



Effect of Nanosilica Incorporation on Flexural Strength, Shear Bond Strength, and Color of Veneering Porcelain after Thermocycling

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Abstract

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under the terms of the Creative Commons Attribution-NonCommercial 4.0 International License (CC BY-NC 4.0) **AIM:** The focus of this research was to see how silica nanoparticles changed veneering porcelain over a zirconia core affected flexure strength, shear bond strength, and color.

METHODS: A total number of 30 zirconia core veneer samples were constructed and classified according to silica nanoparticles modification of veneering porcelain into two groups: Group 1 (control group) veneering porcelain without any modification (n = 15) and Group 2 (modified group) veneering porcelain modified by silica nanoparticles (n = 15). Silica nanoparticles were added to the veneering porcelain powder at a rate of 5% by weight. Silica nanoparticles powder and veneering porcelain powder were manually crushed for about 10 min using a pestle and mortar then the mixed powder was combined with the porcelain moldings liquid to make a paste. After thermal cycling, each group was examined for flexural strength, shear bond strength. The color shift was measured using a spectrophotometer.

RESULTS: Flexural strength levels in the modified group $(280.9 \pm 29.85 \text{ Mpa})$ were substantially higher than in the control group $(431.78 \pm 22.73 \text{ Mpa})$. Shear bond strength values in the modified group (34.31 ± 5.6) were significantly higher than in the control group (26.97 ± 4.03) . Color change was within the clinical acceptable range (1.71 ± 0.32) .

CONCLUSIONS: The addition of silica nanoparticles to veneering porcelain improved the flexural and shear bond strength, as well as, color change was within the clinical acceptable limits.

Introduction

Dental zirconia has traditionally been manufactured mainly from tetragonal zirconia crystals with a minor proportion of yttria stabilizer (3Y-TZP); this type is extremely strong but has low translucency. This was accomplished by producing partially stabilized zirconia with a greater yttria concentration, such as 4 mol% (4Y-PSZ) or 5 mol% (5Y-PSZ). The c-phase reduces the stress-induced toughening of zirconia, resulting in reduced strength and toughness. As a result, the most translucent 5Y-PSZ materials in the anterior zone are limited to single unit crowns and short-span fixed dental prostheses (FDPs).

However, high-stress conditions require stronger restorations, such as multiunit posterior restoration and rehabilitating bruxism patients. Consequently, it is essential to enhance the strength of these ultratranslucent materials. Compared to traditional multilayer restorations, monolithic lithium disilicate offers several benefits. Yttrium-tetragonal zirconia polycrystalline (Y-TZP) material can be used in such a case. However, the translucency of ordinary Y-TZP is only around 70% of that of lithium disilicate [1].

Zirconia has drastically boosted the usage

of ceramic restorations in dentistry since its debut as a restorative core material. Zirconia was chosen as the most popular core material due to its exceptional mechanical qualities, high strength, and attractive color [2]. Because zirconia lacks the translucency of real teeth, it needs to be veneered with porcelain to provide better looks.

Clinical trials of zirconia-based all-ceramic restorations have yielded positive outcomes and excellent survival rates. However, with such restorations, chipping, fracturing, and delamination of the porcelain veneer are the most common problems [3]. Overall strength of the connection between both the porcelain veneer and the basic mechanism must be sufficient to transmit operational stresses from the porcelain veneer to the overarching framework [4].

Nanotechnology is the science and art of material engineering on a scale of <100 nm. By increasing the mechanical and physical qualities of materials, it transformed the medical and dentistry sectors.

Nanoparticles are insoluble particles with the benefit of being tiny particles with a larger surface area to volume ratio that has an antibacterial impact [4]. Because of the increased surface area, certain materials synthesized at the nanoscale have different characteristics than those synthesized at the macroscale.

Nanotechnology has opened up new possibilities for improving the use of materials in dentistry, including silica. Outside its usage as a filler component in resins, technology has broadened the dental applications of silica to also include medication delivery to impact cells and develop bacterial resistance through antibacterial qualities, as well as addressing the dental biological interface through intrinsic bioactive capabilities.

Because of its color, luster, and durability, silica has long been utilized in dentistry. A silica-based nanomaterial is now being researched to improve these innate properties [5].

The goal of this research is to find out how integrated silica nanoparticles in dental porcelain veneers affect flexure strength, shear bond strength, and color.

The null hypothesis:

The null hypothesis in this study was that adding silica nanoparticles to veneering porcelain would boost shear bond strength and flexure strength without changing the color.

Materials and Methods

Sample size calculation

Before the study, the number of samples required in each group was determined after a power calculation according to data obtained from the previous study [6]. In that study, the mean flexure strength in the control group was 64.2 ± 11.28 and in the modified group was 100.2 ± 18.41 . A sample size of five samples in each group was determined to provide 80% power for independent samples t-test at the level of 0.05 significance using G Power 3.1 9.2 software.



Figure 1: Samples of shear bond strength

Thirty zirconia core samples were made with a precision cutting machine (IsoMet 4000, Saw Buehler, USA) from 98 × 14 mm pre-sintered zirconia blanks (Upcera, China). For the flexural strength test, 10 samples of dimensions (25 mm length × 1 mm



Figure 2: A sample of flexural strength

thickness × 2.5 mm width) were used [7]. Ten samples for shear bond strength testing (9 mm length × 4 mm thickness × 4 mm breadth) were constructed [8]. For color change testing, 10 samples were used in the shape of disks (12 mm diameter × 1 mm thickness) [9] (Figures 1-3).

The measurements were generated by estimating the shrinkage rate of zirconia, which was 20:22% according to the manufacturer's instructions to compensate for after sintering shrinkage, and then verifying them using a digital caliper.

The specimens were placed in the sintering furnace, and the sintering cycle began with a progressive increase in temperature until the temperature reached 1500°C. The samples were kept at this temperature for 2 h before being cooled to room temperature.



Figure 3: Color measurement sample

Using a delicate digital balance, silica nanoparticles were added to the veneering porcelain powder at a rate of 5% by weight.

In a control grinding operation, silica nanoparticles powder and veneering porcelain powder were manually crushed for about 10 min using a pestle and mortar (Sajjad a. 2019) [10].

According to the manufacturer's directions, the mixed powder was combined with the porcelain moldings liquid to make a paste.

A 50 μ Al₃O₂ particles were sandblasted onto the surface of the zirconia core that would be veneered. The control samples were veneered with unmodified dentin porcelain (vita VM 9, Germany) and burned in a dental porcelain furnace (Ivoclar, Vivadent, Programat P500, Switzerland), according to the manufacturer's instructions. The changed samples were veneered with silica nanoparticles modified porcelain using the same process. The veneering porcelain had a final thickness of 1.5 mm in flexural strength and color change tests. The porcelain thickness was 3 mm in terms of shear bond strength. A digital caliper was used to determine the thickness of the samples.

The incorporation of silica nanoparticles in the altered veneering porcelain was demonstrated using energy dispersive X-ray spectroscopy (EDX) in two representative samples (control and modified). Gold sputtering machine (JEOL, JFC-1100E, Fine coat, USA) was used to coat the samples, which were subsequently analyzed using a scanning electron microscope (SEM) (JSM-IT200, JEOL Ltd., Tokyo, Japan).

All samples (control and modified group) were subjected to thermo cycling in a specifically developed apparatus (THE 100 SD mechatronic thermocycler), which consists of four tanks filled with deionized water and maintained at standard temperatures. To reflect about 6 months of clinical usage, all samples were exposed to 5000 thermal cycles. The samples were submerged in each tank for 15 s in the following order: 5°C to 37°C to 55°C to 37°C according to ISO 11405 standards.

Universal testing machines include electromechanical and hydraulic systems to perform static testing, including tensile, compression, bend, peel, tear, shear, friction, puncture, and other mechanical tests.

Using this universal testing equipment, a 4-point flexural strength test was done on several porcelain samples (control samples and modified samples) (Instron model 3345, England). Computer software was used to calculate and record the data (Bluehill Universal Instron, England). The sample holder had a span between the two bears of 20 mm. The distance between the two loading pistons was 10 mm. Supports and both loading pistons were steel knife edges, rounded to a radius of 1.25 mm. The flexural strength was calculated according to the equation α =3

 $\frac{\text{fd}}{2\textit{bh}}$ where α is the maximum center tensile stress

(MPa), F the load at fracture (N), d the difference in the distance of the two supports and the distance of the two loading pistons (mm), b the width of the specimen (mm), and h is the height of the specimen (mm).

Universal testing equipment was also used to examine the shear bond strength of various materials. Up to specimen failure, compressive mode force was given at a cross head speed of 1 mm/min. By dividing the load (N) at which failure occurred by the bonding area (mm²), the average SBS (MPa) was calculated:

Shear stress (MPa) = Load (N)/19.625 (mm^2)

After debonding, all zirconia cores have been examined using a scanning electron microscope within a week of encasing the scuffed surfaces with gold using a gold sputtering machine and then examined using a USB Digital microscope with a built-in camera connected to an IBM compatible personal computer at a fixed magnification of $65X^{25}$. The failure modes discovered were cohesive fracture within the veneer, adhesive fracture between both the core and veneer, or a mixture of both.

Before each measurement, the spectrophotometer (UV-Shimadzu 3101 PC Spectrophotometer, Japan) was calibrated. Color consistency was assessed by utilizing L*a*b* values to calculate Delta E (E) on a black backdrop. T1 black values served as the baseline against which Delta E comparisons were made.

The overall color was calculated using the algorithm below [11].

 $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$

Where, L* represents brightness, a* represents the green (-a) and red (+a) axes, and b* represents the blue (-b) and yellow (+b) axes.

Flexural strength, shear bond strength, and color measurement test data were gathered, collated, and statistically evaluated.

Results

Energy dispersive X-ray spectroscopy analysis (EDX)

Since the amount of silica in the modified sample raised from 27 to 37, energy-dispersive X-ray spectroscopy examination (EDX) revealed the integration of silica nanoparticles in the modified veneering porcelain (Figures 4 and 5).

Flexural strength test results

The flexural strength of the control and experimental groups was 280.97 ± 29.85 MPa and 431.78 ± 22.73 MPa, respectively. (P <0.001 was considered). In comparison to control samples, experimental samples demonstrated a significant increase in flexure strength after heat cycling (Figure 6).

Shear bond strength test results

Shear bond strength following thermocycling between the control and modified groups:

The control and experimental groups' shear bond strength findings were 26.97 ± 4.03 MPa and

Display name	Standard data	Quantification met	thod Result Type
Spc_001	Standardless	ZAF	Metal
Element	Line	Mass%	Atom%
C	K	13.84±0.32	21.98±0.51
0	K	40.61±0.49	48.41±0.59
Na	K	3.39±0.13	2.82±0.10
AI	К	6.78±0.14	4.79±0.10
Si	К	27.22±0.28	18.49±0.19
К	K	6.82±0.16	3.33±0.08
Ва	L	1.34±0.14	0.19±0.02
Total		100.00	100.00
Spc_001 Fitting ratio 0.0883			
Uteusity [Counts]	SiKa NaKa a E AlKa KKa	BaLr2 BaLb2 BaLb BaLa BaLr	Spc_001
0		5	10
Energy [keV]			

Figure 4: Analysis of the control and modified samples using energy-dispersive X-ray spectroscopy (EDX)

 34.13 ± 5.6 MPa, respectively (p = 0.049). After thermal cycling, shear bond strength in experimental samples increased significantly when compared to control samples (Figure 7).

Color measurement test results

After thermal cycling, the delta E between control and modified samples was 1.71 ± 0.32 , which is within clinically acceptable limits.

Failure pattern analysis results

Two of the control samples exhibited combination failure, whereas the other three samples showed adhesive failure. Three of the modified samples displayed combination failure, whereas the remaining samples showed adhesive failure mode (Figure 8).

Discussion

Nanotechnology has been implemented into several sectors of research because it gives a variety of major solutions to address scientific and medical challenges. The surface area of 1 g of powder may be tested at various spherical diameters, and the surface area per gram increases exponentially below 100 nm.



Figure 5: Analysis of modified sample using energy-dispersive X-ray spectroscopy (EDX)

This causes a phase shift in certain materials, resulting in an increase in surface energy per gram of substance. This increased surface area may be used for a variety of reasons [12].



Figure 6: Flexural strength test between the control and the modified groups after thermocycling

Restorative dentistry, implantology, prosthetic dentistry, endodontics, periodontology, and oral malignancies can all benefit from nanoparticles. Antimicrobial, antiviral, and antifungal activities are all found in nanoparticles. The use of nanoparticles inhibits secondary caries by preventing biofilm from forming on the composite. These nanoparticles increase the total connection between dentin and biomaterials, increasing bond strength. *In vitro* studies have demonstrated



that these nanoparticles reduce crack propagation

and increase fracture toughness in dental ceramics,

preventing porcelain restorations including crowns,

bridges, and veneers from breaking [12].

Figure 7: After thermal cycling, shear bond strength was tested between the control and modified groups

Recently, nano-sized fillers and bioactive nanoceramics have been incorporated into GIC to improve their physical properties. Addition of lower percentage of nanoparticles such as graphene-silver nanoparticle (R-GNs/Ag) nanocomposite, nano titanium dioxide (TiO2), nano silicon dioxide (SiO2), nano zirconium dioxide (ZrO2), nano chitosan, and nano hydroxyapatite in dental materials led to significant increase of mechanical properties, due to the increase



Figure 8: Combined failure mode

in surface area and surface energy, as well as better particle distribution. In this regard, novel nanoparticles with good antimicrobial activities have become more popular in the field of dentistry [13].

Incorporating silver nanoparticles into dental materials may enhance the mechanical features and antibacterial properties of the dental materials. Their efficacy is due not only to their nanoscale size but also to their large ratio of surface area to volume. Silver nanoparticles can be incorporated into acrylic resins for fabrication of removable dentures in prosthetic treatment, composite resin in restorative treatment, irrigating solution and obturation material in endodontic treatment, adhesive materials in orthodontic treatment, membrane for guided tissue regeneration in periodontal treatment, and titanium coating in dental implant treatment [13].

Due to its unique features, such as high stiffness, silica nanoparticles have become a very promising and intensively researched material. Because of their size, surface area, biocompatibility, low toxicity, low density, and adsorption capability, silica-based NPs play an important role in nanotechnology [14].

Zirconia is currently the most used tooth-colored restorative material. Although polyaryletherketone (PEEK) was introduced to the dental market as highperformance and chemically inert biomaterials, the crowns made of zirconia produced 3 times more antagonist wear, maintained higher color stability, and offered the least displacement compared with crowns made of PEEK. The PEEK crowns showed minimal abrasion and better stress modulation through plastic deformation, which makes it a promising material for fabrication of the crown. The clinician can choose the material for the fabrication of dental prostheses according to the application considering its properties, advantages, and limitations [15].

Because of their high opacity, high strength ceramic core materials, such as alumina-based ceramic and zirconia-based ceramic core materials, must be veneered with feldspathic porcelain to mimic the esthetics of a real tooth [16].

Dentistry ceramics have evolved greatly in recent years as one of the most preferred materials for satisfying esthetic needs in dentistry. Bilayered zirconia crowns are more typically used than monolithic zirconia crowns for to esthetic reasons. The bilayered zirconia crown, however, suffers from ceramic chipping because to the difference in thermal expansion coefficient, shear bond strength, and tensile strength [17].

The study employed silica nanoparticles with an average size of 50 nm since the smaller the particle, the greater the surface area and hence the larger surface energy [18], [19].

Silica nanoparticles were added to the veneering feldspathic porcelain in a 5% by weight ratio because Nathan *et al.* found that 5% had the maximum value compared to 2.5% and 7.5%. Even though excessive nano zirconia silica fibers might cause so much voids due to nanofiber agglomeration, the fracture toughness value decreased when the samples were reinforced with 7.5 wt% nanofibers; thus, trying to increase the nanoparticles percentage results in greater capacity to a certain restriction, after which any rise mostly in percentage of nanoparticles results in a decrease in the strengths [20].

Using a mortar and pestle, the silica nanoparticles were combined with the veneering porcelain. One of the most widely used small-scale mixing equipment is the mortar and pestle. Comminution and mixing are combined in a single action with the mortar and pestle technique. As a result, it is particularly beneficial when particle size reduction and mixing are both necessary. Mortars and pestles are composed of the same materials as mortars and come in a variety of forms and sizes [10].

Because it replicates intraoral temperature variations and chewing pressures, thermocycling was chosen. Thermocycling was performed at regulated temperatures to imitate the dynamic environment of the oral cavity during function and to test the material's long-term efficiency [21].

For characterization, EDX analysis was performed since it produces a spectrum with peaks connected to the elemental makeup of the material under investigation. This characterization approach may also be used to produce a sample's elemental mapping.

Flexural strength was chosen as the mechanical attribute to be measured in this study because it is critical in predicting the performance of a material, especially one that is delicate, and it is heavily influenced by defects and flow (2017, Elsheemy *et al.*) [22].

This study used a 4-point flexural strength test because it produces a better flexural test result than

a 3-point load test because a 3-point bending test produces peak stress at the material mid-point and reduced stress elsewhere, whereas a 4-point bending test produces peak stresses across a larger region of the material, thus exposing a longer length of the material [23].

In the present study, the bond strength was evaluated using shear bond strength; the force application was to the nearest point to the two materials connection and parallel to it till the two materials become separated [24], [25], [26]. The ease of preparing samples and executing the test, the necessity for technical precision is not as stringent as in micro tests, the test can be readily replicated when necessary, and it is a test technique that can be performed fast are all reasons why this approach is the most popular [25], [27].

Spectrophotometers and colorimeters can be used to assess the look of esthetic restorations and the natural color of teeth. Spectrophotometers detect the wavelength that is reflected or transmitted from a single item at a time, unaffected by subjective color interferences. Colorimeters, on the other hand, provide a broad estimate of the amount of light retained.

A spectrophotometer is a device that detects shade differences and converts measurements into numerical data. Accuracy, the capacity to examine the major components of a series of spectra, and the ability to convert data to multiple color measurement systems are all advantages of this equipment [28].

Contrasts in the color coordinates L^* , a^* , and b^* determine color difference (E). Computerized color analysis allows for color assessment of dental restorative materials without the subjectivity of human observation.

Measurement of color variations of a tooth in the oral environment includes the following classification for the clinical importance of the observed ΔE_{ab} value, according to basic colorimetric principles that have been established for years: (<1: Undetectable color changes, $1 \le \Delta E \le 3.7$: Clinically minor observable difference, and >3.7: Clinically substantial visible difference) [29].

Sandblasting was used to treat the zirconia substructure in this investigation since Harding *et al.* (2012) found that using a liner lowers the zirconia-veneering porcelain bond strength when compared to sandblasting [30].

The results of this study showed that incorporating silica nanoparticles increased flexural strength in experimental samples after thermocycling when compared to control samples and that this improvement in strength can be attributed to a reduction in particle size, which increases surface area and thus massive surface energy. As a result, stress concentration at the NPs/matrix interphase decreases, resulting in greater flexural strength values for the associated material [18], [19] The results of our flexural strength tests were consistent with those of Salman et al., 2017, who found that adding silica nano particles to polymethyl methacrylate increased its flexural strength and that this improvement can be attributed to the interfacial strength between nanoparticles and matrix created by crosslink bonding covering the nanoparticles fillers, which prevents crack propagation [31].

The results of Kanat *et al.* who evaluated the flexural strength of veneered zirconia with different veneering processes were similar to the flexural strength test values of the veneered zirconia without any alteration in our study [32].

After thermal cycling, the insertion of silica nanoparticles enhanced the shear bond strength in modified samples compared to control samples, according to the findings of this study. According to recent investigations by Jowkar *et al.* and Fattah, the introduction of nanoparticles increases the homogeneity and improves the consistency of the material [33].

Because of weak bonding between the porcelain and the zirconia substrate, the control samples mainly displayed adhesive failure mode at the interface between the veneering porcelain and the zirconia substrate. The modified samples exhibited a mixed failure mechanism mainly, with adhesive failure at the interface and cohesive failure in the veneering porcelain. Three samples of the modified samples showed a mixed failure mechanism, which might be due to porosities in the veneering ceramics acting as stress stem sites. After SEM investigation of zirconia press on veneer interface, Aboshelib et al. discovered porosities-free veneer. As a result, the veneering process may impact the mean bond strength of the veneering ceramic by influencing the existence of pores within the veneering ceramic [2].

The results of this study showed that adding 5 wt percent silica nanoparticles to zirconia-veneer samples did not change the color of the veneering porcelain to some extent, because the average measured Δ Eab values were 1.71 ± 0.32, which were significantly lower than the value 3.7, which is clinically undetectable, and this indicates that silica nanoparticles addition to zirconia-veneer samples did not change the color.

This might be explained by the fact that materials with the smallest reinforcing agents have the highest overall light transmission and the least amount of light scattering within the material. The findings of this study are consistent with those of Alexandros *et al.* [34].

In contrast, Kotanidis *et al.* reported that using SiO_2 nanoparticles to reinforce PMMA resin for interim restorations resulted in significantly lower color change as measured by the ΔEab value when compared to the upper clinically recommended limit, and they attempted to explain that the filler average size was in the nanometer scale (12 nm), which was much shorter

than the average size of those used in the studies of Arikawa *et al.* and Mami et al. The cause, they added, is the formation of non-spherical nanoparticle agglomerates [34].

The null hypothesis is totally accepted within the scope of the current investigation.

Because all of the dynamic changes that occur in the oral environment cannot be repeated, the results obtained are likely to be influenced; an *in vivo* investigation is advised in the future study.

Conclusions

The addition of silica nanoparticles to the veneering porcelain improves its flexural strength significantly.

The use of nanosilica improves the veneering porcelain's shear bond strength to the zirconia substrate.

The addition of silica nanoparticles had no effect on the color of the veneering porcelain.

Recommendations

- 1. Different types of veneering porcelain were needed to be tested and also their different application processes are required.
- 2. Silica nanoparticles with different sizes and mixing ratios are required for testing.

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