

# Effect of Zirconia Silica Nanofibers on Flexural Strength of Feldspathic Ceramic - An Experimental Study

## Abstract

**Background:** Dental ceramics exhibit good optical and esthetic properties due to their translucency. Feldspathic ceramic is the most widely used veneering ceramic with brittleness, which accounts for most of its failure. Hence, this study was done to evaluate and compare the flexural strength of feldspathic ceramic reinforced with zirconia-silica nanofibers in the ratio of 2.5, 5, and 7.5 wt% with conventional feldspathic ceramic. **Materials and Methods:** According to ISO 6872, a master die was prepared from which resin bars were fabricated with 4.0 mm in width × 1.2 mm thickness × 25.0 mm length, Zirconia-silica nanofibers were produced by sol-gel electrospinning go around with calcination and blended with feldspathic ceramic through ball milling method. The samples were prepared with 0, 2.5, 5, 7.5 wt% nanofibers reinforced ceramic. The flexural strength of the samples was evaluated using three-point bending test. **Results:** The flexural strength values of zirconia-silica nanofibers reinforced ceramic groups were higher than control group. There was a gradual increase in the flexural strength values of feldspathic ceramic groups with increase in wt% of nanofibers. **Conclusion:** The flexural strength of feldspathic ceramic samples reinforced with zirconia-silica nanofibers by 5 and 7.5 wt% were statistically significant compared to control, whereas the flexural strength of 2.5 wt% was statistically insignificant compared to the control group.

**Keywords:** Ceramics, dental esthetic, flexural strength, nanofibers, zirconium

## Introduction

Dental ceramics are nonmetallic, inorganic material mainly consists of oxygen with one or more metallic or semimetallic elements. They are composed of three-dimensional network of covalent bonds between the silica tetrahedra.<sup>[1]</sup> They exhibit chemical, mechanical, physical, and thermal properties that distinguish them from other materials such as metals and resins.

Ceramic is a brittle material that leads to its failure. The latest researches in materials and fabrication methods made the ceramics withstand disruptive stresses such as compressive and shear stresses. Nanotechnology integrated into dentistry enables a new stream of development such as nanoparticles, nanofibers, nanotubes, and nanofilaments into the material part of dental science. Nanofibers are fibers with diameter  $\leq 100$  nm.<sup>[2]</sup>

Recently, zirconia and zirconia-silica nanofibers have been used as reinforcement material for dental composites in restorative

dentistry without any adverse effects. The mechanical properties of composite resin have been enhanced by the incorporation of other materials with ceramic.<sup>[3,4]</sup> Hence, this study was done with the objective of evaluate and compare the flexural strength of feldspathic ceramic reinforced with zirconia-silica nanofibers at 2.5, 5, 7.5 wt%. A hypothesis was formulated that the flexural strength of feldspathic ceramic would be such as the zirconia-silica nanofibers reinforced ceramic.

## Materials and Methods

An experimental study was done in the laboratory for which a master die was prepared according to ISO 6872 with the dimensions of 4.0 mm width × 1.2 mm thickness × 25.0 mm length, it was duplicated in addition silicone impression material (Aquasil, Dentsply, Germany) in putty consistency to get a mold space and auto-polymerizing PMMA resin (DPI Cold cure, Mumbai, India) was used to fabricate the resin bars. The samples were grouped as Group A (control), Group A1, Group A2,

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and Group A3 based on incorporation of zirconia-silica nanofibers by 0, 2.5, 5, and 7.5 wt%, respectively. A total of twenty samples were prepared.

Distilled water was added to ceramic (IPS Classic V Dentin Body, Ivoclar) and condensed with piston of a syringe and blotting paper. It was carefully detached from the syringe tube and sintered at 900 degree centigrade to produce feldspathic ceramic pellets. A sprue was fixed with the resin bar and invested in phosphate bonded investment (IPS Press Vest Speed, BEGO, Germany) then burnout was done. The preformed feldspathic ceramic blocks were melted and ejected into the mold space under pressure and high temperature.

Zirconia-silica nanofibers were prepared using sol-gel electrospinning (ESPIN-NANO, PECO, Chennai) method. Zirconia-silica nanofibers were incorporated into feldspathic ceramic in the ratio of 2.5%, 5.0%, and 7.5% by weight. The blending of nanofibers to feldspathic was done through ball milling, then mixed with distilled water separately, then condensed and sintered at 90°C to produce ceramic pellets. The pellets were condensed under pressure at high temperature into the mold space and subjected to sintering.

The flexural strength was investigated using three-point bending test in an UTM (Autograph universal testing machine, Shimadzu corp, Japan) at 1 mm/min a cross-head speed. Flexural strength (M) =  $3WI/2bd^2$  MPa where M W = fracture load (N); I = distance between support points (mm); b = width of sample (mm); and d = thickness of sample (mm). The surface of the fractured samples was viewed under standard error of the mean (SEM) (Phenom ProX, Phenom-World BV, Netherland).

## Results

The flexural strength values obtained in the study were statistically analyzed using the software SPSS version 22.0. (IBM; Armonk, New York, United State) The mean flexural strength of each group was calculated, and the normality of the data was analyzed using the Kolmogorov-Smirnoff test. The flexural strength values did not follow normal distribution, therefore nonparametric tests were applied to analyze the flexural strength. Comparison between all the four groups was done using Kruskal-Wallis test and Mann-Whitney test, with Bonferroni correction applied for pair-wise group comparisons.

The mean flexural strength and standard deviation of Group A, Group A1, Group A2, and Group A3 were  $141.08 \pm 31.27$ ,  $176.70 \pm 5.51$ ,  $189.07 \pm 5.52$ , and  $196.71 \pm 5.25$  Mpa, respectively [Table 1]. Comparison of flexural strength between groups A, A1, A2, and A3 was done using Kruskal-Wallis test [Table 2]. The significant value *P* was 0.001, which was <0.05, hence it was considered statistically significant. Pair-wise comparison of flexural strength of group A, A1, A2, and A3 was done with MannWhitney test with Bonferroni correction [Table 3]. The comparison of flexural strength

between Group A against Group A2 and Group A against Group A3 showed that the statistically significant value *P* < 0.05, hence it was considered as statistically significant, but it was insignificant for comparison with other groups. The mean flexural strength of the test groups was higher than control group. It was highest for Group A3 (7.5 wt% nanofibers), followed by Group A2 (5 wt% nanofibers) and Group A1 (2.5 wt% nanofibers) [Graph 1].

Under SEM, feldspathic ceramic showed smooth areas that were related to the glassy matrix. These irregular areas were related to the presence of leucite clusters dispersion heterogeneously throughout the glassy matrix of feldspathic ceramic with dendritic shape and porosities of different diameter [Figure 1]. The test group revealed clusters of Zirconia silica fine nanofibers dispersion in some regions of the glassy matrix along with porosities of different diameter [Figure 2].

## Discussion

Dental ceramics possess high esthetic and wear resistance properties utilized the ceramics for various restorative dental procedures.<sup>[5]</sup> In the present-day, the increased demand for highly esthetic and natural-appearing restorations led to the blooming of new all-ceramic materials with improved mechanical properties.<sup>[6-8]</sup>

On reviewing the literature<sup>[1,9,10]</sup> several methods were employed to strengthen ceramics such as chemical strengthening methods by exchange of smaller alkali

**Table 1: Mean and standard deviation of flexural strength**

Flexural strength	Group A	Group A1	Group A2	Group A3
<i>n</i>	5	5	5	5
Mean	141.08	176.70	189.07	196.71
SD	31.27	5.51	5.52	5.25

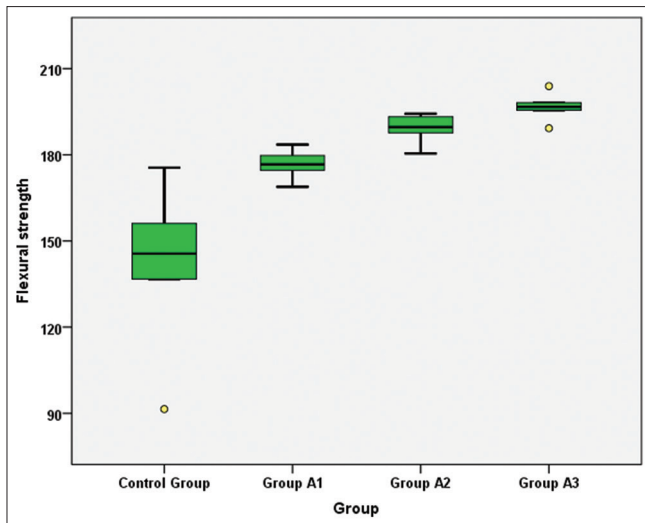
SD: Standard deviation

**Table 2: Comparison of flexural strength of samples using Kruskal-Wallis test**

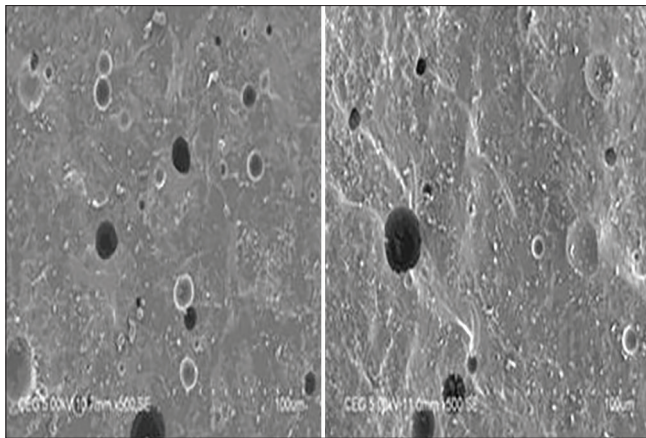
Variable	Group	<i>n</i>	Mean difference	<i>P</i>
Flexural strength	Group A	5	3.40	0.001
	Group A1	5	7.80	
	Group A2	5	13.40	
	Group A3	5	17.40	

**Table 3: Pairwise comparison of flexural strength using Mann-Whitney test with Bonferroni correction**

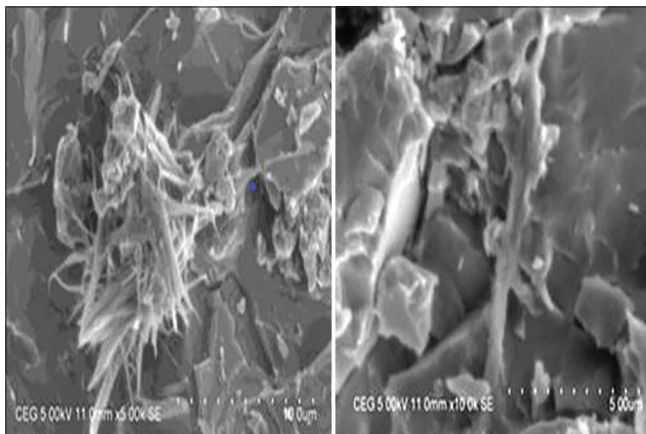
Group	<i>P</i>
Control Group A versus Group A1	0.999
Control Group A versus Group A2	0.045
Control Group A versus Group A3	0.001
Group A1 versus Group A2	0.807
Group A1 versus Group A3	0.062
Group A2 versus Group A3	0.999



**Graph 1: Comparison of flexural strength (Mpa) of conventional pressable feldspathic ceramic with zirconia-silica nanofibers reinforced pressable feldspathic ceramics by 2.5, 5, and 7.5 wt%**



**Figure 1: Standard error of the mean image of fractured surface of control sample**



**Figure 2: Standard error of the mean image of fractured surface of test sample**

ions for larger ions, crystalline incorporation to interrupt crack propagation, thermal tempering, and transformation toughening. Glazing was the rarely used method to

strengthen ceramics which depends upon the formation of a low expansion outer layer at the elevated temperature.

The previous studies<sup>[11-18]</sup> described various techniques to reinforce veneering or metal ceramics. Nanoscience is the manufacturing of materials in billions of meters or nanometer to 2–3 atoms. The latest research evidenced that the reinforcement of nanofibers alters the physical properties and also improves the mechanical properties of ceramic materials.<sup>[19,20]</sup>

In this study, the flexural strength of ceramic reinforced with zirconia silica nanofibers at 2.5%, 5%, and 7.5 wt% were  $176.70 \pm 5.51$ ,  $189.07 \pm 5.52$ , and  $196.71 \pm 5.25$  Mpa, respectively. The 7.5 wt% zirconia-silica nanofibers reinforced samples had higher values followed by samples reinforced with 5 wt% zirconia-silica nanofibers then samples reinforced with 2.5 wt% zirconia-silica nanofibers. This shows that there was improvement in the flexural strength with zirconia-silica nanofibers incorporation. Hence, this study rejected the null hypothesis.

Previous study<sup>[21]</sup> results showed the flexural strength of ceramic integrated with nano zirconia at 5%, 10%, 15%, and 20% were 79.5, 79.1, 92.0, and 96.9, and 92.9 MPa. In this study, the rise in flexural strength of zirconia-silica nanofibers integrated feldspathic ceramic was due to the cross over (bridging) the fracture area. Microcrack propagation in dental ceramic can be resisted by the nanofibers across the crack planes and support the subjected load. Therefore, crack initiation is tolerated by the bridging nanofibers in the ceramic. The phase transformation mechanism of zirconia also had a major role in the toughening effect of ceramic. While propagating the crack, the concentrated stress at the crack spot enables tetragonal crystals of zirconia to transform to stable monoclinic zirconia which tends to close the crack.<sup>[3]</sup>

### Clinical implication

The zirconia-silica nanofibers reinforced feldspathic ceramics have the property of bridging the crack area which further prevents crack propagations. Hence, it is recommended in the areas where subjected to high stresses in the tooth areas.

### Conclusion

The flexural strength of zirconia silica nanofibers reinforced feldspathic ceramic by 5 and 7.5% weight were  $> 2.5$  wt% and the conventional feldspathic ceramic.

### Author agreement

The authors declared that we did not have receive any funding for this research.

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### Conflicts of interest

There are no conflicts of interest.

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