removed by blotting with a clean thin cloth.

The core–veneer assemblies were placed in a furnace (Programat P500, Vivadent®) and fired at specific temperature and condition under respective Ceramkiss® or InLine® programmes [29,30]. Double firing was carried out to compensate the shrinkage to obtain the required 3 mm by 3 mm veneer geometry [24,25]. Slow cooling was applied [31] and all samples were steam-cleaned. Samples were further cleaned with distilled water ultrasonically for 30 min and stored in water at 37 °C for 24 h [24,25].

2.4. Determination of shear strength value

The test was conducted by using Universal Testing Machine (UTM; Shimadzu®, Japan) with a 5 kN load cell. Ortho-polymerized acrylic resin was fabricated to hold the samples to the UTM. Shearing process was carried out at crosshead speed of 0.5 mm per min until all samples debonded (Fig. 2). The bond strengths was calculated [load (N)/area (mm²)] and recorded as megapascals (MPa) [27,32]. Representative debonded samples from every group were subjected to scanning electron microscope (SEM) (Fei Quanta 200). After being sputter-coated with gold-platter for 30 s, the samples were observed at ×50 magnification at the center of the debonded area to observe the mode of bonding failure (adhesive, cohesive, and mixed adhesive/cohesive) [33].

Previous studies [14,25] were carried out with 10–15 samples. This study was designed to have 90% statistical power based on the results from preliminary test with sample size of 10 per core–veneer assemblies. Independent *t*-test was used to analyze the comparison within core groups and one-way ANOVA with Tukey's post-hoc test were used to compare within the veneering porcelain groups. The interaction effects between cores, veneers and core–veneer combinations were determined by two-way ANOVA analysis. The level of significance for all statistical testing was pre-determined at a *p*-value of 0.05 or less.

3. Results and discussion

3.1. Comparison of SBS of slip casted experimental nano-zirconia, commercial nano-zirconia (cercon®) and metal to ceramkiss® and inline® veneering ceramics

Results of this study appeared to be normally distributed based on statistical Shapiro-Wilk test and Kurtosis value for all tested groups that fall between –2 and +2.

The mean SBS values for six core–veneer assemblies are presented in Table 1. Zir-Ck was comparable to Cer-Ck but slightly lower than

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