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Research / Araştırma Makalesi

Comparison of Mechanical Properties of Aged All-Ceramic Materials

Yaşlandırılmış Tam Seramik Materyallerin Mekanik Özelliklerinin Karşılaştırılması

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ABSTRACT

Background: Our research aimed to compare the biaxial flexural strength of glass ceramic and two different monolithic zirconias after aging, examine the fracture surfaces with scanning electron microscopy (SEM) and perform elemental analysis with energy dispersive spectroscopy (EDS).

Methods: Two types of monolithic CAD/CAM materials (Katana UTML and Prettau Anterior) and a lithium disilicate material (IPS emax Press) were selected for this study. All samples were aged with a thermal cycle of 10000 cycles. The biaxial flexural strength of the disc specimens was determined using the three-ball piston test and a universal testing machine. Biaxial flexural strengths were compared for each group (n = 10). All fractured samples were examined by SEM and elemental analyzes were performed with EDS. Data were analyzed by 1-way ANOVA and Tukey post hoc (p<0.05).

Results: Prettau Anterior showed a statistically significant difference between the other groups (p<0.05). Following Prettau Anterior (1141.81 MPa), IPS Emax Press (278.86 MPa) and Katana UTML (216.14 MPa) showed biaxial flexural strength, respectively.

Conclusion: Compared to the other materials evaluated, Prettau Anterior had much higher biaxial flexural strength. Katana UTML and IPS emax Press show similar strength over the long term. It is seen that the chemical structure of the materials has an effect on the mechanical capacity.

KEYWORDS: Biaxial Flexural Strength, Lithium Disilicate Glass-Ceramic, Monolithic Zirconia

ÖZ

Amaç: Araştırmamız, cam seramik ve iki farklı monolitik zirkonyanın yaşlanma sonrası çift eksenli eğilme dayanımını karşılaştırmayı, taramalı elektron mikroskobu (SEM) ile kırık yüzeylerini incelemeyi ve enerji dağılımlı spektroskopi (EDS) ile element analizi yapmayı amaçladı.

Gereç ve Yöntemler: Bu çalışma için iki tip monolitik CAD/CAM materyali (Katana UTML ve Prettau Anterior) ve bir lityum disilikat materyali (IPS emax Press) seçilmiştir. Tüm örnekler, 10000 döngülük termal siklus ile yaşlandırıldı. Disk numunelerinin biaksiyal eğilme dayanımı, üç bilyeli piston testi ve evrensel bir test makinesi kullanılarak belirlendi. Her grup için (n = 10), çift eksenli eğilme dayanımları karşılaştırıldı. Tüm kırık numuneler SEM ile incelendi ve EDS ile element analizleri yapıldı. Veriler 1-way ANOVA ve Tukey post hoc ile analiz edildi (p<0.05).

Bulgular: Prettau Anterior diğer gruplar arasında istatistiksel olarak anlamlı fark gösterdi (p<0.05). Prettau Anterioru (1141.81 MPa) takiben sırasıyla IPS Emax Press (278.86 MPa) ve Katana UTML (216.14 MPa) çift eksenli eğilme dayanımı gösterdi.

Sonuç: Değerlendirilen diğer materyallerle karşılaştırıldığında Prettau Anterior çok daha yüksek çift eksenli eğilme dayanımına sahipti. Katana UTML ve IPS emax Press, uzun dönemde benzer dayanım göstermektedir. Materyallerin kimyasal yapısının, mekanik kapasitesine etkisi olduğu görülmektedir.

ANAHTAR KELİMELER: Çift Eksenli Eğilme Dayanımı, Lityum Disilikat Cam-Seramik, Monolitik Zirkonya

Introduction

In prosthetic dentistry, the use of all ceramics has been increasing in order to meet the patients'aesthetic expectations. Brittleness is one of the most important disadvantages of full ceramics, and this feature is responsible for the strength behavior of all ceramics.[1] All ceramics fail at the first sign of overload due to their limited plastic deformation capacity.[2,3] Dental restorations are intermittently exposed to occlusal forces during chewing and swallowing. Porcelain materials are brittle, have limited tensile strength, and may lose their resistance to stresses over time.[4] Durability is a key mechanical property that influences the clinical application areas and limitations of porcelain materials. Since most of the chewing forces are in the form of compression, compressive strength is especially important in the chewing process. Therefore, while the demand for all-ceramic systems is increasing rapidly, attention has been focused on long-term studies in which fatigue of systems is investigated, especially by simulating the clinical environment.[5] With durability tests, samples can be loaded periodically and stresses that materials are exposed to as a result of chewing function in the mouth can be imitated. Flexural strength is an important data for materials to resist harsh conditions in the oral environment.

Y-TZP ceramic (tetragonal zirconia polycrystals stabilized with yttria) is one of the strongest material among all-ceramic dental restorations, providing esthetics, durability and function.[6] IPS e.max Press (Ivoclar Vivadent) is a heat-pressed all-ceramic with lithium disilicate reinforcement that is preferred for its durability, aesthetics, and biocompatibility. Because of their high performance and mechanical properties, alumina and zirconia-based ceramics are viable options for all-ceramic restorations in high-stress areas.[7] The optimum mechanical performance of Y-TZP depends on phase transformation hardening, which increases the fracture strength.[8] The crystal phase transformation of the near-defect metastable Y-TZP grains from the tetragonal-monoclinic crystal system is initiated by the surface tension surrounding the microcracks.[9] The volumetric expansion of the involved grains results from the phase change that places compressive stress on the developing fracture and stops or slows crack development. However, depending on body temperature and humidity, low temperature degradation (LTD) and tetragonal-monoclinic phase change may occur naturally over time.[6] LTD is a time-dependent and thermodynamic phenomenon. After water diffuses into the zirconia lattice, it can be activated by water molecules[8,12] over a large temperature range (37-500°C) by breaching an energy barrier. [8,12,13] Through a mechanism known as toughening, aging first causes an increase in the material's mechanical qualities, and later it causes mechanical deterioration.[16-18]

Dental restorations are exposed to various harmful stimuli such as temperature changes in the oral environment, chewing and the effect of water. There is currently little information available examining the effect of aging on the mechanical properties of Y-TZP monolithic zirconia.[6]

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The biaxial flexural strength assessment of brittle materials are considered more accurate than the uniaxial one as false edge fractures are minimized and maximum tensile stresses occur within the center loading area. This results in unaffected by the edge condition of the sample and makes it possible to evaluate slightly distorted samples. The biaxial bending test provides less variable data when determining strength.[19]

Observing surface microstructures or composition or performing surface analysis requires the use of EDS or FE-SEM (field-emission scanning electron microscopy).[20,21] In this in vitro study, the mechanical properties of various ceramic materials after thermal cycling aging were investigated. The null hypothesis is that there will be no difference in biaxial flexural strength between all hydrothermally aged ceramic groups.

MATERIAL AND METHODS

In this study, two monolithic CAD/CAM materials, Katana UTML and Prettau Anterior, as well as a lithium disilicate ceramic IPS emax Press, were used. **Table 1** shows information about the specimens, manufacturers, and material composition. For each group, 10 disc-shaped specimens with a diameter of 13 mm and a thickness of 1 mm were prepared (n=10) with the Wieland Dental Zenotec Select Hybrid CAD-CAM device. All specimens were polished with a Renfert (GMBH, Germany) polishing disc for 5 minutes.

Table 1. The study's ceramic systems

Type	Material	Manufacturer	Composition
Yttrium-stabilized zirconia	Katana UTML	Noritake Dental Co	ZrO_2, Y_2O_3, HfO_2, other oxides
Yttrium-stabilized zirconia	Prettau Anterior	Zirkonzahn GmbH	Y_2O_3, Al_2O_3, Na_2O, SiO_2, Fe_2O_3
Lithium disilicate reinforced glass ceramic	IPS emax Press	Ivoclar Vivadent	SiO_2, Li_2O, K_2O, P_2O_5, ZnO, ZrO_2, other oxides and ceramics pigments

Aging process

The samples underwent 10000 cycles of thermocycling in a distilled water bath at 5-55°C. In the thermal cycle test, it is reported that 10000 cycles are equivalent to approximately 1 year of function *in vivo.* [22]

Test of biaxial flexural strength

The biaxial flexural strength of all-ceramic disc specimens was determined using the three-ball piston test and a universal testing machine according to ISO 6872[23] (Devotrans, Turkey) (**Figure 1**). The samples were placed at an angle of 120o to each other on three steel balls with a diameter of 3.4 mm located at the device's base. On the upper side of the device, a pressure tip with a diameter of 1.4 mm was placed, which was arranged to contact the sample from the center. A force of 1 mm per minute was applied until the pressure tip broke the sample. The force values at the moment the sample was broken were noted in Newton (N). Since the recorded values are N units, the biaxial bending test result was converted to megapascal (MPa) units using the formula below.

$S = -0.2387 \times P \times ((X-Y) / (d2))$,

P: fracture load (N);

S: biaxial flexural strength (MPa);

d: thickness of the specimen disc (mm).

Following are the calculations for X and Y:

$X = (1 + \upsilon) \ln (r2/r3)^2 + [(1 - \upsilon)/2] (r2/r3)^2$,

$Y = (1 + \upsilon) [1 + \ln (r1/r3)^2] + (1 - \upsilon) (r1/r3)^2$,

r1: the radius of support circle;

r2: the radius of loaded area;

r3: the radius of the specimen and

υ: the Poisson’s ratio (0.25).

Figure 1. Machine for biaxial flexural testing with a disc-shaped specimen.

Scanning electron microscopy (SEM) evaluation

After biaxial flexural strength testing, all fractured specimens were coated with Au and analyzed using SEM (EVO LS 10; Zeiss, Germany). SEM images were examined at 10000 x magnification, 15 kV of acceleration voltage, distance to sample 11mm. Chemical composition of the samples was determined using SEM equipped with an EDS (EVO LS 10; Zeiss, Germany) at 10000 x magnification, 15 kV of acceleration voltage.

Statistical analysis

Data were analyzed by one-way ANOVA (IBM SPSS 20.0 software; SPSS Inc., Chicago, IL) and to detect differences among all the groups that Tukey honest post hoc test. Statistical significance level was $p < 0.05$.

RESULTS

Table 2 displays the mean and standard deviation of the mechanical properties tested for each ceramic. Prettau Anterior showed a statistically significant difference between the other groups ($p<0.05$). Following Prettau Anterior, IPS Emax Press (278.86 MPa) and Katana UTML (216.14 MPa) showed biaxial flexural strength, respectively.

Table 2. Results of biaxial flexure tests

Material	Mean ±SD Biaxial Flexural Strength (MPa)
Prettau Anterior	1141.81±59.09 a
Katana UTML	216.14±11.67 b
IPS emax Press	278.86±24.57 b

**Different superscript letters indicate statistically significant group differences (P.05).*

Figure 2 shows images from the scanning electron microscope. Samples differ in grain size and shape. **Table 3** shows the average values of the chemical elements of each group obtained as a result of EDS. The result of the EDS analysis shows that the chemical components of each material are as declared by the manufacturers, except for the absence of Li in the IPS emax Press. Although the IPS emax Press manufacturer states that it contains lithium, this element was not found in the EDS result.

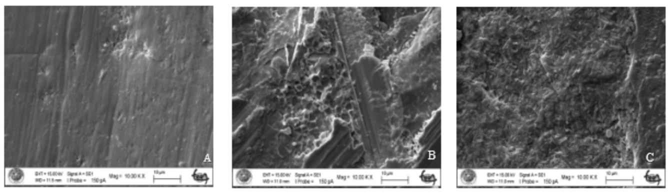

Figure 2. SEM images of fractured specimens (original magnification ×10 000). A, Prettau Anterior; B, Katana UTML, C, IPS emax Press.

Table 3. EDS analysis results (weight%)

Element	Material		
	Prettau Anterior	Katana UTML	IPS Emax Press
Zirconium	73.69	75.76	20.14
Oxygen	10.46	10.28	42.18
Yttrium	3.66	6.82	
Aluminium	0.12	0.03	
Hafnium	4.50	6.65	
Silicon	0.15		25.87
Iron	0.66		
Sodium	0.50		
Potassium			2.88
Zinc			8.90
Phosphorus			0.02

DISCUSSION

This study compared and evaluated the mechanical properties of three different all-ceramic material after aging with biaxial flexural tests. Additionally, microstructural composition and micromorphology of the materials in relation to their mechanical properties were investigated. Significant differences in biaxial flexural strength were observed between the materials and thus the null hypothesis was rejected. Prettau Anterior had the highest mean value in biaxial flexural strength, followed by IPS emax Press and Katana UTML.

Conventional zirconia is categorized as 3 mol% yttria stabilized tetragonal zirconia polycrystal (3Y-TZP), whereas highly translucent zirconia generally contains a significantly higher amount of yttria along with a higher amount of c-ZrO_2 phase. The latter is often referred to as Y-PSZ. Furthermore, due to reduced transformation toughening, Y-PSZ in general possesses lower fracture toughness, strength and crack resistance than conventional 3Y-TZP. Despite these less favorable material properties, their increased translucency makes them attractive for full-contour restorations, promoting the development of Y-PSZs for monolithic restorations.[24] In this study, yttrium-stabilized zirconia and lithium disilicate glass-ceramic as control group were examined.

Many *in vitro* studies have reported that aging processes greatly reduce the fracture strength of zirconia.[25-28] It is not known how the material will be affected in the long term as a result of direct fluid contact in monolithic zirconia restorations. In the in vitro study of Nakamura et al. in 2015, it was reported that the fracture strength was sufficient for the posterior regions, although there was an increase in monoclinic phase transformation as a result of low heat degradation in monolithic zirconia restorations.[29]

Chai et al.[30] examined the bending strength of three different zirconia and glass ceramics in their study. Uniaxial flexural strength and biaxial flexural strength tests were applied. According to the data obtained, the results of uniaxial bending tests showed statistically low fracture strength compared to the results of biaxial bending tests. Bending tests performed on two axes by applying a load to the center points of disc-shaped specimens eliminate the negative effects of bending tests performed on one axis. In the bending tests carried out in two axes recommended by the ISO 6872 standard, the faults on the surfaces of the samples can be tolerated and since the force is applied from the center, they reported that the durability values are not affected by the cracks on the disc edges.[30] Therefore, we preferred the biaxial flexural strength test in our study.

Flinn et al., in a study of yttria-stabilized zirconia polycrystalline samples, examined the effect of different accelerated aging procedures on durability and found that hydrothermal aging triggered the conversion from tetragonal to monoclinic and caused a significant decrease in durability.[16] Dikicier et al. found that UV aging had no significant effect on the biaxial flexural strength of all selected ceramics.[31] While commercial 5Y-PSZ showed no significant influence of aging on strength, hydrothermal reactor aging significantly decreased the in house 5Y-PSZ characteristic strength.[32] In this study, hydrothermal aging was performed by thermal cycle.

The differences between the materials were revealed in the SEM images of the fractured slot surfaces. The appearance of the fracture pattern and fracture surfaces varies depending on the variations in the microstructure.[33] Coldea et al suggested that “propagating cracks are deflected and experience a more tortuous path resulting in rough surfaces.” In this study, it shows that Prettau anterior fracture surfaces were smoother compared to Katana UTML and IPS e.max press.[34]

There is no statistically significant difference between IPS emax Press and Katana UTML for biaxial flexural strength results. Similar results were found for Katana UTML and IPS emax in a study by Reyes et al. in which they compared the biaxial flexural strength of recently developed high translucent zirconia, high strength zirconia and lithium disilicate ceramics.[35]

The relatively larger particles in the cubic structure in the Katana UT structure contain more Y_2O_3 to provide phase stabilization.[36] This explains the fact that the biaxial flexural strengths of the katana samples are significantly lower than the other groups. It has been shown in previous studies that alumina has a positive contribution to mechanical properties.[37] It is known that the increase in the amount of Y_2O_3 has a direct negative effect on the mechanical properties of cubic zirconia. As seen in previous studies, lower Y_2O_3 content positively affects material durability.[38,39] Our study supports these. Based on EDS results, Katana UTML contains 6%, Prettau anterior 3% yttrium. According to the biaxial flexural strength test results, Prettau anterior was found to be more strength than Katana UTML.

Pereira et al[40] tested Katana UTML discs for biaxial flexural strength without aging and reported a mean value of 470 MPa. In our study, the biaxial flexural strength value of the katana was 216 MPa. Considering the effect of aging in our study, the fact that it was lower was consistent with the current study.

This in vitro study failed to include environmental factors such as watery conditions and chewing force in the oral cavity. This is one of the limitations of the present study. Existing findings should be supported by further in vitro and in vivo research to clarify changes in these and other properties.[41]

CONCLUSION

Within the constraints of current in vitro research, the following conclusions can be drawn.

Compared to the other materials evaluated, Prettau Anterior had much higher biaxial flexural strength. Katana UTML and IPS emax Press show similar strength over the long term. It is concluded that the chemical structure of the materials has an effect on the mechanical durability.

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Etik Beyan / Ethical statement

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It is declared that during the preparation process of this study, scientific and ethical principles were followed and all the studies benefited are stated in the bibliography.

Benzerlik Taraması / Similarity scan

Yapıldı – ithenticate

Etik Bildirim / Ethical statement

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Çıkar Çatışması / Conflict of Interest

Yazarlar çıkar çatışması bildirmemiştir. | The authors have no conflict of interest to declare.

Yazar Katkıları / Author Contributions

Çalışmanın Tasarlanması | Design of Study: TT (%70), ART (%30)
Veri Toplanması | Data Acquisition: TT (%70), ART (%30)
Veri Analizi | Data Analysis: TT (%70), ART (%30)
Makalenin Yazımı | Writing up: TT (%80), ART (%20)
Makale Gönderimi ve Revizyonu | Submission and Revision: TT (%100)

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