Optical properties of CAD-CAM monolithic systems compared: three multi-layered zirconia and one lithium disilicate system

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ABSTRACT

Objective: this in vitro investigation aims to evaluate and compare optical properties of three types of esthetic CAD-CAM monolithic multi-layered zirconia materials with a control (conventional lithium disilicate, IPS e.max CAD).

Methods: Four monolithic CAD-CAM ceramic materials were investigated: Ceramill Zolid® FX Multilayer (ZF), IPS e.max® ZirCAD MT Multi (ZM), Katana® STML (KS) and one lithium disilicate glass-ceramics as a control (IPS e.max® CAD LT; LC). A total of 72 (15/C2 15/C2 1 mm) samples were CAD CAM fabricated and sintered based on sample-size power calculations, and each material comprised 18 samples. The translucency and opalescence parameters with the contrast ratio were evaluated with a dental spectrophotometer over the backgrounds of black and white. The data were analyzed by ANOVA, then Bonferroni post hoc comparison test was made between groups. The statistical significance was set at p < 0.05.

Results: Zirconia materials revealed lower optical properties than the lithium disilicate control LC (P < 0.05). TP values ranged from 14.174 to 20.439. No differences were detected between the zirconia products in terms of TP and CR (P = 1.000). OP values ranged from 5.068 to 10.097. The lowest OP values were found statistically significant for ZF followed by KS and ZM (P = 1.000). LC had the highest TP and OP values, as well as the lowest CR (p < 0.05).

Conclusions: The optical properties of monolithic zirconia systems tested here are still lower than those of lithium disilicate glass-ceramics. The only difference observed between the monolithic CAD CAM zirconia materials, was for the low opalescence parameter for the ZF.

Clinical significance: Multi-layered monolithic zirconia systems have better esthetics by shade layers resembling natural tooth color gradients. CAD-CAM technology allowed for in-office milling and shaping of restorations using these systems. This study reports on their optical properties affecting human vision/perception of natural tooth shade to conclude about their use in the esthetic zones.

1. Introduction

As light contacts the natural tooth surface, it is reflected, diffused, absorbed, or transmitted. Thus, any tooth restoration must match not only the color but also other optical properties that rule such light interactions of the natural teeth [1, 2, 3, 4, 5]. To measure color, illumination patterns, color systems and concepts of numerical color differences (∆E) were developed by the Committee on Illumination (CIE) [6, 7] and recognizes the CIE-LAB color coordinates (L*:sample value; a*: redness-greenness; b*: yellowness-blueness). When using these coordinates alone, other dimensions of color interpretation are neglected; these include: translucency, opalescence, fluorescence, and surface texture [5]. Many studies have then reported methods for calculating these values using the same LAB outcomes from a spectrophotometer, but obtained from different samples put on different backgrounds and then numbers were applied in more advanced calculations; Eqs. (1), (2), (3), and (4) [3, 8, 9, 10, 11].

When esthetic restorations match the shade, combined with the right amount of translucency, opalescence, fluorescence, and surface texture, they can be chosen for anterior teeth. Translucency means the colored material's ability to show through an underlying background, and it lies somewhere between total opacity and complete transparency [12, 13, 14]. Numerical translucency values are represented by the translucency parameter (TP) or contrast ratio (CR). To measure these values, the

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sample is put over a white then over a black background to evaluate the amount of reflected light. TP measures the difference between the two measured values using a spectrophotometer’s CIE L* a* b* color coordinates [4, 15]. CR is the ratio of a material’s light reflectance over a black backing to the same object’s reflectance over a white backing [11, 16].

Scattering short wavelengths of light as it transmits through an object providing a bluish color under reflected light, and a brown-red color under transmitted light is called opalescence. Materials with opalescent properties that truly resemble the natural tooth can be used to create highly esthetic unnoticeable restorations. The opalescence parameter (OP) is the method for quantifying the opalescence of materials [17, 18].

Lithium disilicate glass-ceramics became the choice for monolithic anterior teeth restorations because they are midway between better mechanical and adequate optical properties compared with feldspathic porcelains, (the most esthetic dental material used), and have lower strength but higher translucency than conventional zirconia material. Lithium disilicate is used to make monolithic restorations and further surface characteristics are added to customize opacities and shades [19, 20].

The use of conventional zirconia ceramics had become feasible by the advancements in the technology that made computer-aided design and computer-aided manufacturing (CAD-CAM) technology suitable in clinical settings. These conventional zirconia systems are composed of 3 mol % yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) and have the best mechanical properties. They are also opaque and can mask tooth discolorations. However, this chalky appearance is troublesome when trying to match the natural tooth color. So, conventional zirconia needs to be layered (by a lab technician) using another more esthetic material which further needs firing and glazing steps. This also mandates the removal of more tooth structures to allow space for the second layer of more esthetic materials. This core-layering technique, to shape the final esthetic restoration, suffered chipping problems during clinical service. The weak link was located between the two materials. Monolithic (single bulk) zirconia restorations were developed to overcome the problem. These need no extra lab effort as the bulk of the restorations are milled in the clinical settings. These new materials have good mechanical properties reflecting on the preservation of tooth structure during preparations. However, their ability to achieve optimal esthetics is still challenged [21, 22].

Alterations in the composition and firing recommendations, for CAD-CAM monolithic zirconia ceramic systems, allowed an increase in their translucency. The crystal structure was altered so that residual pores and impurities were minimized during processing, as they -if present-produce volumes of different refractive indices and induce light scattering on the surface, resulting in reduced translucency [22, 23, 24, 25, 26]. The most common impurity reduced or eliminated to increase translucency is alumina, a reinforcing material that also avoids low-temperature degradation (LTD) [27, 28, 29].

Recently, the new generations of CAD-CAM monolithic zirconia ceramic systems had also increased the yttria content to approximately 4 and 5 mol% Y-TZP. These structures have more cubic polycrystals that are voluminous and have a more isotropic cubic phase, which allows light transmission, so better translucent monolithic zirconia restorations [21, 22]. One type of this generation is called multi-layered and has layers of polychromatic and translucent zirconia gradient running from enamel to dentine shades in the single block before milling. Both posterior and anterior restorations are possible using these recent multi-layered zirconia materials. However, there are few studies that look at the complete scope of optical properties of these new zirconia varieties.

A search for PUBMED articles using the terms (monolithic, zirconia, optical) reveals 111 search results (30/7/2021). The bulk of these articles were published between 2019 and 2021. Most are in Q1 journals and backed by serious reviews that reveal the increased interest in this new line of esthetic dental ceramics. The number of new monolithic materials put to the market, to match the esthetic-driven trend, has exceeded the speed by which the studies that verify their optical properties are produced. As their main drive for their use is esthetics (which is also complicated further by tooth and cement optical properties), there is a need to isolate and examine the optical properties derived from the material itself, from other properties affected by the huge variation in the production variables; as the need to fire, temperatures used, polishing recommendations and thickness limitations (etc.). Then later, other contributing clinical factors can be further tested along with their clinical service and durability.

So, this in vitro study aims to study and compare the optical properties of three recently introduced types of CAD-CAM monolithic translucent multi-layered zirconia systems with a conventional lithium disilicate monolithic material (IPS e.max CAD). The null hypothesis to be tested is that there are no optical variations between different multi-layered zirconia materials nor between them and a lithium disilicate CAD-CAM monolithic ceramic system in terms of optical properties.

2. Materials and methods

Four brands of monolithic CAD-CAM ceramic materials: three zirconia systems (presented as discs) were investigated in this study and compared to one lithium disilicate system (presented as glass-ceramic blocks) that acted as a control (Table 1). Zirconia systems were: a polychromatic, super high translucent zirconia Zolid® FX Multilayer (ZF); a new full-contour zirconia brand IPS e.max® ZirCAD MT Multi(ZM), and a super translucent multi-layered zirconia Katana® STML (KS). The control was lithium disilicate glass-ceramics IPS e.max® CAD LT(LC). The materials used in the study were chosen from the same product lot number. Furthermore, the material blocks or discs, used in the tests, were all requested to have the general A2 shade and the samples were collected from the surface designated for the enamel side of the restoration.

Power analysis using G*Power statistical software (G*Power Ver. 3.0. 10, Franz Faul, Universität Kiel) was used to determine the sample size. Samples per group were set considering power: 0.93, or 0.23, and effect size: 0.38. A total of 72 samples were fabricated, each material comprises 18 samples. All samples were taken from the same side in the block to standardize shade differences in multi-layered monolithic zirconia. So, the layer is taken from the enamel shade side of the blocks for all materials.

Square-shaped test samples (N = 72/n = 18) with dimensions of 15.0 mm × 15.0 mm × 1.0 mm were fabricated. In the green stage, samples with the required dimensions for each test were milled from zirconia discs/lithium disilicate block by using a computer-aided manufacturing (CAM) machine (K5+, vhf camfacture AG). Dimensions of the zirconia samples were determined to taking into consideration the shrinkage that occurs during sintering.

All samples were then polished gradually before sintering using 600, 800 and 1000 grit silicon carbide(SiC) papers in a polishing & grinding device (echo LAB POLI-IX/250) with water for lithium disilicate samples, and without water for zirconia samples as recommended by the manufacturers, to the final dimensions. Each polishing step was carried out for 60 s at 300 rpm, by one single operator. After that, the samples were fully sintered/crystallized according to the manufacturers’ parameters (Table 2). Then an additional polish was done after sintering with polishing paste (S100000, all-in-one, Renfert). The dimensions of all samples were verified individually using a digital caliper (Insize 1111-75A) which has a 0.01 mm accuracy. After polishing, all samples were immersed in alcohol that was activated as an ultrasonic bath of for 5 min before testing.

A dental spectrophotometer (VITA Easyshade compact, Vita Zahnfabrik) was used to record the CIELAB coordinates (L*, a* and b*) of the ceramic samples against black (Figure 1) and white (Figure 2) backgrounds. L represents the sample’s value, a* redness to greenness and b* yellowness to blueness. A better optical contact was achieved by applying a thin layer of petroleum jelly in-between the sample and the background for [30].
The precision of the test spectrophotometer was evaluated in terms of the consistency of its repeated measurements during the calibration protocol. Environmental variables were minimized by many ways. Measurement values were obtained by a single operator. All samples were viewed under noon’s daylight, between 12.00-1.00 pm. The room also had the same fluorescent lighting.

Using the equations in Table 3, the following optical property parameters were calculated.

1. Translucency parameter (TP); Equation (1)
2. Contrast ratio (CR); Equations (2, 3)
3. Opalescence parameter(OP); Equation (4)

2.1. Statistical analysis

Descriptive statistics were computed for each property within each material. Statistical analysis was done using a statistical software (SPSS Statistics v25.0; Chicago, USA). The Kolmogorov-Smirnov test Normalities showed that groups were found to be distributed normally. Data were analyzed with one-way ANOVA, with the Bonferroni correction to adjust for multiple comparisons. When p was less than 0.05, then differences were considered statistically significant.

3. Results

TP values ranged from 14.174 to 20.439 (Table 4). The analysis revealed that no statistical differences in TP were found between the zirconia materials (p = 1.000). LC, the control group, revealed the highest TP and lowest CR compared with the other monolithic multi-layered zirconia (p < 0.05). On the other hand, CR values ranged from 0.592 to 0.710. The data were ranked as follows: LC > ZF > KS > ZM. No statistical differences in CR between the zirconia materials were observed (P = 1.000). For all measured samples, there was a clear association between TP and CR parameters, with the TP decreasing as the CR increased.

The comparisons showed that the opalescence of tested materials varied greatly (Table 4) except for ZM (8.3 ± 1.5) and KS (8.2 ± 0.9), which had no statistical differences between them (P = 1.000). ZF had the lowest opalescence (5.06 ± 0.3), while LC was significantly more opalescent (10.09 ± 0.4) than all zirconia groups (p < 0.05).

4. Discussion

The optical properties of three commercially available CAD-CAM monolithic transparent multi-layered zirconia ceramics were analyzed and compared to lithium disilicate glass-ceramics as a control in this analysis. The results of this study rejected the null hypothesis regarding the optical properties as they revealed statistically significant differences amongst the tested materials.

As a higher is the TP value indicates that translucency is higher, the variations in the translucency arising from the various types of ceramic materials [35] were confirmed in this research. It showed that LC had higher translucency than other monolithic translucent multi-layered zirconia systems, consistent with previous studies [35, 36]. For LC, Nassary Zadeh et al [36] reported a higher value (40.4 ± 0.4), in contrast, Della Bonia et al [31] reported a lower value (17.35 ± 0.81) than the present study. Furthermore, there were no statistical differences determined amongst all tested zirconia systems regarding TP studied here.

Esthetically pleasing restorations should have their TP values equal to that of natural enamel. But few experiments compared the translucency of enamel with restorative material since it is difficult to prepare standard enamel samples [37]. Furthermore, the translucency of enamel varies by gender, age, and the color of teeth. Studies on human teeth concluded the TP value to be 18.7 for enamel, and for dentin it was 16.4 at 1.0 mm thickness [38]. To visually perceive a translucency difference, ΔTP between restorative material and enamel should be more than 2. If this value was applied here, TP of monolithic material has an adequate match to that of human dentin with its low translucency. If the visual perceptibility was combined with clinical thickness recommendations for the materials, then the optical properties of monolithic material differences at 1 mm thickness (Which is actually the clinical thickness recommended) will approach those values for lithium disilicate glass-ceramics at 1.5 and 2.0 mm clinical thickness.

Just a few experiments have been conducted to determine the TP values of recently introduced monolithic CAD-CAM restorative materials [30, 39, 40]. In a recent review, Nassary Zadeh et al. [36] compared the TP values of different CAD-CAM products and discovered a major difference between lithium disilicate glass-ceramics and cubic/tetragonal zirconia. The mean TP value of lithium disilicate glass-ceramics was found to be higher than that of zirconia ceramics. This variation in translucency was clarified by the researchers to be the result of increasing

| Table 2. Sintering/Crystallization parameters used for tested materials. |
|-----------------------------|------------------|------------------|------------------|------------------|
| Material | Heating Rate and Eventual Heating Steps | Final Temperature (°C) | Holding Time | Cooling Rate up | Furnace brand name |
| ZF | 8° C/min | 1450 °C | 120 (min) | 10 h | CeramillTherm, AmannGirrbach |
| ZM | 10° C/min until 900 °C is attained, after holding for 30 min, heating rate of 3 C/min until 1500 °C | 1500 °C | 120 (min) | -10° C/min from 1500-900 °C, then -8° C/min from 900-300 °C | inFire HTC speed, Dentsply Sirona |
| KS | 10° C/min | 1550 °C | 120 (min) | -10° C/min | CeramillTherm, AmannGirrbach |
| LC | 1: 90° C/min until 820 °C is attained, holding for 10 min, 2: 30° C/min until 840 °C is attained, holding for 7 min. | 1540 °C | 120 (min) | 0° C/min | Programat P5010 (Ivoclar-Vivadent) |

| Table 1. Materials used in this study. |
|-----------------------------|------------------|------------------|------------------|
| Material/Shade | Composition | Manufacturer Code |
| Zolidfx Multilayer; A2/A3 | ZrO2 + HfO2 + Y2O3; Y2O3:8.5-9.5%; HfO2:≤5%; Al2O3:≤0.5%; Other oxides:≤1% | AmannGirrbach AG ZF |
| IPS e.maxZirCAD MT Multi; A2 | ZrO2:86.0-93.5%; Y2O3:6.5-8.0%; HfO2:≤5%; Al2O3:≤1.0%; Other oxides:≤1.0%; | IvoclarVivadent, Schaan ZM |
| Katana STML; A2 | ZrO2:88-93%; Y2O3:7-10%; Other oxides:≤2% | Kuraray noritake KS |
| Lithium disilicate | SiO258-80%; Al2O30-11%; K2O0-13%; Li2O1-19%; ZrO2:0-4%; Al2O3:0-5%; | IvoclarVivadent, Schaan LC |

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the volume of stabilizing oxides. Furthermore, as the thickness of the monolithic ceramics reduced, the translucency increased exponentially [4]. A review studied porcelain laminate veneers in terms of their spectral transmission. These were measured at three thicknesses and three opacity groups. The reviewed studies found that the thickness of the veneer was the main factor affecting light transmission, but not the material’s opacity [41]. The clinical inference of such results supported the use of dual-cured cements, with less light requirements, under restorations; more than 1.5 mm in light-shaded zirconia and 0.5 mm in darker-shaded zirconia.

The translucency of samples studied here was examined for at the same thicknesses (1.0 ± 0.05 mm) for standardization purposes. However, in order to survive mechanical tension in the oral cavity, lithium disilicate restorations are required to have an additional thickness occlusally (1.5 mm–2.0 mm). While monolithic translucent zirconia can be used to create a monolithic tooth restoration with a thinner occlusal layer, even less than the studied samples (1 mm), and hence require less tooth reduction [37, 42, 43]. As a result, transparent multi-layered zirconia with a reduced thickness could be proposed to have comparable translucency values to restorations that have increased lithium disilicate thickness that follow clinical recommendations.

A more recent study had approached the clinical thickness issue by studying thicker samples (1 mm, 2 mm, and 4 mm samples) for the exact lithium disilicate material used in our study and reported TP values as follows: [9.74 (0.1) at 1 mm, 1.93 (0.03) at 2 mm and 0.58 (0.10) at 4 mm thickness] [44]. Thus, it indirectly infers that the optical properties’ difference could fade when the comparison is made between the recommended clinical thickness of monolithic zirconia material to the

Figure 1. Sample tested against black backgrounds.

Figure 2. Sample tested against white backgrounds.
Contrast ratio (CR) [5, 30, 31]

Bona et al [31] found a CR value of 0.62. In contrast, Elaska et al [47] found a CR value of 0.56 for ZF, lower than that reported in this study.

The current study’s CR value for KS is close to those stated by Baldissara et al [39, 45]. A negative association between TP and CR was discovered: as TP declines, CR rises. This correlation was present in this study in conformity with previous studies [5, 10].

Antonson and Anusavice reported the CR value of feldspathic porcelain at 1.0 mm thickness in the range of 0.60–0.78, which is in accordance with this study’s finding for LC, indicating improved optical properties of new monolithic zirconia [46]. The current study’s CR values for LC were significantly smaller than those found in another in vitro test. Bona et al [31] found a CR value of 0.62. In contrast, Elaska et al [47] found a CR value of 0.56 for ZF, lower than that reported in this study. The current study’s CR value for KS is close to those stated by Baldissara et al (0.79) [48].

The visual translucency perception threshold was not confirmed with TP values. However, CR values of 0.06 were found by Liu et al as the translucency perception threshold [45]. It is obvious that the value difference between zirconia material is less than 0.06, such a difference between them was clinically undetectable. In contrast, the CR values difference between LC and zirconia materials is being more than 0.06, so it is clinically perceivable.

Only one source cited the CR values for human enamel (0.55) and dentin (0.6), but when the reference was revisited, the values were not present and if they were calculated, calculations were not reported [49].

The use of various measurement instruments and polishing methods is most likely to account for the variations in the values between the tests. Monolithic zirconia material had different chemical compositions with different microstructure and particle sizes. Added to that are the different polishing & finishing protocols used on the studies. All could have affected the TP and CR properties [50, 51].

To produce a dental restoration with improved color depth and a natural appearance, materials with opalescent properties should be used. OP values were measured using a dental spectrophotometer [34, 52]. In the current analysis, ZF had the lowest values, while LC had the highest opalescence, and there were no major variations with the other tested zirconia ceramic systems. The variance was suggested to be related to variations in the elements and microstructure of these materials.

The opalescence of human enamel differs depending on the configuration of the measuring spectrophotometer. The mean OP value for human enamel reported by Lee et al [52] was 22.9 ± 1.9 at a thickness range of 0.9–1.3 mm. This indicates that the OP of the measured products in this sample, which ranged from 5.06 to 10.09 at 1.0 mm thickness, was less than that of human enamel opalescence. Bona et al [31] compared the OP values of various CAD-CAM materials of different shades at a thickness of 1.0 mm and stated that IPS e. max CAD LT was less than that observed in this analysis among the materials with the highest OP values (6.96). In contrast, Shiraishi et al [53] reported OP values to range from 5.27 to 12 11 for 1.0 mm thick porcelain, which is a little higher than those found in the current study (5.06–10.09). This variation is attributed to the variance in the tested ceramic system, shade, and thickness used. The higher the chromatic shade, the higher the oxides such as ZrO2, Y2O3, SnO2, and V2O5 which influences OP values.

Looking more into the differences and effect of chemical composition on materials’ optical properties is needed. The improvements in the translucency of recent zirconia was done by changing the yttria content, the amount of chemical impurities, and using various grain sizes, but further research is essential. By decreasing the alumina and increasing the yttria content, the latest monolithic zirconia brands manufacturers enhanced the translucency of 3Y-TZP [23, 24, 28]. The yttria content of the KS is up to 10%, and that of ZF is up to 9.5% while the yttria content of the ZM is up to 8%, less than the others. According to mol%, ZF and KS monolithic zirconia are considered to be 4Y-TZP, while ZM is considered to be 5Y-TZP. Using a higher yttria content led to a greater content of the cubic phase (approximately 50%) of zirconia, which enhances the translucency property but reduces the mechanical properties [22, 54]. The alumina content of material was different. ZF contained a lower amount (<0.5%) compared with ZM (<1%) monolithic zirconia.

The sintering temperature also affects translucency. Various manufacturers’ recommendations were followed for each material; i.e. heating rate, holding time, and cooling (Table 2). Therefore, the sintering parameters are suspected to have an impact on translucency. The higher sintering temperatures, in the range 1510°C–1550°C closes residual pores at the grain boundaries level, thus increasing the density of the material, and decreasing the refractive index and light scattering [54, 55].

One of the limitations of the present analysis is that, for ease of use and standardization, only a single polishing and finishing protocol was used for all of the tested ceramic systems.
Future in vitro studies may further use various polishing materials and methods, as well as different surface treatments to test their effect on optical properties of these materials. Furthermore, the influence of various material thicknesses, acidic conditions, sintering temperatures, and aging has not yet been tested and should be examined.

Since the samples were not exposed to saliva or thermal fluctuations, thus this in vitro study could not fully replicate clinical conditions. Further studies are needed using simulated media to match the intraoral environment variables to make more definitive clinical recommendations and confirm the clinical applications and limitations of these new restoration systems.

5. Conclusions

Based on the results presented and within the limitations of this in vitro study, the following conclusions can be stated:

1. The optical properties were affected by the type of monolithic CAD-CAM restorative material.
   a. The optical properties (TP, CR, and OP) of lithium disilicate ceramic are superior to those of CAD-CAM translucent multi-layered zirconia ceramic systems at 1.00 mm thickness. But, there is a need to further study different multi-layered zirconia systems at their clinically recommended thicknesses to compensate their difference with human enamel values and compare them to the values of recommended thicker lithium disilicate.
   b. This study’s findings indicate improved translucency and other optical properties of tested monolithic multi-layered zirconia materials in comparison with conventional zirconia-based ceramics but still further matching is required since its translucency limits its masking and color blending abilities with adjacent enamel and underlining dentin of the abutment.

2. More in vivo studies are required to validate if CAD-CAM translucent multi-layered zirconia ceramic systems are reliable restorative material for anterior restorations, matching the light transmission, light-curing efficiency, and the effect of final shade of the underlying resin-based luting agents on the final esthetic result. Then followed up longitudinally to test their clinical service and durability.

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