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ORIGINAL ARTICLE

Evaluating The Fracture Toughness and Flexural Strength of Pressable Dental Ceramics: An In Vitro Study

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Abstract The study was undertaken to evaluate the biaxial flexural strength, biaxial flexural strength after etching with 9 % HF acid and fracture toughness of three commonly used pressable all ceramic core materials. Ninety glass ceramic specimens were fabricated from three commercially available leucite based core ceramic material (1) Esthetic Empress, (2) Cergo, and (3) Performance Plus. Thirty discs of each material were divided into three groups of 10 discs each. Biaxial flexural strength (30 discs,) Biaxial flexural strength for samples treated with 9 % HF acid (30 discs) and fracture toughness (30 discs) were evaluated. Core material Performance Plus had the lowest biaxial strength of 124.89 MPa, Cergo had strength of 152.22 MPa and the highest value of 163.95 was reported for Esthetic Empress. For samples treated 9 % HF, Performance Plus had the lowest biaxial strength of 98.37 MPa, Cergo had strength of 117.42 MPa and the highest value of 143.74 was reported for Esthetic Empress. Core material Performance Plus had the lowest fracture toughness of 1.063 MPa, Cergo had strength of 1.112 MPa and the highest value of 1.225 was reported for Esthetic Empress. The results shows that Esthetic Empress had better mechanical properties compared to Cergo had Performance Plus in relation to the parameters tested.

Keywords Metal ceramics · Biaxial flexural strength · Fracture toughness

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Introduction

Driven by a debatable need for metal-free restorations, the evolution of all-ceramic systems for dental restorations have been remarkable in last three decades. Processing techniques novel to dentistry have been developed, such as heat pressing, slip-casting and computer aided design computer aided machining (CAD-CAM). Concurrently, all-ceramic materials have been developed to match dental requirements, offering increasingly greater performance from a mechanical standpoint. As opposed to metal-ceramics, all-ceramics contain a significantly greater amount of crystalline phase, which is about 35–99 vol%. This higher level of crystallinity is responsible for an improvement in mechanical properties through various mechanisms, such as crystalline reinforcement or stress induced transformation.

The interest of dentists, dental technicians and patients in all-ceramic materials is rapidly increasing as stronger and tougher materials are developed and commercialized along with novel processing technologies. Currently, a wide range of materials and systems are available. However, relatively little is known about their microstructure and toughening mechanisms and their relationship to the mechanical properties of the corresponding ceramic. The present study was undertaken to evaluate the biaxial flexural strength [1], biaxial flexural strength after etching with

9 % HF acid [2] and fracture toughness [3–5] of three commonly used pressable all ceramic core materials.

Materials and Methodology

Ninety glass ceramic specimens were fabricated from three commercially available leucite based core ceramic material (1) Esthetic Empress, (2) Cergo and (3) Performance Plus by the lost wax and hot pressed ceramic fabrication technique following the manufacturer's instructions. Thirty ceramic disc specimens were fabricated for each group from wax patterns, approximately 15 mm in diameter and 1.2 mm (±) 0.2 mm thick [6]. For obtaining specific diameter of disc, a stainless steel die was prepared measuring 15 mm in diameter and 1.5 mm in thickness. Molten inlay wax was flown into the die and wax discs were formed according to the specific dimensions. The obtained ceramic samples were divided into equal numbers (N = 10) and were tested for above mentioned parameters.

Test

Thirty discs of each material were divided into three groups of 10 discs each. Following tests were done for the obtained samples of three different materials.

Parameter 1—biaxial flexural strength (30 discs)
Parameter 2—biaxial flexural strength treated with 9 % HF acid (30 discs)
Parameter 3—fracture toughness (30 discs)

Parameter 1: Biaxial Flexural Strength

The biaxial flexural strength of 10 disc specimens per group was determined using the piston on three ball test or three point bending test (ASTM standard F394-789). Disc specimens were centered and supported on three steel spheres of 4 mm diameter positioned 120° apart on 18 mm diameter circle. The load was applied to the centre of the specimen by a special custom made jig having circular cylinder of hardened steel. The jig was having a diameter of 1.4 mm with flat end perpendicular to the axis which was attached to the upper member of the universal testing machine (Fig. 1). A thin plastic sheet was placed between the specimen surface and the flat ended loading cylinder to distribute the load uniformly. The specimens were loaded in a universal testing machine at a cross head speed of 0.5 mm/min till the specimen fractured.

Testing was performed at room temperature conditions. The maximum tensile stress (MPa), which corresponded to the biaxial flexural strength, was calculated. The equation

Fig. 1 Metal die and jig

suggested by the test standard (ASTM standard F394-789) was as follows [1].

$$\sigma_{ba} = AF/t^2,$$

$$A = 3/4\pi\left[2(1+V)\ln a/r_0 + (1-v)\left(2a^2 - r_o^2\right)/(1+v/2b)\right],$$

$$r_o = \sqrt{1.6r^2} + t^2 - 0.675t,$$

where σ_{ba} modulus of rupture (MPa), F force at fracture (N), t disc thickness, v Poisson ratio, a radius of supporting balls, b disc diameter, r radius of loading chisel, r_0 equivalent radius.

Parameter 2: Biaxial Flexural Strength Treated with 9 % HF Acid [2, 7]

Ten specimens from each ceramic group were etched with 9 % HF acid gel for 2 min, washed in running water and then cleaned ultrasonically for 15 min in distilled water. Biaxial flexural strength of specimens was tested as was done in parameter 1.

Parameter 3: Fracture Toughness [3–5]

Ten specimens from each group were mounted on Vickers hardness tester with indentation loads of 40, 60, and 100 N. In the present study, each sample was placed on Vickers indenter and a gradual force was applied. Force was applied from 10 N onwards and the point where indentation occurred was taken as the base load for the respective material. Indentations were formed at a base load of 100 N for Esthetic Empress, 60 N for Cergo and 40 N for Performance Plus. Three acceptable indentations for each load were chosen. The optical microscope on an ultra micro indentation system was used to perform the measurements of the radial cracks within few hours after indentation.

The fracture toughness was calculated using the formula given by Anstis et al. [6]

$$K_{1C} = 0.016(E/H)^{1/2}\left(P/C^{3/2}\right),$$

where 0.016 is material independent constant, E is the elastic modulus, H the hardness, P the indentation load (N) and C the crack length measured from the middle of the Vickers.

Results

Biaxial Flexural Strength

The values for the three all ceramic core materials were presented in Tables 1, 2 and 3. Core material Performance Plus had the lowest strength of 124.89 MPa, Cergo had strength of 152.22 MPa and the highest biaxial strength value of 163.95 was reported for Esthetic Empress (Fig. 2).

Biaxial Flexural Strength Using 9 % HF Acid (Tables 1, 2 and 3)

Core material Performance Plus had the lowest strength of 98.37 MPa, Cergo had strength of 117.42 MPa and the highest biaxial strength value of 143.74 was reported for Esthetic Empress (Fig. 3).

Table 1 Biaxial flexural strength values of non-etched and etched samples

Biaxial flexural strength		Biaxial flexural strength using 9 % HF acid	
Load in Newton	MPa	Load in Newton	MPa
82.3	158.48	81.5	156.93
85.7	165.03	78.7	151.55
89.5	172.34	75	144.42
81.75	157.41	65.5	126.13
79.6	153.28	66	127.09
87.5	168.49	73.75	142.01
83	159.83	80.25	154.53
87.2	167.92	75.10	144.61
91.35	175.91	78.5	151.16
83.5	160.79	72.15	138.93

Table 2 Biaxial flexural strength values of non-etched and etched samples

Biaxial flexural strength		Biaxial flexural strength using 9 % HF acid	
Load in Newton	MPa	Load in Newton	MPa
86.25	163.69	61.5	116.72
77.76	149.73	66	127.08
75.35	145.10	55.70	105.71
77.5	149.23	67.25	127.63
88.5	167.96	59.34	112.62
73.15	140.86	53.75	102.01
81.5	156.93	57.70	109.50
85.25	164.15	67.50	129.97
72.5	139.60	62.3	118.24
75.25	144.90	65.48	124.74

Table 3 Biaxial flexural strength values of non-etched and etched samples

Biaxial flexural strength		Biaxial flexural strength using 9 % HF acid	
Load in Newton	MPa	Load in Newton	MPa
55.23	104.82	56.75	107.70
70	134.79	47.5	90.15
67.30	127.72	57.5	110.72
71.10	136.91	54	102.48
57.5	110.72	52.5	99.64
63	121.31	48	91.1
77.5	147.08	42.5	80.66
67.25	129.49	50.5	95.84
60	115.5	53.25	101.06
63.5	120.51	55	104.38

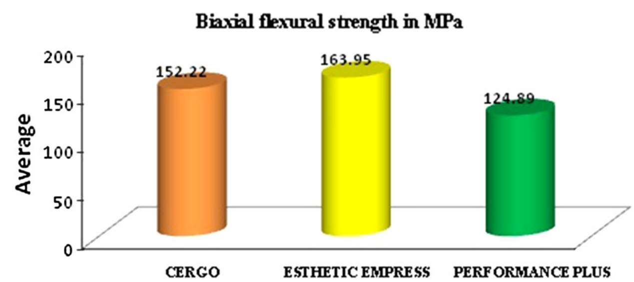

Fig. 2 Mean values for biaxial flexural strength

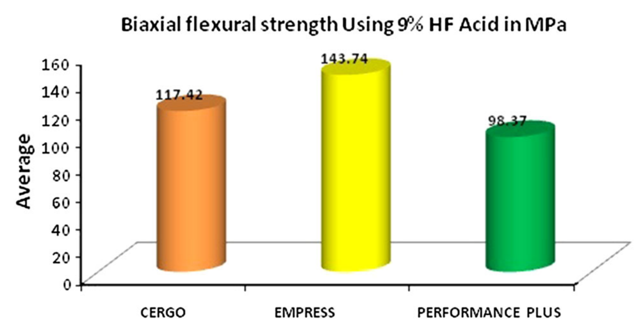

Fig. 3 Mean biaxial flexural strength using 9 % HF acid

Fracture Toughness (Table 4)

Core material Performance plus had the lowest fracture toughness of 1.063 MPa, Cergo had strength of 1.112 MPa and the highest value of 1.225 was reported for Esthetic Empress (Fig. 4).

Discussion

Of all materials used in dentistry to restore the natural dentition, ceramics have the best optical properties to

Table 4 Fracture toughness values of three all ceramic core materials

Fracture toughness values (MPa $m^{1/2}$)		
Esthetic Empress	Cergo	Performance Plus
1.21	1.15	1.07
1.17	1.11	1.07
3	1.06	1.06
1.28	1.09	1.08
1.22	1.14	1.04
1.24	1.11	1.12
1.24	1.15	1.06
1.29	1.13	1.05
1.17	1.11	1.02
1.18	1.07	1.06

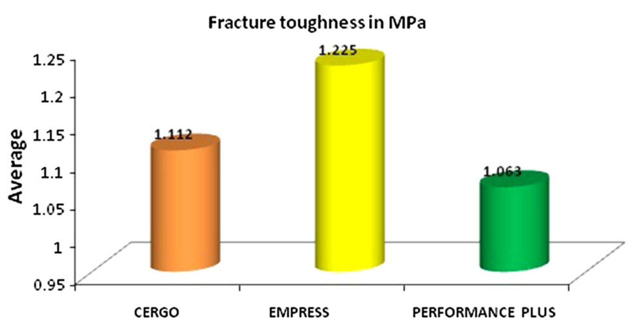

Fig. 4 Mean values of fracture toughness

mimic the tooth structure in appearance. Highly desirable esthetic qualities of dental ceramics are because of their optical properties like translucency [6].Though esthetically superior, the main drawback of ceramics is their brittleness and lack of fracture resistance.

The optimum strength of any ceramic is dependent on the fabrication procedure and minimization of flaws. Furthermore, several factors can also influence the definitive strength of ceramic materials, including dimension of specimens, test environment, polishing procedures, rate of stressing area of specimen subjected to the stresses, and testing methods [8]. Various tests used to test strength of brittle materials were reported in literature. The measurement of the strength of brittle materials under biaxial flexure conditions rather than uniaxial flexure (3- or 4-point flexural tests) is often considered more reliable, because the maximum tensile stresses occur within the central loading area and edge failures have no effect on specimen fracture [9]. Besides, the biaxial test is simpler to perform and provides a better simulation of clinically relevant sample size than that used for other strength tests [9]. Previous biaxial flexural studies [10–12] that reported lower strength values of IPS Empress 1 and Empress 2 (133–136 MPa) used piston tip diameter of 1.6 mm whereas this study adopted a smaller piston tip diameter of 1.4 mm. In the present study, a special custom made jig was prepared measuring 18 mm in diameter with three mounted steel spheres attached to the lower member. Disc specimens were centered and supported on three steel spheres of 4 mm diameter positioned 120° apart on 18 mm diameter circle. The mean values obtained by one-way ANOVA test were 124.89 MPa for Performance Plus, 152.22 MPa for Cergo and 163.95 for Esthetic Empress.

Resin composites are usually used to bond ceramic restorations to the tooth structure and also to repair fractured ceramics in repair systems. The establishment of the bond between ceramic and resin composite is usually created via micro-mechanical attachment by hydrofluoric (HF) acid etching and/or grit blasting followed by chemical bonding with a silane coupling agent. However, a major concern exists about the use of HF acid etching due to its hazardous effects on health and possible deleterious effects on ceramic strength. A study by Hooshmand et al. [1] concluded that a durable resin–ceramic bond could be obtained by using an appropriate silane application without the need for HF acid etching the ceramic surface, confirming the earlier observation. Mechanical strength is an important property that determines the performance of brittle materials. Ever since HF acid etching was first suggested as a ceramic surface pretreatment for resin bonding, many different etching periods have been advocated and used. The manufacturer's recommended etching time for cementation of the IPS Empress ceramic restorations with a luting resin is 60 s and for IPS Empress 2 is 20 s; however, the most profound ceramic surface roughness and the highest bond strength data at the ceramic–resin interface have been obtained by 2-min HF acid etching. Manufacturers most commonly recommend an etching time of 1–2 min for 9–10 % HF acid in ceramic repair systems. Other studies on the bond strength analysis of resin composite to ceramic have also applied a 2-min HF acid etching for the IPS Empress and IPS Empress 2 ceramic surface treatments [13, 14]. Thus, the effect of HF acid with an etching time of 2 min on the biaxial flexural strength of hot-pressed glass ceramics Esthetic Empress, Cergo and Performance Plus was assessed in the present study.

The biaxial flexural strength mean values with Cergo (117.42 MPa), Esthetic Empress (143.74 MPa) and Performance Plus (98.37 MPa) were obtained from the acid-etched specimens with 9 % HF acid. Both ceramic systems showed statistically significant differences with that of nonetched groups. In other words, the etching process reduced the biaxial flexural strength significantly for the three types of glass ceramics, but no significant interaction was reported between the ceramic type and etching. In the present study, the weakening effect of acid etching for three commercially available leucite based hot-pressed glass ceramics supported similar findings from studies on the other types of dental ceramic systems, such as aluminous or feldspathic ceramics [2]. The weakening effect of HF acid on leucite-based glass ceramic (IPS Empress) has also been confirmed by the fracture surfaces and bond strength data obtained from other studies [13, 15]. Preferential attack of HF acid on the grain boundaries at the interface of leucite crystals and the glass phase and its weakening effect have been reported for the leucite based glass ceramics [13, 15].

One important feature of fracture toughness is its ability to indicate a material's serviceability in the oral cavity. The application of the indentation fracture technique (IF) in studying the behavior and properties of brittle materials is specifically appropriate because only small dimensional specimens are required and the crack growth parameter is similar to those cracks expected in clinical conditions [16].

Esthetic Empress, Cergo and Performance Plus, were included in this study mainly as a benchmark for the more recent peer materials. The microscopic observations reported in this study support previous statements regarding the toughening mechanism. This mechanism is based on a uniform distribution of the leucite crystals and the microcrack toughening due to the mismatch of the coefficient of thermal expansion between the crystalline and glassy phases [17]. The role of the micro cracks in glass–ceramics is contradictory. It has been proposed that the microcracks can contribute to deflecting a crack and dispersing its energy, increasing the strength and fracture toughness of a given ceramic. However, if clusters of crystals are present, microcracks tend to coalesce, forming a crack, which surrounds the cluster (decoupling of leucite particles) as if it was a single grain, reducing the strength and fracture toughness [11]. In Esthetic Empress, Cergo and Performance Plus, pressing contributes to generating an even distribution of the grains, which plays an important role in avoiding such a phenomenon. Even then, the proposed mechanism is scarcely effective and Esthetic Empress is not significantly stronger than some frit feldspathic ceramics. Hence the considerable clinical success of Esthetic Empress, Cergo and Performance Plus cannot be explained on the basis of its mechanical properties. Other factors, such as layering, glazing, staining and adhesive cementation, have been proposed to explain the apparent greater strength and reliability of the in-service restoration.

Conclusions

Within the limitations of the present in vitro study, the following conclusions could be drawn:

1. Considering biaxial flexural strength values, Esthetic Empress had highest strength value, Cergo had relatively moderate strength and Performance Plus had lowest strength value.
2. It was concluded that biaxial flexural strength with 9 % HF acid etching had a weakening effect on the hot pressed leucite based all ceramic materials.
3. The indentation fracture toughness test indicates that Esthetic Empress had highest fracture toughness, Cergo had relatively moderate fracture toughness and Performance Plus had lowest fracture toughness.

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