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ORIGINAL RESEARCH

# Comparative Evaluation of Flexural Strength of Nano-zirconia-integrated Pressable Feldspathic and Lithium Disilicate Ceramics

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## ABSTRACT

**Aim:** The purpose of the study was to evaluate and compare the flexural strength of nano-reinforced zirconia feldspathic porcelain, lithium disilicate ceramics, and zirconia.

**Materials and methods:** Ten bar-shaped specimens of computer-aided design (CAD)/computer-aided manufacturing (CAM) zirconia, reinforced feldspathic porcelain, and reinforced lithium disilicate were fabricated in accordance to International Organization for Standardization (ISO 6872; n = 10). Feldspathic porcelain and lithium disilicate ceramic specimens were reinforced with 5, 10, 15, and 20% of zirconia nanoparticles through a customized technique. The specimens were subjected to three-point flexural strength test using universal testing machine (UTM) and examined for crack propagation using a scanning electron microscope (SEM). Oneway analysis of variance (ANOVA) and Tukey test were used to analyze the data ($p < 0.05$).

**Results:** The flexural strength of feldsphatic porcelain increased with the increase in the concentration of zirconia particles. The mean flexural strength of 5, 10, 15, and 20% nano-zirconia-incorporated lithium disilicate was 93.8, 97.1, 100.6, and 100.8 MPa respectively, and was lower than the control group (221.7 MPa). A significant difference in the flexural strength was found with the incorporation of nano-zirconia particles.

**Conclusion:** The flexural strength of zirconia-integrated feldspathic porcelain increased and lithium disilicate ceramics decreased with the nano-zirconia reinforcement.

**Clinical significance:** The simplified approach of reinforcing feldspathic porcelain with zirconia nanoparticles can be adapted in clinical situations of higher masticatory forces.

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## INTRODUCTION

Dental ceramics are widely preferred material in restorative and rehabilitation dentistry. However, the limitations, such as reduced plastic deformation, brittleness, low fracture resistance, and decreased impact toughness have restricted its applications, especially on posterior teeth.[1,2] To triumph over the weakness of ceramics and growing patient's demand for esthetic and natural-appearing restorations, research is leading to the development of advanced ceramic materials whose mechanical characteristics have been dramatically improved to provide suitable longevity and reduced technical shortcomings.[3-6] A significant upgrading in clinical performance was initiated with lithium disilicate pressable ceramics which displayed elevated flexural strength and appealing translucency than the conventional ceramics. Development of machinable ceramics improved mechanical properties and brought the best interest of all ceramics to prosthetic dentistry.[7,8] Various researches had led to the advent of polycrystalline zirconium dioxide ($ZrO_2$), and yttrium-stabilized tetragonal zirconia polycrystal-stabilized material was the most popular and frequently used form of zirconia for dental applications. The advent of machinable ceramics had satisfied the mechanical and esthetic properties of dental ceramics; however, their clinical usage was not increased due to high cost and complex equipments required to fabricate the framework.

Lacunae persist in utility and application of older systems, especially the pressable system which had lesser disadvantages. The introduction of nanotechnology in dentistry drastically improved the properties of the materials used. Nanotechnology was the science of manipulating matter measured in the billionth of meters or nanometer roughly to the size of two to three atoms. Nanoproducts were manufactured to homogeneously distribute in the ceramics to produce nanocomposites. The addition of nanoparticles not only changes the physical properties but also influences the mechanical properties of the material, such as strength, stiffness, and elasticity; reduces weight; and improves functional quality of the material.[9] The effective employment of nanoparticles to improve the properties of conventional ceramics is limited in literature.[10-13] This study evaluated and compared the flexural strength of reinforced feldspathic porcelain and lithium disilicate ceramics with various percentages of zirconia nanoparticles.

## MATERIALS AND METHODS

Ten samples were prepared for each group according to ISO 6872. Custom metal dies were fabricated for standardizing the samples approximately 4.0 mm in width × 1.2 mm thickness × 25 mm in length.

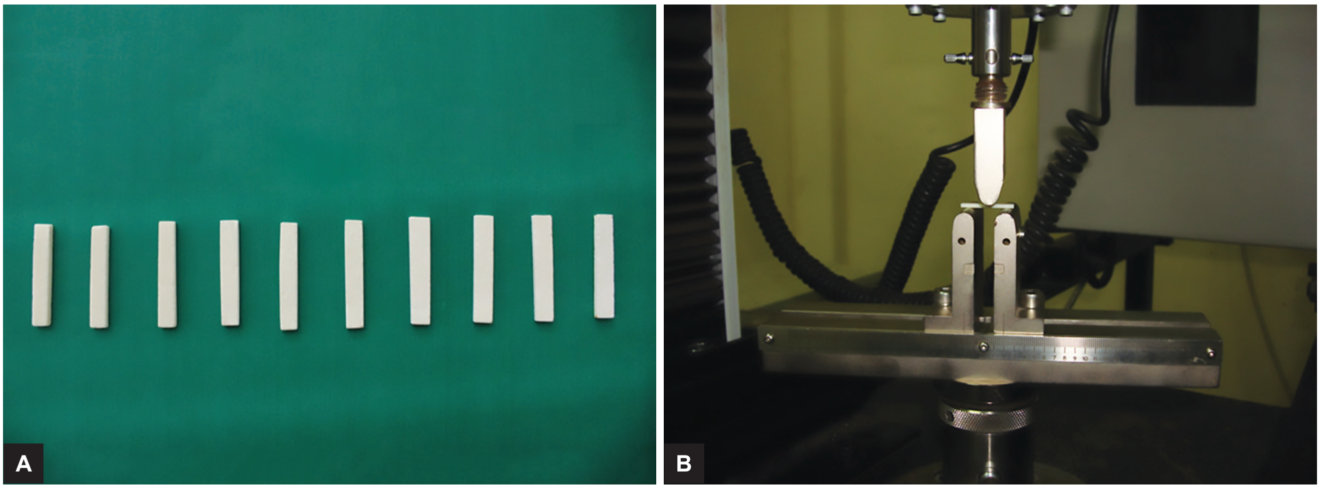
Three groups of samples were prepared and labeled as groups I, II, and III. Group I samples were prepared from conventional feldspathic porcelain (IPS-Classic V Dentin Ivoclar Vivadent, Liechtenstein) through pressable technique. About 100 mg of feldspathic powder was mixed with water to form powder slurry which was condensed to produce ceramic pellets. A 10 mL syringe was used to condense the powder slurry. The condensed samples were detached from the tube and sintered at 900°C to produce feldspathic ceramic pellets.[14] The samples were prepared from sintered pellets through pressable technique using resin pattern made from the dies. The specimens were then reduced to a final dimension with a diamond bur on a high-speed handpiece, followed by grinding with a rubber wheel and polished with SiC abrasive disks. The dimensions of the samples were evaluated using digital caliper and labeled as group I (control group). The specimens were cleaned with distilled water in an ultrasonic cleaner for 5 minutes, stored dry, and then immersed in room temperature (23°C) distilled water for 24 hours before testing.

The zirconia nanopowder of size 45 nm (Sisco laboratory, Chennai) was added to conventional feldspathic ceramic through planetary milling in the ratio of 5, 10, 15, and 20% by weight. The samples prepared from this mixture were grouped as I1, I2, I3, and I4 respectively (Fig. 1A).

Samples prepared from lithium disilicate-pressable ingots (Ivoclar e-max Vivadent, Liechtenstein) through pressable technique using resin pattern dies were labeled as group II (control group). The lithium disilicate ingots were quenched at 850°C to produce amorphous crystals which were the ball milled (jar milling) for 12 hours to get uniform particles. The nano-zirconia reinforcement particles of 5, 10, 15, and 20% were added to the ball-milled particles and mixed by planetary milling. The samples were prepared from this mixture through pressable technique and were labeled as groups II1, II2, II3, and II4 in accordance with the reinforcement.[15]

The samples prepared for zirconia group were dry milled to the designated dimension in the CAD/CAM machine and sintered at ceratherm for 1,200°C for 12 hours and labeled as group III to serve as control group.

The samples were subjected to a three-point bending test on a UTM at a cross-head speed of 1.0 mm/min. The test span was measured between the two hardened steel cylindrical supports. The load was applied at the midpoint between the supports by means of a third hardened steel cylinder. The load required to break the test piece was measured to the nearest 0.1 N (Fig. 1B). The mean flexural strength for each group was derived, and the data



**Figs 1A and B:** (A) Samples, (B) testing of samples

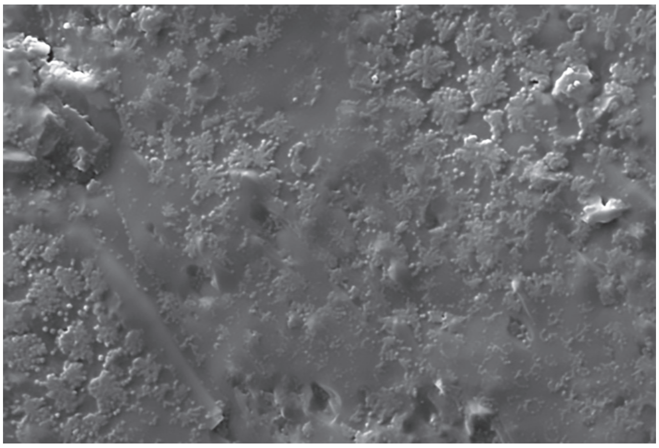


**Fig. 2:** Scanning electron microscope—fracture surface of feldspathic control group

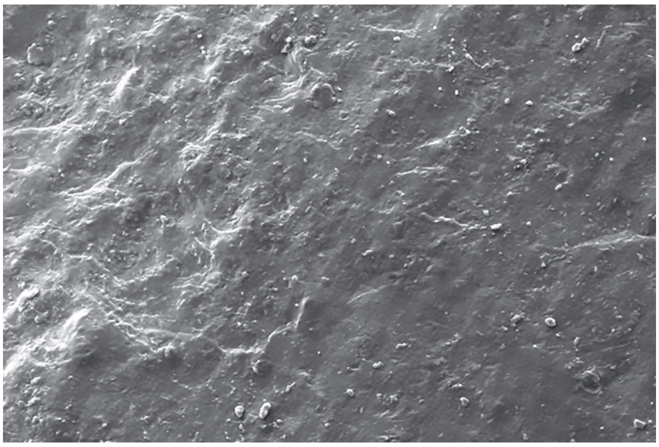


**Fig. 3:** Scanning electron microscope—fracture surface of nano-zirconia integrated feldspathic group

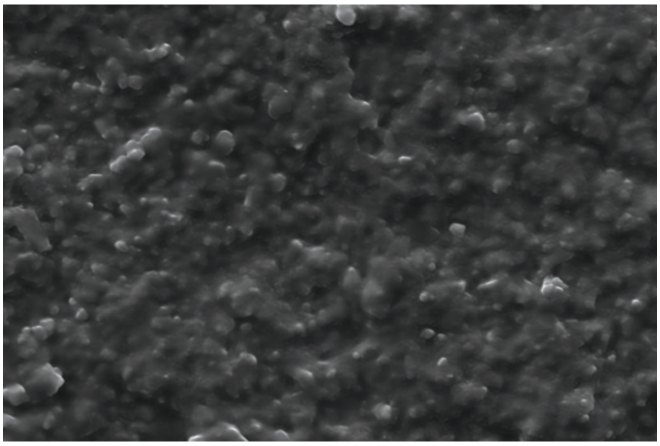


**Fig. 4:** Scanning electron microscope—fracture surface of lithium disilicate ceramic (control group)

**Table 2:** Statistical analysis of flexural strength of feldspathic ceramics

| *Groups* | *n* | *Mean* | *Standard deviation* | *Standard error* |
|---|---|---|---|---|
| Group I | 10 | 79.710 | 4.1010 | 1.2978 |
| Group I1 | 10 | 82.500 | 3.9875 | 1.2610 |
| Group I2 | 10 | 84.020 | 4.9835 | 1.5759 |
| Group I3 | 10 | 96.870 | 9.2174 | 2.9148 |
| Group I4 | 10 | 92.980 | 5.8733 | 1.8573 |
| Total | 50 | 87.216 | 8.7429 | 1.2364 |
|  | *Sum of squares* | *df* | *Mean square* | *Significance* |
| Between groups | 2,152.185 | 4 | 538.046 | 0.000 |
| Within groups | 1,593.302 | 45 | 35.407 |  |
| Total | 3,745.487 | 49 |  |  |

df: Degree of freedom

were analyzed statistically using one-way ANOVA. The fractured surface of the ceramic samples was analyzed under SEM under specific magnification to identify the dispersion of particles and voids (Figs 1 to 4).

## RESULTS

The mean flexural strength values of control group samples of feldspathic, lithium disilicate, and CAD/CAM-milled zirconia bars were 79.7, 222.6, and 800.2 MPa respectively (Table 1). In the reinforced feldspathic group, the mean value of group I was 79.7 MPa, group I1 was 82.5 MPa, group I2 was 84.02 MPa, group I3 was 96.9 MPa, and group I4 was 92.98 MPa (Table 2). The ANOVA test (Table 2) revealed sum of squares, and mean squares between groups were 2152.185 and 538.046 respectively, a significant difference ($p < 0.000$) was determined. The flexural strength significantly increases with the increase in reinforcement with $p < 0.000$ (Tables 2 and 3).

**Table 1:** Flexural strength of pressed feldspathic ceramic, lithium disilicate ceramics, and CAD/CAM-milled zirconia bars—control group

| *Type of ceramic* | *Groups* | *Flexural strength (MPa)* |
|---|---|---|
| Feldspathic | I | 79.7 |
| Lithium disilicate | II | 221.7 |
| CAD/CAM-milled zirconia | III | 800.2 |

The mean flexural strength of lithium disilicate-reinforced sample and comparison between groups is listed in Tables 4 and 5. The flexural strength was significantly reduced with the incorporation of nano-zirconia reinforced lithium disilicate group when compared with the control group ($p < 0.000$). The fractured surface of the samples was analyzed using SEM, and surface characteristics were evaluated for the presence of porosities and particle distribution.

## DISCUSSION

The advent of machinable ceramics reduced the use of feldspathic porcelain and pressable ceramic system. Over the decades, it has satisfied the mechanical and esthetic properties for clinical use, and additionally, it requires

**Table 3:** Comparison of flexural strength of feldspathic ceramics integrated with $ZrO_2$ nanopowder within the group

| *(I) Group (J) Group* | *Mean difference (I–J)* | *Standard error* | *p-value* |
|---|---|---|---|
| *Group I* | | | |
| Group I1 | −2.7900 | 2.6611 | 0.831 |
| Group I2 | −4.3100 | 2.6611 | 0.493 |
| Group I3 | −17.1600 | 2.6611 | 0.000 |
| Group I4 | −13.2700 | 2.6611 | 0.000 |
| *Group I1* | | | |
| Group I | 2.7900 | 2.6611 | 0.831 |
| Group I2 | −1.5200 | 2.6611 | 0.979 |
| Group I3 | −14.3700 | 2.6611 | 0.000 |
| Group I4 | −10.4800 | 2.6611 | 0.003 |
| *Group I2* | | | |
| Group I | 4.3100 | 2.6611 | 0.493 |
| Group I1 | 1.5200 | 2.6611 | 0.979 |
| Group I3 | −12.8500 | 2.6611 | 0.000 |
| Group I4 | −8.9600 | 2.6611 | 0.013 |
| *Group I3* | | | |
| Group I | 17.1600 | 2.6611 | 0.000 |
| Group I1 | 14.3700 | 2.6611 | 0.000 |
| Group I2 | 12.8500 | 2.6611 | 0.000 |
| Group I4 | 3.8900 | 2.6611 | 0.592 |
| *Group I4* | | | |
| Group I | 13.2700 | 2.6611 | 0.000 |
| Group I1 | 10.4800 | 2.6611 | 0.003 |
| Group I2 | 8.9600 | 2.6611 | 0.013 |
| Group I3 | −3.8900 | 2.6611 | 0.592 |

**Table 5:** Comparison of flexural strength of lithium disilicate ceramics integrated with $ZrO_2$ nanopowder within the group

| *(I) Group (J) Group* | *Mean difference (I–J)* | *Standard error* | *p-value* |
|---|---|---|---|
| *Group II* | | | |
| Group II1 | 127.8300 | 8.1489 | 0.000 |
| Group II2 | 124.5700 | 8.1489 | 0.000 |
| Group II3 | 121.0200 | 8.1489 | 0.000 |
| Group II4 | 120.8800 | 8.1489 | 0.000 |
| *Group II1* | | | |
| Group II | −127.830 | 8.1489 | 0.000 |
| Group II2 | −3.2600 | 8.1489 | 0.994 |
| Group II3 | −6.8100 | 8.1489 | 0.918 |
| Group II4 | −6.9500 | 8.1489 | 0.912 |
| *Group II2* | | | |
| Group II | −124.5700 | 8.1489 | 0.000 |
| Group II1 | 3.2600 | 8.1489 | 0.994 |
| Group II3 | −3.5500 | 8.1489 | 0.992 |
| Group II4 | −3.6900 | 8.1489 | 0.991 |
| *Group II3* | | | |
| Group II | −121.0200 | 8.1489 | 0.000 |
| Group II1 | 6.8100 | 8.1489 | 0.918 |
| Group II2 | 3.5500 | 8.1489 | 0.992 |
| Group II4 | −0.1400 | 8.1489 | 1.000 |
| *Group II4* | | | |
| Group II | −120.8800 | 8.1489 | 0.000 |
| Group II1 | 6.9500 | 8.1489 | 0.912 |
| Group II2 | 3.0900 | 8.1489 | 0.991 |
| Group II3 | 0.1400 | 8.1489 | 1.000 |

**Table 4:** Statistical analysis of flexural strength of lithium disilicate ceramics

| *Group* | *n* | *Mean* | *Standard deviation* | *Standard error* |
|---|---|---|---|---|
| Group II | 10 | 221.660 | 32.5917 | 10.3064 |
| Group II1 | 10 | 93.830 | 11.7729 | 3.7229 |
| Group II2 | 10 | 97.090 | 12.5576 | 3.9711 |
| Group II3 | 10 | 100.640 | 13.3780 | 4.2305 |
| Group II4 | 10 | 100.780 | 11.0897 | 3.5069 |
| Total | 50 | 122.800 | 52.9612 | 7.4898 |
| | *Sum of squares* | *df* | *Mean square* | *Significance* |
| Between groups | 122495.1 | 4 | 30623.777 | 0.000 |
| Within groups | 14944.234 | 45 | 332.094 | |
| Total | 137439.3 | 49 | | |

df: Degree of freedom

more strength to match the newer-generation ceramic systems.[1-3] The evolution of ceramics, techniques, and advancements in technology provided superior ceramic material and properties, but lacunae persisted in literature as no nano-reinforcement methods were tried to improve the properties of conventional system.[7] The improvement in the strength can enhance the applications and can produce most cost-effective restorations. Hence, this study was done to evaluate an increase in the flexural strength of feldspathic and lithium disilicate ceramics with the incorporation of nano-zirconia particles. Zirconia, being more ideal and proven material for ceramic reinforcement, was added in the form of nano-zirconia particles that displayed superior strength and material properties.[8,12,14]

The study employed a customized procedure in formulating a pressable ingot rather than powder slurry technique because the sintered ceramic pellets had high density when pressed under high pressure and temperatures and fabricate samples of desired dimension effortlessly with a reduced number of voids compared with powder slurry technique.[14,15] The blending of nanopowders with ceramic is done using planetary ball milling. Collision energy was supplied for 0 to 20 hours to downgrade the particles and for manipulation. After 20 hours of milling, gradual refinement of the powder is done to obtain the required reinforced material. The test samples were made in accordance to ISO 6872 standardization and evaluated using three-point bending test in UTM for standard results.

The mean value of flexural strength of nano-reinforced feldspathic control group samples was 79.1 MPa, and for test group samples they were 79.5, 79.1, 92.0, and 96.9 and 92.9 MPa for 5, 10, 15, and 20% incorporation of nano-zirconia respectively. On comparison with control group, the reinforced feldspathic ceramic showed an increase in strength with reinforcement. The nano-zirconia-integrated feldspathic had improved strength of ceramic due to dispersion strengthening mechanism. The nanopowder has strong agglomeration tendency due to its high superficial energy, and they tend to increase the density if they were uniformly distributed.[16] The nano-zirconia reinforcement hindered the crack propagation through the material, increased the density of the sintered samples when pressed, and thus improved the strength of reinforced feldspathic samples.[17-20]

The incorporation of nano-zirconia in lithium disilicate does not improve the strength, rather it decreased the mean flexural strength of lithium disilicate. The SEM revealed that nanopowder had not fused to glass matrix of lithium disilicate structure, instead it acted as weak site with increased porosity and drastically reduced the strength of glass ceramics. The cause for the decrease in strength may be due to the method adopted in fabrication of samples and zirconia reinforcement.[21-23] The nano-zirconia can neither be absorbed nor could it act as a nucleating agent. It resulted in inhibition of crystallization of lithium disilicate crystals. Since there are no studies in literature to reinforce lithium disilicate, this necessitates observing other options for reinforcement.

The fractured samples were analyzed using scanning electron microscopy for distribution of particles and presence of porosities. The SEM of the feldspathic samples, both test and control group, had similar structure (Figs 2 and 3). In the lithium disilicate samples, the fracture surface had irregular regions dispersed in a large smooth area corresponding to the glassy matrix. These irregularities are related to the presence of clusters heterogeneously dispersed throughout the glassy matrix of this material, and they had a dendritic morphology. The test group revealed clusters of zirconia fine particles dispersed in some regions of glassy matrix crack deflection, which has been shown to be the main toughening mechanism in this ceramic. The SEM of lithium disilicate samples revealed uniform glass matrix in control group (Figs 4 and 5). Crack deflection around crystalline particles was also observed on the fracture surfaces of control group. Glass ceramic presented many twist hackles on the fractured surfaces which are indicative of successive changes in the plane of crack propagation, resulting in the formation of steps on the crack path, and the test group revealed increased number of surface porosities and irregular matrix.

The study used monoclinic nano-zirconia which was unstable at higher temperature, and during phase transformation if not stabilized can act as stress centers and may propagate the cracks through the material.[1] Future studies can use yttrium-stabilized nano-zirconia particles which act through phase transformation toughening mechanism. In addition, the methodology used can be ball-milling process to manipulate material instead of planetary milling.[3,24] The planetary ball mills are smaller than common ball mills and are mainly used in laboratories for grinding sample material down to very small sizes. In future, the X-ray diffraction analysis should be employed in nano-reinforcement of dental materials. It is a useful tool to identify the crystals and its distribution of nanoparticles in the samples.[25] Nanopowder has strong agglomeration tendency due to its high superficial energy, and they tend to increase the density if they were uniformly distributed and this agglomeration can be prevented by various other methods, such as layer-by-layer technique, spark plasma sintering method, and dynamic method of incorporating zirconia, which can be tried to improve the property of the material.[26,27]

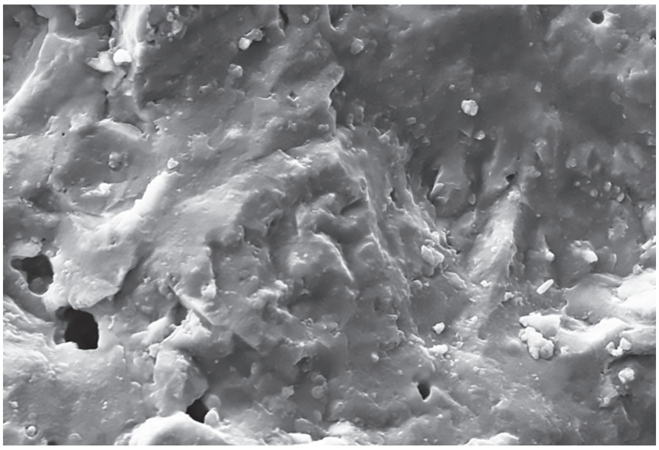


**Fig. 5:** Fracture surface of nano-zirconium dioxide integrated lithium disilicate ceramic

## CONCLUSION

Within the limitations of the study, the following conclusions were drawn:

- The mean flexural strength of feldspathic, lithium disilicate, and CAD/CAM-milled zirconia ceramics was 79.7, 222.7, and 800.2 MPa respectively.
- There was a gradual increase in the flexural strength of feldspathic ceramics reinforced with $ZrO_2$ nanopowder. The highest flexural strength of 96.9 MPa was observed in feldspathic ceramics reinforced with 20% of $ZrO_2$ nanopowder.
- The mean flexural strengths of lithium disilicate ceramics reinforced with 5, 10, 15, and 20% $ZrO_2$ nanopowder were 93.83, 97.1, 100.6, and 100.8 MPa respectively. All the test groups had lower values compared with the control group.
- The strengths of nano-zirconia-incorporated feldspathic and lithium disilicate samples were lower than CAD/CAM-milled zirconia bars, which had flexural strength of 800 MPa.

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