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Original Article

Evaluation of fracture toughness of zirconia silica nano-fibres reinforced feldespathic ceramic

Ahila Singaravel Chidambara Nathan*, Rajdeep Tah, Muthu Kumar Balasubramanium

SRM Dental College, Ramapuram, Chennai, 89, Tamil Nadu, India

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A B S T R A C T

Background: Dental ceramics exhibit great optical properties and better esthetics due to their translucency. Concern with this material is its brittleness, which accounts for its failure. Feldspathic ceramic is the most widely used veneering ceramic.
Aim and objectives: To evaluate and compare the fracture toughness of pressable feldspathic ceramics reinforced with zirconia silica nano-fibres with conventional pressable feldspathic ceramics.
Materials and method: According to ISO 6872, a master die was prepared from which, Bar shaped samples were formed in acrylic resin with the specified dimensions of 4.0 mm in width ×1.2 mm thickness ×25.0 mm. The zirconia silica nano-fibres were prepared by sol gel electro-spinning followed by calcination then they were incorporated into feldspathic ceramic through ball milling process. The samples were prepared with addition of 0, 2.5, 5, 7.5 weight % nano-fibres. The fracture toughnes was evaluated using the indentation strength method. The values were statistically analysed using the one sample Kolmogorov–Smirnoff test, Kruskal Wallis test and pair wise group comparison was done using Mann Whitney test with Bonferroni correction.
Results: The fracture toughness values for 2.5 wt% and 5 wt% groups were higher than control group, while the values for 7.5 wt% groups were lower compared to control group.
Conclusion: The fracture toughness values of feldspathic ceramic samples reinforced with zirconia silica nanofibers by 2.5 and 5 wt% were statistically significant when compared to samples reinforced with 7.5 wt%.

1. Introduction

Ceramic materials have been used over the years in dental applications for fabricating dental crowns, fixed partial dentures, laminate veneers, inlays, onlays and implants.

Dental ceramic is a brittle material, suffer from an inability to absorb appreciable quantities of plastic strain energy prior to fracture. This liability is manifested as flaw sensitivity, low tensile strength, and catastrophic failure.[1] Though with invent and advancement of material science and processing techniques, various newer ceramics are now able to withstand cracks and fracture. One measure of the strain energy absorbing ability of a brittle material is fracture toughness. The fracture toughness of a material is related to the level of tensile stress which must be attained in the vicinity of a crack before a catastrophic fracture process. Mechanical properties tensile strength, compressive strength and impact strength, thermal shock resistance, and susceptibility to erosive wear are all controlled by this parameter.[2]

Zirconia silica nanofibres have been used as reinforcement material for dental composites successfully. Incorporation of these fibres can significantly increase stiffness, flexure strength, fracture toughness and fatigue resistance of the composites.[3] Hence this study was done to evaluate and compare the fracture toughness of feldspathic ceramic reinforced with zirconia silica nano-fibres in the ratio of 2.5, 5, 7.5 wt% with conventional feldspathic ceramic.

2. Materials and method

According to ISO 6872, master die was prepared with a dimensions of 4.0 mm width ×1.2 mm thickness ×25.0 mm length and was duplicated in putty consistency addition silicone (Aquasil soft putty, Dentsply,Germany.) Autopolymerizing resin was used to make resin bars with ISO specified dimensions from the mold. A total of 40 samples were prepared and grouped as Group a (control), Group a1, Group a2 and Group a3 based on incorporation

* Corresponding author: Department of Prosthodontics, SRM Dental College, Ramapuram, Chennai, 89, Tamil Nadu, India.
E-mail address: ahilasc@yahoo.co.in (A. Singaravel Chidambara Nathan).

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of zirconia silica nano-fibres by 0, 2.5, 5 and 7.5 wt% respectively (Fig. 1).

2.1. Sample preparation

According to manufacturer instructions,feldspathic powder (IPS Classic V Dentin Body, Ivoclair, Vivadent) was mixed with distilled water, then condensed with the help of syringe piston on one side and absorbent paper on other side. It was carefully detached from the syringe tube and sintered at 900 °C to produce feldspathic ceramic pellets. The resin bars were sprued and invested in phosphate bonded investment (IPS Press Vest Speed, BEGO, Germany) followed by burnout. The preformed feldspathic pellets were pressed under pressure and high temperature into the mold space. The control samples were divested and recovered.

Zirconia silica nanofibres were prepared using sol gel electrospinning and subsequent calcinations, with help of electrospinning set up (ESPIN- NANO, PECO, Chennai,India). Zirconia silica nanofibres were added to conventional feldspathic ceramic in the ratio of 2.5, 5.0 and 7.5% by weight separately. The mixing of nanofibres to feldspathic powder was done through ball milling. The three groups of powder obtained were mixed with distilled water separately, and then condensed with help of syringe piston and absorbent paper. It was carefully detached from the syringe tube and sintered at 900 °C to produce three feldspathic ceramic pellets. The pellets were pressed under pressure and high temperature into the mold space and the test samples were divested after sintering.

2.2. Evaluation of fracture toughness

The samples were evaluated with the indentation strength method. Vickers indentation (CSM instruments, Switzerland) was placed on the center of each specimen at 9.8 N. Then the specimens were tested in three-point bending machine (Autograph universal testing machine, Shimadzu corp, Japan) at a crosshead speed of 0.1 mm/min and a test span of 25 mm.

Fracture stresses (sf) were calculated from the formula $sf = 3 WL/2bd^2$. Where W is the breaking load, L the test span, b the specimen's width, and d the specimen's thickness (height). The hardness (H) was calculated from the indentation load (P) divided by the projected area of the indenter on the surface, where l is the indenter half-diagonal length using $H = 0.5\ P/l^2$. Fracture toughness were calculated from the formula $K_{IC} = 0.59\ (E/H)^{1/8}\ (S_f P^{\ 1/3})^{3/4}$, relating the elastic modulus (E), indentation load (P), hardness (H) and fracture stress (sf). The dynamic elastic modulus (E) was previously determined.

3. Results

The software SPSS version 22.0 was used to analyze the data. The mean fracture toughness of each group was calculated, and the values were statistically analysed using Kolmogorov–Smirnoff test (Normality test) and the results showed that the variables did not follow normal distribution. Therefore to analyze the data, non-parametric tests were applied. Comparison between all the four groups were done using Kruskal Wallis test and Mann Whitney test with Bonferroni correction was used for pair wise group comparisons.

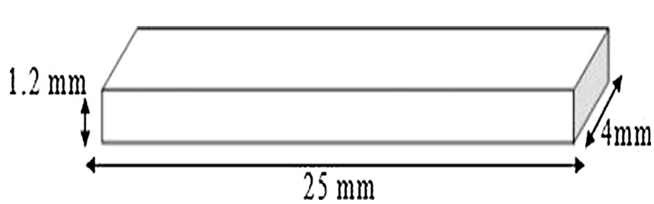

Fig. 1. Schematic diagram of master die.

The mean fracture toughness of group a, group a1, group a2 and group a3 were 0.936 ± 0.017, 0.968 ± 0.037, 0.972 ± 0.074, 0.858 ± 0.065 Mpa$\sqrt{m}$ $^{1/2}$ respectively (Table 1). The comparison between the fracture toughness values for group a, group a1, group a2 and group a3 was done using Kruskal Wallis test. The significant value P was 0.023 which is less than 0.05, hence it is statistically significant (Table 2). Pair wise comparison of group a, group a1, group a2 and group a3 was done using Mann Whitney test with Bonferroni correction. The comparison between group a1 against group a3 and group a2 against group a3 showed that the significant value P is less than 0.05, hence it is statistically significant, but it is statistically insignificant for other group (Table 3).

The comparison of the mean fracture toughness for group a1 (2.5 wt% nanofibers) and group a2 (5 wt% nanofibers) were higher than control group, while the values for group a3 (7.5 wt% nanofibers) were lower compared to control group. The mean fracture toughness value was highest for group a2 (5 wt% nanofibers) and lowest for group a3 (7.5 wt% nanofibers) among the test groups (Graph 1).

4. Discussion

Traditionally, ceramics were used in the fabrication of artificial teeth for dentures, crowns and FPD's. From 1980 the ceramics had wider application which includes veneers, inlays/onlays, crowns and short span fixed partial dentures. With the development of newer materials with remarkable mechanical properties ceramic posts, abutments and implants were made.

In the last 30 years, the growing patient's demand for highly esthetic and natural-appearing restorations had led to the

Table 1
Descriptive statistics for fracture toughness.

Fracture toughness	Group a	Group a1	Group a2	Group a3
N	5	5	5	5
Mean	0.936	0.968	0.972	0.858
Std. Dev	0.017	0.037	0.074	0.065
1st Quartile	0.930	0.950	0.930	0.830
Median	0.940	0.970	0.980	0.870
3rd Quartile	0.950	0.980	1.030	0.910

Table 2
Comparison of fracture toughness values by using Kruskal Wallis test.

Variable	Group	N	Mean Rank	P-Value
Fracture toughness	Group a	5	10.40	0.023
	Group a1	5	14.00	
	Group a2	5	13.70	
	Group a3	5	3.90	

Table 3
Pair wise comparison of fracture toughness using Mann Whitney test with Bonferroni correction.

Group	P-Value
Control Group a vs Group a1	0.990
Control Group a vs Group a2	0.995
Control Group a vs Group a3	0.488
Group a1 vs Group a2	0.998
Group a1 vs Group a3	0.041
Group a2 vs Group a3	0.048

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