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In-Vitro Comparative Evaluation of the Physical Properties of Three Dimensional-Printed and Milled High Performance Ceramics

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In-Vitro Comparative Evaluation of the Physical Properties of Three Dimensional-Printed and Milled High Performance Ceramics

Abstract

Aim: to investigate the potential role of 3D printing to produce zirconia

restorations and to assess the mechanical properties of the 3D printed zirconia.

Hypotheses: 1) The flexural strength of 3D printed yttria-stabilized zirconia is comparable or superior to milled yttria-stabilized, isostatic pressed zirconia, and 2) thermocycling and chewing simulation does not affect the flexural strength of 3D printed yttria-stabilized zirconia.

Material and methods: 30 bars of printed yttria-stabilized zirconia and 10 bars of milled yttria-stabilized, isostatic pressed zirconia were utilized in this study. Printed zirconia bars were divided in 3 groups (10 bars per group): untreated, thermocycling and chewing simulation. Flexural strength test was performed on all the samples using a three-point bend test. One-way ANOVA analysis compared the 3 groups of printed zirconia samples, and Mann-Whitney test was used to compare the non-treated printed zirconia group to the milled zirconia group.

Results:

No statistically significant difference between the three groups of printed zirconia samples was found ($P = 0.119$). No statistically significant difference between the non-treated printed zirconia group and non-treated samples of milled yttria-stabilized, isostatic pressed zirconia was found ($P = 0.178$).

Conclusion:

No statistically significant differences in flexural strength were detected between yttria-stabilized printed zirconia and milled yttria-stabilized, isostatic pressed zirconia, and non-treated, thermocycling and chewing simulation tested printed zirconia samples. These results indicate the promising role of 3D printing in the fabrication of zirconia. Additional studies are needed to explore the full potential of this technology.

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In-Vitro Comparative Evaluation of the Physical
Properties of Three Dimensional-Printed and Milled
High Performance Ceramics

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Thesis for Masters of Science in Oral Biology

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Literature Review

Ceramics have been the state of art in esthetics dental materials for the past 100 years. Initially, their use was limited to veneers over a metal substructure in the anterior region. However, the lower biocompatibility and translucency of metals, the time consuming and technique sensitive conventional powder build-up and firing as well as the chipping of the veneering porcelain have pushed the industry towards a continuous development and improvement in strength, esthetics, and methods of fabrication of ceramics. (1) Presently, dental ceramics have evolved for use as the substructure in multilayered restorations, with the so-called "all-ceramic materials".

The introduction of all-ceramic materials revolutionized dental materials research as well as clinical practice. All-ceramic materials mimic seamlessly the optical properties of teeth, while at the same time, they enable a reduction in technique sensitivity and production costs.

Over the last several decades, the dental applications for newly developed ceramics have expanded considerably. Although many early ceramics have been replaced, some still remain relevant. According to the latest classification (2), dental ceramics can be classified as follows:

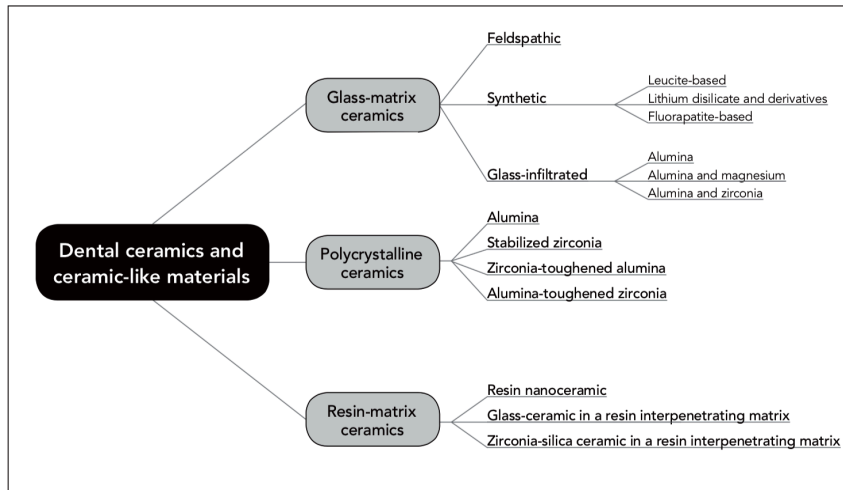


Fig 1. Overview of the proposed classification system of all-ceramic and ceramic-like materials by Gracis et al. 2015 (2).

✓ Glass-matrix ceramics:

- Feldspathic: A ternary material composed of clay/kaolin (hydrated aluminosilicate), quartz (silica), and naturally occurring feldspar (a mixture of potassium and sodium aluminosilicates). This ceramic provides the best esthetic results amongst the currently available ceramics. However, its brittleness and mechanical properties limit the utility of this material. This ceramic is primarily used as a veneering material, either applied on a metal alloy, a ceramic substrate, or bonded to the tooth surface. Its fracture toughness is approximately $1.0 \text{ MPa m}^{1/2}$ and its flexural strength is approximately 100 MPa . Given these properties, feldspathic porcelain is not a suitable material for load-bearing molar restorations (1).
- Synthetic: These leucite, lithium-disilicate and fluorapatite-based materials are industrially synthesized. Clinical uses include veneering material, inlays and onlays, partial and full contour crowns, and three-unit fixed partial dentures (FPD) in the anterior, premolar, or posterior region. The most commonly used synthetic ceramic is the lithium-disilicate. When fully

crystallized, it possesses a flexural strength up to 400 MPa and optimal translucency for producing highly esthetic dental restorations (3). Additionally, lithium disilicate reinforced with zirconia was introduced to enhance the strength of this material.

- Glass infiltrated: These material are produced by adding oxides such as alumina, alumina and magnesium, and alumina and zirconia to feldspathic porcelain, reducing the quartz content. Proportion of alumina exceeding 50% results in a significant reduction of translucency of the final product. As an aside, due to its biocompatibility, low friction and relatively resistance to wear and corrosion, alumina is suitable to be used as bone replacement material (2). Alumina exhibits low thermal conductivity and its flexural strength is approximately 500 MPa. In dentistry, restorations made out of this glass-infiltrated material were initially fabricated using the slip-casting technique. With the development of the CAD-CAM (Computer Aided Designed - Computer Aided Manufactured) technology, alumina blocks produced with high-purity Al_2O_3 (to 99.5%) were introduced to the market to replace older manufacturing methods. However, high elastic modulus (the highest of all ceramics) ($E = 300 \text{ GPa}$) makes the material prone to bulk fractures (2, 4). This, along with the increased popularity of lithium disilicate and zirconia, decreased the fabrication of alumina restorations.

- ✓ Resin-matrix ceramics: Recently, a new category of hybrid dental materials has been promoted. These materials were not considered ceramics until 2013, when the ADA definition of porcelain/ceramic changed from "*nonmetallic inorganic materials usually processed by firing at a high temperature to achieve*

desirable properties” to “*pressed, fired, polished, or milled materials containing predominantly inorganic refractory compounds—including porcelains, glasses, ceramics, and glass-ceramics*”. Resin-matrix materials are composed of an organic matrix (polymer) highly filled with ceramic particles (> 50% by weight) (4). The modulus of elasticity of these materials is similar to dentin, with a modulus of resilience significantly higher than previously described ceramics. This means that significantly higher stress can be absorbed by the material without permanent deformation. Commercially, blocks for milling are widely distributed. Compared to other ceramic materials, milling time for resin-matrix ceramics is shorter and milling burs have a longer lifetime. There is no need for sintering or crystallization firing after milling; simply final gloss and smoothness of the restoration are needed. This is typically rendered via surface polishing. Restorations made from these materials are “gentle” to the opposing dentition, and may be easily repaired intraorally, if necessary. Thus, they are mainly used for chairside-fabricated restorations.

✓ Polycrystalline ceramics: Alumina and zirconia.

The German chemist Martin Heinrich Klaproth discovered zirconia in 1789. This material, due to its mechanical properties (high mechanical strength, toughness, corrosion resistance, and excellent biocompatibility) (1) has increased in popularity over the past few years, becoming one of the most widely used all-ceramic dental materials. Pure zirconia is a polymorphic material that exhibits allotropy, forming three crystallographic structures, each determined by the temperature. The phases observed are the following: cubic phase (c) from 2680°C (melting point) to 2370°C; tetragonal phase (t) from 2370°C to 1170°C; and monoclinic phase (m)

from 1170°C to room temperature. The spontaneous transformation from the t phase (higher material density) to the more stable m phase (lower material density) is associated with a volumetric increase of approximately 4%. If a crack were initiated by external stresses (grinding, cooling, and impact) on the surface of zirconia, the stress concentration at the top of the crack would transform the small tetragonal particles to larger monoclinic particles as the material expanded between the aforementioned phases. This transformation leads to compressive stress in the vicinity of the crack that halts crack propagation, eventually preventing the failure of the zirconia restoration and enhancing the fracture toughness (1). This phenomenon is appropriately called “phase transformation toughening” (4).

Different oxides are added to zirconia to stabilize its tetragonal phase at room temperature. The most commonly form of dental zirconia is partially stabilized tetragonal zirconia polycrystals (TZP) stabilized with a 3mol% of yttria (Y_2O_3) in a solid solution (Y-TZP 3mol%). This form has a flexural strength of more than 1200 MPa and a fracture toughness of 5 to 10 $MPa \cdot m^{1/2}$ after machining and sintering (5-7).

These characteristics of Y-TZP enable its use initially in monolithic dental restorations. However, concerns arose regarding the effect of its hardness on the wear of the opposing teeth/restorations relative to that observed with other ceramics. However, recent studies have shown that the wearing rate of polished zirconia on the opposing enamel is within the physiological range reported in the literature (1).

Traditionally a zirconia core was milled and posteriorly veneered with porcelain, usually feldspathic, to fabricate a restoration with the strength and toughness of the zirconia core and the esthetics of the veneering porcelain. However, this outer layer is susceptible to failure from delamination, cracking, and chipping (4, 8), limiting its use, especially in areas with heavy occlusal forces or in patients with bruxism. Monolithic zirconia restorations, with no veneering material, may overcome the disadvantage of fractures observed in this low-strength veneering material, but at the expense of esthetics, due to the high opacity of the zirconia.

The dental industry aimed to create a more esthetic monolithic zirconia without drastically compromising the mechanical properties that make this material popular. Today, the development of high translucent zirconia (which shows outstanding translucency for esthetic restorations, requiring no facial feldspathic veneering with preserved mechanical properties of zirconia) and the coloring pre-sinterization process have overcome this issue. The need for less invasive tooth preparations and the advances in CAD-CAM technology that made possible the milling of full-contour restorations with high-strength Y-TZP, have promoted increased clinical use of monolithic zirconia restorations.

CAD-CAM is the technology used to mill zirconia restorations. This subtractive manufacturing technique uses a computer for both design and fabrication of a dental restoration. Subtractive manufacturing involves removing sections of a large block of a given material by machining or cutting it away until the designed shape remains.

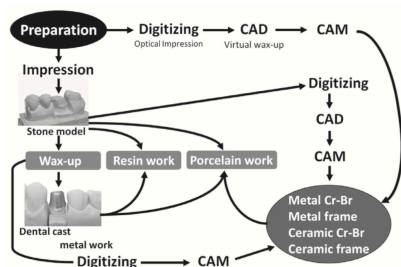


Fig 2. Overview of the current dental CAD-CAM workflows used for the fabrication of Crown-bridge restorations from Miyazaki et al. 2019 (5).

Instead of taking conventional impressions, that are poured and subsequently scanned and transferred to the design software, the advances in the digital workflow allows us now to directly digitize teeth using an intraoral scanner. The scanner generates a digital file, which is then transferred into a program used to design a virtual wax-up, establishing the shape and dimensions of the restoration (Computer Aided Designed). After that, CAM technology transforms the design into a restoration, milling it from a block made of the elected dental material (subtractive manufacturing) (5, 9) (Fig 2).

Before CAD-CAM was developed, metal restorations were fabricated by casting, which resulted in greater defects and cracks in the microstructure of the final restorations (9). Currently, CAD-CAM systems provide more standardized fabrication process, eliminating the variability associated with casting or press manufacturing. Clinicians are able to deliver restorations with improved fitting accuracy with CAD-CAM compared to those obtained in the past, with a tooth-restoration gap smaller than 80 μm (10).

As mentioned before, CAD-CAM is the current technique for fabrication of zirconia restorations. Blocks of this material are available in its pre-sintered or sintered state. To improve the quality of the restoration and enhance the durability of the drills, pre-sintered chalk-like blocks ("green" stage) are the most commonly used. The milling process produces restorations that are enlarge relative to their desurde size. The next step in the fabrication process is sintering (1350–1500°C), where shrinking of the restorations (20–25%) occurs, causing structure densification, that hielos the final physical properties of the material. The volumetric change depends on the specific composition of the block and it must be accounted for during the designing phase (5). Zirconia blocks are also available

as sintered blocks composed of hot isostatic pressed 'HIP' zirconia ("white blocks"). In this case, the starting material exhibits its final strength and does not need to be sintered. However, milling time is longer and the wear of the milling drills is also higher.

Recent studies have shown that clinically acceptable marginal fits may be achieved (marginal opening below 120 μm) utilizing CAD-CAM technology and this fit is superior to that achieved with cast restorations (5, 6, 10, 11).

In addition to reducing labor and fabricaciones, the possibilities offered by CAD-CAM technology in conjunction with the advent of new materials, are virtually limitless (9). To overcome the disadvantages of CAD-CAM as a subtractive technique, additive manufacturing was developed. The great advantage associated with all additive techniques is minimal to no material waste.

Currently, numerous additive techniques have dental applications including selective laser sintering, direct three-dimensional (3D) printing, and stereolithography. Selective laser sintering is commonly used to fabricate metal alloy structures. Three-dimensional printing and stereolithography have been extensively used to fabricate anatomical models based on CT scans, surgical guides for reconstructive and implant surgeries, and more recently, for customized bone grafts or scaffolds for tissue regeneration. In the field of dental prosthetics, the application of 3D printing to the fabrication of ceramics is yet to be explored (12). 3D printing zirconia could potentially minimize or eliminate the waste of starting material of CAD-CAM, enhance production efficiency and allow for multilayer restorations. Although these future directions are not yet utilized clinically, this technology is currently in development.

The direct 3D printing of dental ceramics works similar to the traditional inkjet printing, but instead of ink; it employs a suspension containing ceramic particles mixed with a photosensitive liquid polymer that acts as a binder (acrylates or epoxy monomers). This binder is polymerized during printing and subsequently burnt out during the ceramic sintering process.

Studies on application of 3D printing technologies to ceramics are still lacking. In particular strength and fracture toughness of the resultant products are areas that demand further research. Therefore, the aim of this project is to investigate the potential role of 3D printing to produce zirconia restorations and to assess the mechanical properties of 3D printed zirconia.

References

1. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. *Journal of prosthodontic research* 2013;57:236-261.

2. Gracis S, Thompson VP, Ferencz JL, Silva NR, Bonfante EA. A new classification system for all-ceramic and ceramic-like restorative materials. *The International journal of prosthodontics* 2015;28:227-235.
3. Willard A, Gabriel Chu TM. The science and application of IPS e.Max dental ceramic. *The Kaohsiung journal of medical sciences* 2018;34:238-242.
4. Bajraktarova-Valjakova E, Korunoska-Stevkovska V, Kapusevska B, Gigovski N, Bajraktarova-Misevska C, Grozdanov A. Contemporary Dental Ceramic Materials, A Review: Chemical Composition, Physical and Mechanical Properties, Indications for Use. *Open access Macedonian journal of medical sciences* 2018;6:1742-1755.
5. Miyazaki T, Hotta Y, Kunii J, Kuriyama S, Tamaki Y. A review of dental CAD/CAM: current status and future perspectives from 20 years of experience. *Dental materials journal* 2009;28:44-56.
6. Hamza TA, Sherif RM. In vitro evaluation of marginal discrepancy of monolithic zirconia restorations fabricated with different CAD-CAM systems. *The Journal of prosthetic dentistry* 2017;117:762-766.
7. Harada K, Shinya A, Gomi H, Hatano Y, Shinya A, Raigrodski AJ. Effect of accelerated aging on the fracture toughness of zirconias. *The Journal of prosthetic dentistry* 2016;115:215-223.
8. Zhang Y, Lawn BR. Evaluating dental zirconia. *Dental materials : official publication of the Academy of Dental Materials* 2019;35:15-23.
9. Silva LHD, Lima E, Miranda RBP, Favero SS, Lohbauer U, Cesar PF. Dental ceramics: a review of new materials and processing methods. *Brazilian oral research* 2017;31:e58.
10. Boitelle P, Mawussi B, Tapie L, Fromentin O. A systematic review of CAD/CAM fit restoration evaluations. *Journal of oral rehabilitation* 2014;41:853-874.

11. Kunii J, Hotta Y, Tamaki Y, Ozawa A, Kobayashi Y, Fujishima A, et al. Effect of sintering on the marginal and internal fit of CAD/CAM-fabricated zirconia frameworks. *Dental materials journal* 2007;26:820-826.
12. Oberoi G, Nitsch S, Edelmayer M, Janjic K, Muller AS, Agis H. 3D Printing- Encompassing the Facets of Dentistry. *Frontiers in bioengineering and biotechnology* 2018;6:172.

Abstract

Aim: to investigate the potential role of 3D printing to produce zirconia restorations and to assess the mechanical properties of the 3D printed zirconia.

Hypotheses: 1) The flexural strength of 3D printed yttria-stabilized zirconia is comparable or superior to milled yttria-stabilized, isostatic pressed zirconia, and 2) thermocycling and chewing simulation does not affect the flexural strength of 3D printed yttria-stabilized zirconia.

Material and methods: 30 bars of printed yttria-stabilized zirconia and 10 bars of milled yttria-stabilized, isostatic pressed zirconia were utilized in this study. Printed zirconia bars were divided in 3 groups (10 bars per group): untreated, thermocycling and chewing simulation. Flexural strength test was performed on all the samples using a three-point bend test. One-way ANOVA analysis compared the 3 groups of printed zirconia samples, and Mann-Whitney test was used to compare the non-treated printed zirconia group to the milled zirconia group.

Results:

No statistically significant difference between the three groups of printed zirconia samples was found ($P = 0.119$). No statistically significant difference between the non-treated printed zirconia group and non-treated samples of milled yttria-stabilized, isostatic pressed zirconia was found ($P = 0.178$).

Conclusion:

No statistically significant differences in flexural strength were detected between yttria-stabilized printed zirconia and milled yttria-stabilized, isostatic pressed

zirconia, and non-treated, thermocycling and chewing simulation tested printed zirconia samples. These results indicate the promising role of 3D printing in the fabrication of zirconia. Additional studies are needed to explore the full potential of this technology.

Introduction

The fabrication of dental restorations has substantially changed over the past decade. Two major advancements in dental prostheses manufacturing were the development of CAD-CAM technology and the introduction of “all ceramic” materials. “All ceramic” materials for dental restorations mimic very naturally the optical properties of teeth. Additionally, some ceramics, like zirconia, present mechanical properties that allows them to be employed as monolithic materials. This eliminates risks of facial veneering and porcelain chipping without compromising esthetics or long-term physical stability of the prosthesis (1). Currently, CAD-CAM technology enables fabrication of zirconia restorations. This subtractive manufacturing technique involves removing sections of a large zirconia block by machining or cutting until the designed shape remains. According to previous reports, up to 90% of the prefabricated block is wasted during this process (2).

To overcome this major disadvantage, additive manufacturing was developed. Selective laser sintering, direct 3D printing, and stereolithography are some additive techniques that have been applied to dentistry. Selective laser sintering is commonly used to fabricate structures of metal alloys. 3D printing and stereolithography are extensively used to fabricate anatomical models based on CT scans, surgical guides for reconstructive and implant surgeries, and more recently, for customized bone grafts or scaffolds in tissue regeneration (3). Regarding dental prosthesis manufacturing, printing of “all ceramic” materials has yet to be explored and limited research has been conducted in this field.

The aim of the present study was to investigate the potential role of 3D printing to produce zirconia restorations and to assess the mechanical properties of the 3D printed zirconia. Our hypotheses are 1) the flexural strength of 3D printed yttria-stabilized zirconia is comparable or superior to milled yttria-stabilized, isostatic pressed zirconia, and 2) thermocycling and chewing simulation does not affect the flexural strength of 3D printed yttria-stabilized zirconia.

Materials & Methods

Statistical power analysis to estimate the sample size needed was performed using the data from Kosmac et al. 2000 (4). Thirty rectangular bars of printed 3 mol% yttria-stabilized zirconia (LithaCon 3Y 230 ceramic, Lithoz, Austria), which dimensions were 25 mm x 5 mm x 2 mm, were utilized for this project. The zirconia suspension for printing was fabricated using a binder for joining particles. After printing the bars, the binder was burned out during sintering. Twenty bars received a treatment to investigate the possible effects of aging and chewing in the flexural strength of printed zirconia (thermocycling or chewing simulation), while ten were untreated controls. Additionally, 10 bars of milled yttria-stabilized, isostatic pressed zirconia (Prettau® Zirconia, Zirkonzahn, Italy) with the same dimensions were tested for comparison as a standard in zirconia for dental prostheses fabrication.

- Thermocycling

Ten bars of printed zirconia underwent 40,000 thermal cycles in a thermocycler (SD Mechatronik GMBH, Feldkirchen-Westerham, Germany) to simulate 4 years of artificial aging by means of cyclical temperature changes. The thermocycler consists basically on two baths and a basket where the samples are placed. This

basket is immersed in an alternating manner from a bath of warm water to a second bath filled with cold water. The parameters for the thermocycling of our samples were the following:



- Temperature of hot bath: 55°C / 131 F
- Temperature of cold bath: 5°C / 41 F
- Dwell time (hot & cold): 30 s
- Drain time: 5 s

Photograph of the thermocycler used to treat our samples.

- *Chewing simulation*

Ten bars of printed zirconia underwent 960.000 cycles in a chewing simulator machine (SD Mechatronik GMBH, Feldkirchen-Westerham, Germany) to simulate 4 years of chewing loading in the mouth. The chewing simulator consists of several chambers in which an antagonist strikes the specimens. This repetitive loading force is produced by weights and these weights are mounted on a traverse that is raised or lowered by a servo motor. The traverse connects all the weights together so that the kinematics are identical for all antagonists, with the aim of producing comparable results. Samples were surrounded by artificial saliva throughout the testing. The parameters for the chewing simulation of our samples were the following:

- Load vertical: 49 N
- Upward travel: 2 mm
- Upward speed: 60 mm / s
- Downward travel: 2 mm
- Downward speed: 40 mm / s

- *Flexural strength testing*

Flexural strength of the printed and milled bars was determined using a three-point bend test on a universal testing machine (Instron 4204, Norwood, MA, USA) following ISO 6872. The load frame is a tension/compression type employing a moving (screw-driven) crosshead. A 50KN load cell and a crosshead speed of 1mm/min were used in this experiment.

The load at fracture was used to calculate flexural strength using the following formula:

$$\sigma = \frac{3FL}{2bd^2}$$

where F is the load to fracture at the fracture point, L is the length of test specimen between supports (mm), b is the specimen width (mm) and d is the specimen thickness (mm).

The values obtained were used to calculate the mean flexural strength and standard deviation of each of the four sample groups (printed non-treated, printed thermocycled, printed chewing simulated and milled samples). One-way analysis of variance (ANOVA) was used to statistically determine the presence of significant differences in the flexural strength among the three groups of printed zirconia samples. The values of flexural strength of the milled isostatic pressed zirconia samples were compared to the flexural strength of our non-treated printed zirconia samples. This data was not normally distributed, and Mann-Whitney Test was used to perform this statistical analysis.

Results

The mean values of flexural strength and their respective standard deviations are presented in Table I. One-way ANOVA test showed no statistically significant difference among the three groups of printed zirconia samples ($P = 0.119$). These results indicate that thermocycling and chewing simulation did not affect the flexural strength of our printed zirconia.

Data obtained from flexural strength tests on Prettau® Zirconia samples was used to compare our non-treated printed zirconia to the standard milled isostatic pressed zirconia commercially available. Mann-Whitney test (Table II) showed no statistically significant difference between the non-treated printed zirconia group and the non-treated samples of milled yttria-stabilized, isostatic pressed zirconia ($P = 0.178$). Table III shows flexural strength comparison between non-treated printed and isostatic pressed zirconia expressed in means and standard deviations.

Table I. Flexural strength values for the printed zirconia samples expressed in means and standard deviations.

Group Name	N	Missing	Mean	Std Dev	SEM
Printed No Rx	10	0	855.401	112.560	35.595
Pr Load Cycled	10	0	888.380	59.255	18.738
Printed Thermo	11	0	789.649	133.846	40.356

Table II. Flexural strength comparison between non-treated printed and isostatic pressed zirconia expressed in median for Mann-Whitney test.

Group	N	Missing	Median	25%	75%
Prettau No Rx	9	0	978.383	773.362	1172.344
Printed No Rx	10	0	832.021	818.790	865.659

Table III. Flexural strength comparison between non-treated printed and isostatic pressed zirconia expressed in means and standard deviations.

Group Name	N	Missing	Mean	Std Dev	Std. Error
Prettau No Rx	9	0	936.269	255.021	85.007
Printed No Rx	10	0	855.401	112.560	35.595

Discussion

The present study is, to our knowledge, the second report of flexural strength testing on printed yttria-stabilized zirconia. Furthermore; it is the first attempt to compare flexural strength of this material to the standard in the field of computerized dental prosthesis fabrication, which is undeniably the milled yttria-stabilized, isostatic pressed zirconia.

Our results showed no statistically significant difference in flexural strength between these two materials, suggesting that 3D printing is a potential new method for the fabrication of zirconia. Lack of other studies on this material demand its further exploration.

Two different treatments were carried out on our samples; thermocycling and chewing simulation, to determine if the exposure to the oral environment would affect the flexural strength of this material. Our data showed no statistical significant difference among the non-treated and the treated groups.

Additive manufacturing presents several advantages over conventional milling technology, including reduced production costs and material wastes. This technology is routinely used in dentistry for the fabrication of surgical guides and prosthetic models, among others (3) and undoubtedly, the industry of dental material manufactures is looking at 3D printing as an alternative to compete with commercial CAD-CAM systems in the fabrication of "all ceramic" restorations. The present report is an example of this trend, despite the developmental phases of this technology.

To our knowledge, Osman et al. 2017 (5) represents the only other study published in the literature that investigates the flexural strength of printed zirconia. They utilized a bi-axial flexural test performed on 45 disc-shaped samples that were divided in three groups according to their build direction (0°, 45° and 90°). Their flexural strength values ranged from 822.3 MPa for the 90° group to 943.2 MPa for the 0° group, existing a statistical significant difference between the groups. The values we obtained from our samples are also within the same range and these results are comparable to the ones expected from milled yttria-stabilized isostatic pressed zirconia published on previous reports in the literature. However, no treatment was given to their discs and no milled samples were analyzed in their study.

The possible effect of thermocycling and chewing simulation in milled yttria-stabilized zirconia has been previously studied. It has been determined that they do not affect the translucency (6) or the mechanical properties (7) of this material. The present study aimed to test the effect of these treatments on our 3D printed zirconia bars. It could be hypothesized that, due to the presence of the binder and its elimination after printing, this material could potentially be more porous and therefore more prone to fluid leakage, which could affect its mechanical properties. 960,000 cycles in a chewing simulator or 40,000 thermal cycles in a thermocycler, simulating 4 years of aging in the oral cavity, did not affect the flexural strength of this material, as no statistically significant difference among the treated and non-treated groups was found. These data suggest that printed zirconia flexural strength should be stable over time when exposed to function in the oral cavity.

Although no statistical significant differences were found, the flexural strength of the printed zirconia groups was always lower than the milled zirconia. Could this still be clinically significant? How high does the flexural strength need to be in a dental restorative material? The answers to these questions depend on the uses given to the specific material. The flexural strength is a property that defines, in part, the application of the different ceramics. A comparison in flexural strength of the most commonly used "all ceramic" materials according to their indicated uses may be made using data reported in previous literature. Feldspathic porcelain presents a flexural strength of around 100 MPa, which is enough for this ceramic to be used as a veneer material (8) but not to support the bite loading. Lithium disilicate has been reported to have around 400 MPa of flexural strength, similarly to Alumina which flexural strength ranges around the 500 MPa. These attributes permit use as inlays, onlays, single crowns and anterior 3-unit bridges (9). Zirconia

is known for its superior flexural strength, ranging from 800 to 1200 MPa. This ceramic is capable of supporting posterior load of the bite. It is employed for posterior or anterior restorations, individual or multiple units. Furthermore, optical properties of the last generations of zirconia allow its use as a monolithic material with high esthetic quality.

Although the results presented on this report are promising, the limitations of our study have to be taken into consideration also. Our sample size was small although it falls within our power analysis. Without previously published studies investigating the flexural strength of printed zirconia via the methods described herein, data from flexural strength of milled zirconia was adapted to our power analysis to determine our sample size.

The standard deviations of our samples are considered high. This could be attributed to the printing process itself. In the fabrication of the milled zirconia, the material is pressed, obtaining a dense and homogeneous material. However, in 3D printing, layers of the suspension are deposited over each other, without pressure. In addition, our standard deviations may also be a consequence of the presence of the binder. It is difficult, if not impossible, to predictably control where the binder is deposited within each layer. Binder must be burned out printing, potentially leaving voids that are not equally distributed throughout the material. This most likely makes the material non-homogenous. Furthermore, the composition of the printable powder and the percentages of ceramics or binder in the suspension may alter the mechanical properties of the samples.

Future studies focusing on the development of suitable materials for printing, the improvement of the printing process, and the standardization of the suspensions

are needed. Such studies will determine if homogeneity of printed zirconia is achievable. The optical properties of this material need to be examined to determine if they are comparable to those of the current commercially available standards, like high translucent milled isostatic pressed zirconia.

Clinical significance

In our study, yttria-stabilized printed zirconia showed flexural strength comparable to that of milled yttria-stabilized isostatic pressed zirconia. Thermocycling and chewing simulation did not affect the flexural strength of this printed zirconia. Our results indicate that mechanical properties of printed and milled zirconia may be comparable. Although future studies are needed, 3D printing emerged as a promising method for the manufacturing of zirconia for dental applications as shown in this study.

Appendix

Sample size for ANOVA:

Sample size	9.000
Difference in Means	207.000
Standard Deviation	89.300
Number of Groups	3
Power	0.990
Alpha	0.0500

References

1. Gracis S, Thompson VP, Ferencz JL, Silva NR, Bonfante EA. A new classification system for all-ceramic and ceramic-like restorative materials. *The International journal of prosthodontics* 2015;28:227-235.
2. Strub JR, Rekow ED, Witkowski S. Computer-aided design and fabrication of dental restorations: current systems and future possibilities. *Journal of the American Dental Association* 2006;137:1289-1296.
3. Oberoi G, Nitsch S, Edelmayr M, Janjic K, Muller AS, Agis H. 3D Printing- Encompassing the Facets of Dentistry. *Frontiers in bioengineering and biotechnology* 2018;6:172.
4. Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. Strength and reliability of surface treated Y-TZP dental ceramics. *Journal of biomedical materials research* 2000;53:304-313.
5. Osman RB, van der Veen AJ, Huiberts D, Wismeijer D, Alharbi N. 3D-printing zirconia implants; a dream or a reality? An in-vitro study evaluating the dimensional accuracy, surface topography and mechanical properties of printed zirconia implant and discs. *Journal of the mechanical behavior of biomedical materials* 2017;75:521-528.
6. Alraheem IA, Donovan TE, Rodgers B, Boushell L, Sulaiman TA. Effect of masticatory simulation on the translucency of different types of dental zirconia. *The Journal of prosthetic dentistry* 2019.
7. Harada K, Shinya A, Gomi H, Hatano Y, Shinya A, Raigrodski AJ. Effect of accelerated aging on the fracture toughness of zirconias. *The Journal of prosthetic dentistry* 2016;115:215-223.
8. Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. *Journal of prosthodontic research* 2013;57:236-261.

9. Willard A, Gabriel Chu TM. The science and application of IPS e.Max dental ceramic. The Kaohsiung journal of medical sciences 2018;34:238-242.