The Effect of Hydrothermal Aging on Biaxial Flexural Strength of Contemporary CAD/CAM Dental Ceramics

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Abstract

Various Computer-Aided Design/Computer-Aided Manufacturing (CAD-CAM) restorative materials are currently available in the dental market. It is claimed that the new materials meet patients' esthetic expectations and mechanical requirements. However, most of these materials are still inadequately tested by research.

The purpose of this research was to study the effect of low-grade hydrothermal aging on flexural strengths of three commercially available dental ceramic materials; IPS e.max Cad (E- group), Vita Suprinity (S-group) and Ceramill zolid ht+ (Z-group) under ISO standards.

Using computer-aided machining, disc-shaped specimens (n=180) were designed and sliced from prefabricated blocks of the tested materials. Specimens in each group were subdivided into two halves; the first half (n=30) were loaded until fracture without aging and the other half were subjected to a low-grade hydothermal aging, then were loaded until failure. A universal testing machine was used to determine the flexural strength for the various ceramic discs. Before-and after-aging data were compared and analyzed for the various groups. One-Way_ANOVA and Post-Hoc (Bonferroni) tests were used for multiple comparisons across the groups at a significance level of p-value < 0.05.

The mean ±SD flexural strength values were 228.18 ±73.83, 242.74 ±52.85 and 589.84±96.48 for E group, S group and Z group respectively. A statistically significant reduction in the flexural strength was seen in the S and Z groups whereas E group' strength was not significantly influenced by the aging process.

Monolithic zirconia had the highest flexural strength, however, this strength was significantly reduced by exposure to low-grade hydrothermal aging. Hence, the addition of Zirconia particles to lithium crystals improved their flexural strength but lowered their resistance to hydrothermal aging.

Experimental article (J Int Dent Med Res 2023; 16(2): 580-587)

Keywords: Flexural strength, CAD-CAM, ceramics, hydrothermal aging.

Received date: 21 February 2023 **Accept date:** 02 April 2023

Introduction

The ever-increasing esthetic demands by dental patients and the rising request for metal-free restorations, as well as the clinicians' interest in esthetic dentistry and tooth-colored prostheses, have led to a variety of new all-ceramic materials with optimized esthetic properties. These materials have been introduced to the dental market and become available to clinicians and patients[1]. All-ceramic systems have become more preferred because of their esthetics, chemical stability, and biocompatibility[2]. However, they require substantial preparation of the tooth structure to provide sufficient durability. In order to overcome this issue and ensure conservation of tooth structure, innovative restorative materials have been developed to meet the aesthetic expectations of patients as well as the mechanical requirements.

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Furthermore, the development of Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) technology has opened new possibilities for restorative dentistry[3,4] and enabled continuous innovations and improvements in machinable materials to meet the increased demands for highly biocompatible, aesthetic, and long-lasting restorations[5].

CAD/CAM materials can be classified as ceramics (conventional feldspathic and high-strength) and hybrid materials. High-strength ceramics include glass ceramic strengthened by lithium disilicate and zirconia reinforced by lithium silicate. Hybrid materials that combine ceramic and resin have been also developed[6]. CAD/CAM glass ceramics are divided into mainly glass ceramics, leucite-reinforced glass-ceramics and lithium-disilicate glass-ceramics. Glass ceramics vary in their microstructure, i.e. in the size and distribution of their crystals; which influences their mechanical behavior[7] . Lithium disilicate ceramic has been considered the strongest glass-ceramic as it has a large number of interlocking, needle-like lithium disilicate crystals embedded into the glassy matrix which gives this type of ceramic higher mechanical properties compared to other types[8]. The most widely used ceramic in this group is the IPS e.max® CAD (Ivoclar Vivadent) that was introduced in 2005 became very well-known for their excellent optical properties[9].

Recently, a new class of improved glass-ceramics; a Zirconia-reinforced lithium silicate ceramic (Vita Suprinity® PC, VITA-Zahnfabrik H. Rauter-GmbH-Germany), has been introduced[10]. This type of glass- ceramic is fortified by 10% nanoparticles of zirconium dioxide. This combination strives to combine the esthetic properties of glass-ceramic and the mechanical strength of Zirconia[11].

The CAD/CAM materials have gained popularity due to their superior esthetics properties, more homogenous structures, higher-precision prostheses with less contraction on polymerization, more time efficiency and cost-effectiveness[12]. On the other hand, a new high-translucent yttria-stabilized tetragonal monolithic zirconia block (e.g. Ceramill Zolid White HT) has been developed in recent years to fulfill the esthetic requirements of patients[13]. The advancement in digital technologies and manufacturing techniques has led to a wide range of materials making the decision more challenging to clinicians and requiring full knowledge of the chemical and physical properties and the clinical application of each individual material.

It is worth noting that the newly proposed compositions are still inadequately evaluated. To date, limited research data is available to make comparisons across the various materials. The mechanical properties of dental materials play an essential role in their clinical success. Studies have revealed that the main reason for dental restorations failure is their fracture after clinical use [14-16]. Although long-term clinical studies remain the gold standard for assessing dental materials' clinical performance and longevity, well-designed laboratory investigations can provide a valuable measure and a meaningful prediction of materials' properties. The fracture resistance of ceramics is a critical factor influencing their durability. Flexural strength is frequently used to evaluate fracture resistance of brittle materials. Materials with high flexural strength provide restorations with less susceptibility to bulk fracture [17]. One of the methods that are used to test mechanical resistance is static loading until fracture [6]. In the literature, different loading methods are available to assess the flexural strength of ceramic materials; these include uniaxial and biaxial flexural tests. The biaxial strength testing has been used for many years and is considered a more reliable method of assessing the strength of brittle dental ceramic materials compared to uni-axial flexural tests [18,19].

By virtue of their location in the oral cavity, dental materials are not only required to resist different mechanical forces, but also need to show stability as they are exposed to moisture, various temperatures and fluctuating pH values. These changes can lead to aging, which may affect their mechanical properties over time. In-vitro studies can be designed to simulate the intra-oral conditions to some extent to provide the so-called accelerated artificial aging. The most common method of artificial aging is by exposing dental materials to thermocycling through water baths at 5°C and 55°C or artificial saliva for a number of cycles followed by fatigue or mechanical cycling to assess their durability and behavior [13]. Accurate reproduction of the oral environment is quite difficult and not feasible. However, it has been reported that by applying cyclic forces in artificial saliva, under fluctuating temperature, the effect of hydrothermal aging might be estimated [20-22].

The current in-vitro study sought to compare the mean flexural strength of three commercially available dental ceramic materials; IPS e.max Cad, Vita Suprinity and Ceramill zolid ht+ under ISO standards. The effect of low-grade hydrothermal aging on the flexural strength of these materials was also investigated. To our knowledge, only few studies in the literature have investigated the effect of low-grade hydrothermal aging on Lithium disilicate and Zirconia reinforced lithium silicate ceramic materials and research is still required for valid and evidence-based comparisons across materials.

The null hypothesis (H0) of this study is: Hydrothermal aging has no effect on the flexural strength of IPS e.max Cad,Vita Suprinity and Ceramill zolid ht+ under ISO standard.

Materials and methods

Specimens Preparation

The materials used in the study are listed in Table 1. Using computer-aided machining (Ceramill motion II; Amann Girrbach, Koblach, Austria) disc-shaped specimens, (n=180), were designed and sliced from prefabricated blocks of IPS e.max Cad, Vita Suprinity. and Ceramill zolid ht. The diameter of the discs was 10 mm, and the thickness was 1.2 ± 0.02 mm (Figure 1).

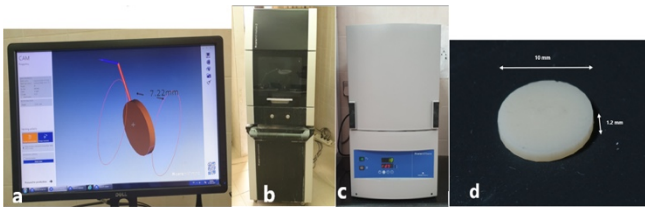


Figure 1. (a) Designing for the specimens. (b) Ceramill motion II machining. (c) Ceramill therm sintering oven. (d) Finished sample.

Sintering was performed in a sintering oven (Ceramill therm; Amann Girrbach, Koblach, Austria) according to the recommendations of their corresponding manufacturer. The compensation for sintering shrinkage for Zirconia was predetermined. Specimen dimensions was measured using a digital caliper (Guanglu, Gullin, China) with an accuracy of ± 0.02 mm (Figure 2). All specimens were stored in distilled water at room temperature for 24 hours prior to testing.

Biaxial Flexure Test

Static loading until fracture using the biaxial flexural test was carried out to load specimens in the present study.

A computer-controlled electromechanical universal testing machine (Jinan Testing Equipment, WDW-20, China) (Figure 3) and test set-up in accordance to the ISO standard 6872 (2008) were used to determine the load at facture (Newton) of the various ceramic discs in the current study. The support area for the discs consisted of three steel balls with a diameter of 3.2 mm each, positioned 120^{O} distance from each other, creating a circle of Ø 10 mm (ISO Standard 6872, 2015) (Figure 4). A flat circular tungsten piston (Ø = 1.6 mm) was used to apply an increasing load (1 mm/min) until catastrophic failure using a universal testing machine (Jinan Testing machine) (Figure3).

Aging procedure

Half of each group (n=30) was submitted to a low-grade hydrothermal aging for 20 hours in a steam autoclave (Euronda, Italy) at 134◦C and 2 bars (200KPa).

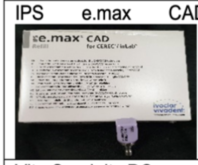
Material	Basic chemical structure /composition	Code	Manufacturer
IPS e.max CAD 	Lithium disilicate glass-ceramic (SiO_2, Li_2O, K_2O, MgO, P_2O_5, Al_2O_3)	E	Ivoclar Vivadent, Schaan, Lichtenstein
Vita Suprinity PC 	Zirconia reinforced lithium silicate glass-ceramic (SiO_2, Li_2O, K_2O, P_2O_5, Al_2O_3, ZrO_2, CeO_2, pigments)	S	Vita Zahnfabrick, Bad Säckingen, Germany
Ceramill zolid ht+ 	Zirconia (3 mol% Y-TZP: ZrO_2 + HfO_2 + Y_2O_3 > 99%, Y_2O_3:)	Z	Amann girbach, Koblach, Austria

Table1. Materials used in this study.

Figure 2. Guanglu digital caliper used to check dimensional accuracy.

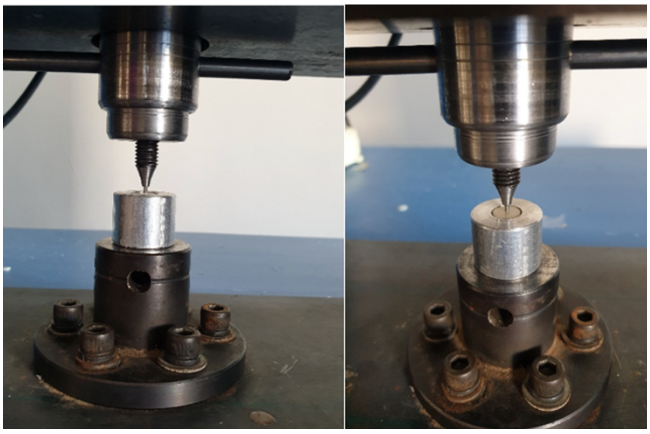


Figure 3. Disc specimen under loading showing specimens in contact with the piston using Jinan Testing machine.

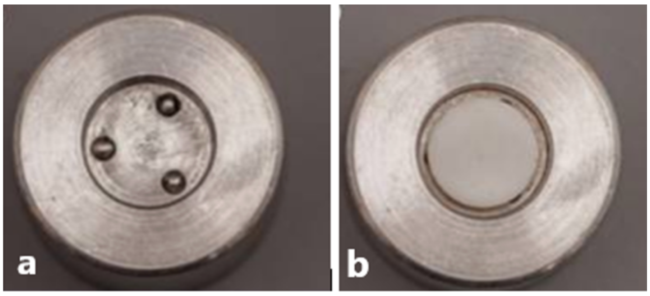


Figure 4. (a)Metal housing device with three balls to support the discs. (b) Specimen positioned in contact with three balls.

Statistical Analysis

Statistical Package for Social Science (SPSS) computer software (SPSS 19.0, Inc. Chicago, USA) was used to analyze the data. Descriptive Analyses (Mean and Standard deviation), paired t-test, independent t-test, One-Way ANOVA and Post-Hoc (Bonferroni) tests were used for multiple comparisons across the groups. A significant difference is assumed to exist among the groups if p-value was less than 0.05 ($p < 0.05$).

Results

The mean values of before- and after-aging flexural strength for the tested materials are shown in Table 2. A statistically significant reduction in the flexural strength was seen in the S and Z groups after aging process (Table 2).

The mean difference value between before and after aging values were compared for the various groups. E group showed a statistically significant higher value compared to Z group. On the other hand, S group mean value between before and after aging values did not show any significant difference compared to the other groups (Table 3). When the mean flexural strength values were compared between groups, a significantly higher strength was seen for Z group compared to the other groups at baseline and after aging (Table 3).

	Before/After	Mean (MPa)	Std. Deviation	Sig. (2-tailed)
E	Before	228.1867	73.83138	0.743
	After	223.1413	39.60235	
S	Before	242.7493	52.85156	0.000*
	After	189.2000	37.91003	
Z	Before	589.8453	96.48818	0.001*
	After	509.4640	84.18663	

Table2. Before- and after- aging values of flexural strength for each group (MPa). (Bonferroni, n=30,*P<0.05).

	(I) E_S_Z	(J) E_S_Z	Mean Difference (MPa)	Std. Error	Sig.
Before	E	S	-14.56267	19.75085	1.000
		Z	-361.65867*	19.75085	0.000*
	S	E	14.56267	19.75085	1.000
		Z	-347.09600*	19.75085	0.000*
	Z	E	361.65867*	19.75085	0.000*
		S	347.09600*	19.75085	0.000*
After	E	S	33.94133	14.97621	0.078
		Z	-286.32267*	14.97621	0.000*
	S	E	-33.94133	14.97621	0.078
		Z	-320.26400*	14.97621	0.000*
	Z	E	286.32267*	14.97621	0.000*
		S	320.26400*	14.97621	0.000*
Mean differen ce	E	S	48.50400	25.29197	0.175
		Z	75.33600*	25.29197	0.011*
	S	E	-48.50400	25.29197	0.175
		Z	26.83200	25.29197	0.875
	Z	E	-75.33600*	25.29197	0.011*
		S	-26.83200	25.29197	0.875

Table 3. The mean difference in flexural strength and the standard deviation were calculated and compared for the study groups. Before, after and, the mean before/after values were compared. (Bonferroni, n=30,*P<0.05).

Discussion

The current in-vitro study is the first to compare the mean flexural strength of three commercially available dental ceramic materials; IPS e.max Cad, Ceramill zolid ht+, and Vita Suprinity under ISO standards. The effect of low-grade hydrothermal aging on the flexural strength of these materials was also investigated.

Recently, a wide variety of new dental materials have been introduced in response to the growing demand for restorations with improved aesthetic and mechanical properties which can serve as alternative to metal and metal-ceramic restorations. The newly introduced ceramic systems present an efficient alternative for metal-ceramic restorations, because of their superior aesthetic properties. However, brittleness is considered the main disadvantage and limiting factor for their use especially in the posterior region [2]. Research is being conducted worldwide to test strengths and weaknesses of these materials.

In the oral environment, dental materials can be subjected to complex compressive, tensile and shear forces. However, since brittle materials are weaker under tensile forces, measurement of tensile strength can be considered a meaningful test of materials' failure. Flexural strength measures a material's resistance to tensile forces. This strength can be evaluated through a uniaxial or biaxial flexural tests. In the current study, samples were tested using a biaxial flexural test. The biaxial flexural test was added to the ISO standard for dental ceramics (ISO 6872-1994) as it is not influenced by the specimen's size, shape, or volume. Other advantages for the biaxial test include ease of test piece preparation and ability to test a large effective surface area. Additionally, the test provides an equi-biaxial stress distribution, which is more searching for defects than a uniaxial distribution (Lohbauer & Belli, 2022).

A wide variety of loading arrangements have been developed for biaxial flexural tests such as ring-on ring, piston-on-ring, ball-on ring, ring-on-ring, and piston-on three balls. The latter is the ASTM standard (ASTM, F394-789, 1991) for biaxial flexure test. In consistence with previous studies [23,24], specimens of all groups have been designed and milled using computerized technology (Ceramill CAD/CAM System, Amann Girrbach) following the recommendations by ISO:6872-2015. As there is no standard method exists to measure the strength of the non-standardized and geometrically complex dental restorations, a well-defined shape specimen like a disc or bar, could be often used as indicators of the material's structural performance.

In the current study, Z group had the highest flexural strength, followed by S group, and E group in a decreasing order. Thus, the first null hypothesis was rejected. The variation in the fracture resistance of the three CAD/CAM material tested in this study could be attributed to their different compositions and microstructure. Zirconia-reinforced lithium sillicate glass ceramics (Vita Suprinity) have a high glass content with a large number of very fine-grained lithium crystallites. Likewise, Lithium disilicate (IPS-emax) consists of 70% lithium disilicate crystals which are embedded in a glassy matrix. The high glass content in these materials contributes to their low flexural strength, whereas Z group is basically composed by crystals with a negligible (almost absent) glassy phase. The higher fracture load values for the Z group compared to the other tested materials were expected, since the transformation toughening phenomena takes place in dense tetragonal zirconia materials doped by yttrium when subjected to mechanical stimuli. The tetragonal zirconia phase transforms to the monoclinic phase, causing a local volume increase of approximately 4% increasing the material strength [25]. This increase in volume counteracts crack propagation by causing compression at the tip of the crack. The high fracture load recorded for the Z group in this study compared to E group is in agreement with previous studies [26-28], justifying their use for restorations subjected to high stress concentration, such as posterior crowns and multi-unit fixed partial dentures.

The flexural strength of the S group was slightly higher than those of E group. This difference could be attributed to the incorporation of Zirconia particle in the glassy matrix. However, this difference was not statistically significant. This finding contradicts other studies in the literature that reported a significantly higher mechanical properties (flexural strength and fracture resistance) for S compared to E group [29,30]. This difference might be attributed to the difference in the size of the specimens and the method of testing.

With regard to the effect of low-grade hydrothermal aging on the flexural strength of the tested materials, the strength of Z group was significantly reduced by this aging. These results are consistent with findings by Flinn et al. [31] However, other studies reported no significant effect on flexural strength after aging [32-35]. This difference could be attributed to differences in the time of treatment, brand and composition of the zirconia system investigated. Nevertheless, it is worth mentioning that despite this reduction in

Zirconia strength, the fracture resistance was still sufficient to withstand the expected occlusal loadings in the molar regions and this concurs with findings of Nakamura et al. [36].

S group showed a significant reduction in its flexural strength after being subjected to the low-grade hydrothermal aging process. This is could be also attributed to the presence of Zirconia and hence the effect of low-grade hydrothermal degradation and subsequent slow tetragonal-to-monolithic phase transformation process which leads to micro-cracking and reduction in zirconia strength [37].

On the other hand, the E group did not show significant reduction in its flexural strength after being subjected to the aging process. This is consistent with Andr´e et. al. study [38]. This seems to be a promising fact considering the long-term durability of the material.

Based on the previous discussion, the null hypothesis (H0) was partially rejected, since hydrothermal aging did affect S and Z group of the ceramic materials for monolithic restorations with different microstructures resulting in different flexural strengths. The one exception was the E group, as aging did not affect the flexural strength of the material.

In the present study, the specimens were subjected to a low-grade hydothermal aging of 134°C, 2 bar (200KPa) for 20 hours, and then they were statically loaded until failure. This aging protocol has been chosen since it was demonstrated that this protocol promotes an extensive tetragonal-monoclinic phase transformation (approximately 55-80% monoclinic-phase content) [9,39-42]. Additionally, it was found that flexural strength was significantly reduced only when at least 50% of monoclinic phase detected on the material's surface [43]. This process accelerates crack propagation as more monoclinic phase is formed and can result in reduced toughness, strength, and density, leading to failure of the restoration [39].

According to theoretical calculations, 1 h at 134◦C corresponds to 3–4 years in-vivo [44]. By this, 20 h of in-vitro aging have theoretically the same effect as a restoration in the oral cavity for 60–80 years, which by far exceeds the expected lifespan for a fixed prosthesis. Despite the criticism that this test does not correspond to actual clinical conditions and lifespan it remains an efficient method to estimate the long-term performance of these materials.

The current study has few limitations; the first being an in-vitro investigation which means that no matter how well the experiment was designed, it cannot reproduce all the relevant clinical conditions. However, laboratory studies do allow measurement of factors that could influence clinical performance and usability of new materials but cannot be tested *in-vivo* for ethical or practical reasons.

In addition, static loading was applied instead of the more clinically-simulating cyclic loading. However, static loading provides the upper limit force that a material can withstand before failure. Thus, it is considered essential for comparing different groups of dental ceramics in terms of fracture resistance.

To date, research data on the new restorative materials are still limited, often controversial and short-term. These highly promising materials need further studies, both in-vivo and in-vitro, in order to precisely define physico-mechanical properties, clinical indications, limitations, and long-term performance.

Conclusion

The flexural strength was highest for yttria-stabilized tetragonal monolithic zirconia. The various compositions and microstructure of the newly proposed ceramics contributed to variable mechanical properties and different behavior under hydro-thermal aging. It can be said that the addition of Zirconia particles to lithium crystals improved its fracture load but lowered its resistance to the low-grade hydrothermal aging.

The recent CAD-CAM restorative materials seem to be highly promising ceramics; however, they need further studies, both in vivo and in vitro, in order to precisely define their clinical indications, physico-mechanical properties, and long-term performance.

Declaration of Interest

The authors report no conflict of interest.

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