ORIGINAL ARTICLE

INFLUENCE OF VARYING SINTERING PROCEDURES ON THE FRACTURE RESISTANCE AND TRANSLUCENCY OF MONOLITHIC ZIRCONIA DENTAL CROWNS

Utkarsh Shishodia[1], Chirag Dayal[2], Rohit Kumar[3], Ravindra Negi[4]

ABSTRACT

The aim of the study was to examine the impact of various sintering methods (long, fast, and high-speed cycles) on the translucency and fracture resistance of monolithic zirconia crowns.

Materials and Methods: *Thirty monolithic zirconia crowns were manufactured through a CAD/CAM process using translucent NexxZr zirconia blanks. The crowns were divided into three groups of ten each based on the sintering conditions (temperature and duration). Group I underwent a long sintering cycle of 1510°C for 120 minutes with an overall cycle time of 8 hours. Group II underwent a fast-sintering process at 1540°C for 25 minutes with a total cycle time of 2 hours. Group III underwent high-speed sintering at 1580°C for just 10 minutes, which was the entire firing cycle. The translucency parameter **(TP)** and contrast ratio **(CR)** were assessed using a spectrophotometer, and the crowns were subjected to compressive load until fracture using a universal testing machine. One disc sample (10mm diameter x 1mm thickness) was created for each group and analyzed using a scanning electron microscope. The data was then statistically analyzed.*

Results: *The results showed that the speed of sintering had a significant impact on the means of translucency **(TP),** contrast ratio **(CR),** and fracture resistance. The long sintering cycle resulted in the highest mean values for translucency **(TP)** and the lowest mean values for contrast ratio **(CR).** The high-speed sintering cycle showed the highest mean values for fracture resistance.*

Conclusion: *Shortening the sintering cycle significantly reduced the translucency and increased the fracture resistance of monolithic zirconia crowns. Based on the threshold for perception of translucency, the speed sintering cycle can be recommended for the sintering of **NexxZr** monolithic zirconia crowns.*

1. MDS Prosthodontics & Implantology, Fellow WCOI Japan).
2. General Manager Lab Services
3. CAD/CAM Technician (Yarrow Dental Labs)
4. CAD/CAM Technician (Yarrow Dental Labs).

Corresponding Author
Dr. Utkarsh Shishodia
(MDS Prosthodontics & Implantology
Fellow WCOI Japan), Technical Head
(Yarrow Dental Labs)

INTRODUCTION

Zirconia has become a popular ceramic material due to its superior mechanical properties, which are not found in any other ceramic system, and its superior appearance compared to metal-ceramics. This is due to the transformation toughening mechanism of zirconia.[1,2]

Zirconia has several advantageous properties such as reduced plaque buildup, excellent biocompatibility, low corrosion, and low thermal conductivity.[3,4] As a result, zirconia can be used in the fabrication of various fixed prostheses such as crowns, fixed partial dentures, implant fixtures, and abutments.[5] However, low translucency and hydrothermal instability are considered challenges when using zirconia-based restorations.[6] The high opacity of zirconia, resulting from decreased light transmission and increased scattering through the restoration, requires zirconia cores to be covered with porcelain to imitate the appearance of natural teeth.

The most common clinical failures in these veneered restorations are cracking or chipping of the veneering porcelain. Setting a strong mechanical or chemical bond between zirconia and veneering porcelain has proven to be challenging. Additionally, these veneered restorations have low fracture strength.[7,8]

However, monolithic zirconia restorations also have some limitations, including their low translucency and discoloration over time due to the accumulation of stains and changes in the surface structure. To address these issues, various sintering protocols have been developed to improve the translucency and resistance of monolithic zirconia restorations. The aim of the current study was to investigate the effect of different sintering protocols on the translucency and fracture resistance of monolithic zirconia crowns.

Furthermore, the use of solid zirconia has gained widespread popularity due to its ability to reduce the need for tooth preparation and the thickness of the restorative material when compared to veneered zirconia restorations. Additionally, minimal wear is seen on opposing enamel in contact with polished solid zirconia.

In addition, research has shown that monolithic zirconia crowns have a higher fracture strength compared to veneered zirconia crowns[8,13-15]. However, other studies have found that monolithic and veneered zirconia have similar strength[16]. Based on these findings, it can be concluded that monolithic zirconia can be a viable alternative to veneered zirconia.

One of the biggest challenges with monolithic zirconia restorations is achieving an acceptable esthetic appearance due to their single-layered structure. Zirconia can be categorized based on its translucency into opaque and translucent varieties. The opaque type of zirconia is stronger and is typically used for posterior restorations, while the translucent type has a more natural appearance due to its greater translucency, making it suitable for use in both anterior and posterior restorations. Efforts have been made to enhance the translucency of monolithic zirconia through modifications in fabrication techniques, coloring techniques, and the sintering process, with the aim of improving its properties[14].

The sintering process is a crucial stage in the fabrication of zirconia restorations and can be modified to optimize its properties. The use of CAD/CAM technology has significantly reduced clinical operating times, but the sintering process still requires a significant amount of time. Changes in sintering parameters have been shown to have a significant impact in dental research, particularly with the introduction of short sintering cycles by manufacturers. Rapid sintering can now be achieved in just a few minutes, enabling the creation of zirconia restorations in a single visit, increasing its practical clinical use[17,18].

Some researchers have found that increasing the sintering temperature and duration leads to larger tetragonal zirconia grain sizes, which can increase translucency[18-20]. However, large grain sizes can result in a spontaneous tetragonal to monoclinic transformation (T-M), causing a decrease in material stability and gradually lowering the strength of the material[21]. Lowering the temperature and/or duration reduces the grain size, which prevents T-M transformation, resulting in maximum strength for small grain sizes[22].

Many studies have investigated the impact of variations in sintering temperature and duration on the grain size, translucency, and flexural strength of zirconia [7,10,18,20], but the effect of these changes on the fracture resistance of zirconia crowns remains uncertain. The aim of this study was to examine the impact of different sintering protocols (long, speed, and high-speed cycles) on the translucency and fracture resistance of monolithic translucent zirconia crowns. The null hypothesis was that the different sintering protocols would not affect the translucency and fracture resistance of zirconia crowns.

In this in-vitro study, a total of 30 monolithic zirconia crowns were fabricated using CAD/CAM technology. The crowns were made from translucent zirconia blanks (NexxZr™ Ivoclar, Schaan, Liechtenstein) and were designed to fit a typodont model of a maxillary right second premolar tooth. The tooth preparation followed standard procedures with a deep chamfer finish line, occlusal reduction, axial reduction, and a 6-degree angle of convergence. The prepared tooth was scanned and the crowns were designed, milled, and then sintered according to the specific protocol of each group.

This research study aimed to evaluate the effects of different sintering protocols on the mechanical properties of CAD/CAM fabricated monolithic zirconia crowns. The crowns were divided into three groups based on their sintering conditions: long sintering, speed sintering, and high-speed sintering. The researchers used a spectrophotometer to measure the translucency **(TP)** and color rendering **(CR)** of the crowns. The crowns were then cemented onto epoxy resin dies and subjected to compressive load until fracture in a universal testing machine. Additionally, a representative disc sample from each group was analyzed using a scanning electron microscope to evaluate the microstructure of the sintered zirconia. The results of this study will likely provide insights into the optimal sintering conditions for monolithic zirconia crowns to achieve the desired mechanical properties, as well as the effects of sintering on the crown's TP and CR.

This study describes the fabrication process of monolithic zirconia crowns. The first step involved digital scanning of the prepared second premolar tooth using a 3D dental scanner (Identical hybrid blue scanner). The resulting scan was then converted to STL format and sent to the laboratory. The crowns were designed using CAD software (Exocad Dental CAD) where the minimum material thickness was set to 1mm and the spacer thickness was set to 40 ìm. The design data was then transferred to the computer connected milling machine to begin milling the full-contour monolithic zirconia crowns, following the manufacturer's instructions. The fabrication process of monolithic zirconia crowns involves several steps, starting with digital scanning of the prepared tooth and ending with milling the crown from a solid block of zirconia. The precise and accurate control over the design and milling process is critical for ensuring that the final product meets the desired specifications and clinical requirements.

The milling of the monolithic zirconia crowns was performed using translucent zirconia blanks of shade A2 (NexxZr shaded blanks). The zirconia blanks were composed of zirconium oxide (ZrO2), yttrium oxide (Y2O3), hafnium oxide (HfO2), and aluminum oxide (Al2O3). The zirconia blanks were inserted into a 5-axis milling machine (PrograMill PM7, Ivoclar) where the crowns were milled with a 20% enlargement to compensate for the sintering shrinkage. After milling, a specific NexxZr finishing bur was used to separate the crowns from the blank. The crowns were then cleaned ultrasonically in distilled water for 10 minutes and dried in a drying unit for 5 minutes at a temperature of 80°C. These steps are important to ensure that the crowns are free from any residual debris or moisture, which can affect the sintering process and final properties of the crowns.

SINTERING OF THE CROWNS:

After the milling process was completed, the crowns were inserted into a high-temperature sintering furnace (Programat S1, Ivoclar) for sintering. The sintering process was carried out at different temperatures and speeds, as specified in the study:

GROUP (I)

- Long sintering (sintering at 1510°C for 120 min holding time and 8 hours total cycle time).

GROUP (II)

- Speed sintering (sintering at 1540°C for 25 min holding time and 2 hours total cycle time).

GROUP (III)

- High-speed sintering (sintering at 1580°C for 10 min holding time representing the total firing cycle).

The sintering process is a crucial step in the fabrication of monolithic zirconia crowns, as it determines the final physical and mechanical properties of the crowns. The sintering temperature and speed play an important role in determining the translucency, color stability, and strength of the crowns.

FABRICATION OF ZIRCONIA DISCS:

A. Designing of the disc-

The design of the zirconia disc involved creating a 2D model of a disc with 12 mm diameter and 5 mm thickness using the CAD software (Exocad Dental CAD, v.2016, GmbH, Darmstadt,Germany). The CAD software was used to create a digital representation of the disc that could be used as a template for the milling process. The design of the disc was done according to the specifications provided, and it is important to note that the dimensions and thickness of the disc play a crucial role in the final outcome of the sintering process and the mechanical properties of the disc.

B. Milling& sintering of the discs-

A disc with dimensions of 12mm in diameter and 5mm in thickness was created using a milling machine as per the manufacturer's instructions. The disc was then cut into slices of 1.2mm thickness with a diameter of 12mm using a water-cooled saw that was 20% larger than the final desired size to account for shrinkage during the sintering process. The slices were sintered, similar to the crown samples of each group, to produce discs with a final size of 10mm in diameter and 1mm in thickness. The thickness of each disc was measured using a digital caliper.

TESTING PROCEDURES:

TRANSLUCENCY MEASUREMENTS

- *Translucency Parameter (TP)-*

The level of translucency was evaluated with the Vita Easyshade spectrophotometer, manufactured by Vita, in Schann, Liechtenstein. To measure the L*, a*, and b* values of each crown, a custom holding device was used to tightly secure it onto the prepared tooth. The probe tip of the Vita Easyshade was placed on the central portion of the crown's buccal surface for measurement.

In order to ensure the accuracy and uniformity of the measurements, the probe tip was positioned in the same location on every crown. The color measurements were taken three times for each crown by the same examiner to maintain consistency. The crowns were tested against both white and black backgrounds, and the mean L*, a*, and b* values were recorded. The device was calibrated prior to each measurement to ensure standardization.

The TP (transparency parameter) values were determined using the following formula:

TP = [(Lb* - Lw*)^2 + (ab* - aw*)^2 + (bb* - bw*)^2]^1/2

Here, "b" and "w" represent the color coordinates against black and white backgrounds, respectively. The scale ranges from 0 (completely opaque) to 100 (completely transparent). A higher TP value indicates greater translucency of the material[24]

- *Contrast Ratio (CR)*

  The contrast ratio (CR) for each crown was calculated according to the following equations:

  The L* values were used to calculate the spectral reflectance (Y) as follows:

  Y= [(L*+16)/116]3 x100. Where, the values recorded on white (Yw) and black (Yb) backgrounds were used to calculate the (CR) as in this equation:

  CR= Yb/Yw. CR values range from 0 (transparent material) to 1 (completely opaque material)

FRACTURE RESISTANCE TESTING

The prepared tooth was replicated into thirty epoxy resin dies. The monolithic zirconia crowns were affixed to their corresponding epoxy dies using self-adhesive resin cement (Relyx U-200, 3M) under a consistent axial load of 5kg through a specially designed cementing apparatus. Subsequently, the resin cement was cured with light from all angles of each crown, and the crowns were stored in an environment with 100% humidity at 37°C for 24 hours. Following this, all zirconia crowns were placed individually on a computer-controlled universal load testing machine, with a load cell of 5 kN. A compressive load was applied occlusally at a cross-head speed of 1mm/min, and the failure loads were measured and recorded in Newtons (N). Please see Figure 1.

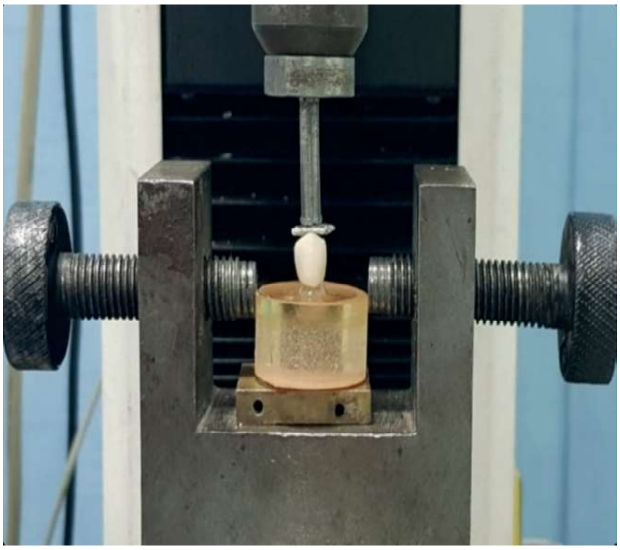

Fig. 1: A universal testing machine

ENVIRONMENTAL SCANNING ELECTRON MICROSCOPIC (SEM) ANALYSIS:

Following the sintering process of the disc samples, they underwent ultrasonic cleaning for 10 minutes using distilled water and were then dried with oil-free compressed air. The discs (as sintered) were analyzed using an Environmental Scanning Electron Microscope. The disc samples were inspected at magnifications of 3000x, 30000x, and 60000x, with an accelerating voltage of 30 K.V.

STATISTICAL ANALYSIS:

The impact of sintering speed on the mean values of TP, CR, and fracture resistance was evaluated using a one-way ANOVA test. Pearson's correlation coefficient was used to investigate the relationship between TP and CR. The significance level was set at P d" 0.05. Statistical analysis was conducted using IBM® SPSS® Statistics Version 20 for Windows.

RESULTS:

The collected data was examined for normality using both the Kolmogorov-Smirnov and Shapiro-Wilk tests, which indicated that the data had a parametric (normal) distribution.

TRANSLUCENCY RESULTS:

- *Translucency Parameter (TP)-*

  The results of the one-way ANOVA indicated that the sintering speed had a significant impact on the mean value of TP. Specifically, the long cycle exhibited the highest mean value of TP, which was statistically significant. There was also a significant difference observed between the long, speed, and high-speed cycles, with the high-speed cycle displaying the lowest mean values of TP. These findings are summarized in **Table 1.**

Sintering cycle	Translucency	
	Mean	SD
Long cycle	13.263 [a]	0.616
Speed cycle	11.603 [b]	0.300
High-speed cycle	10.040 [c]	0.217
p-value	**<0.001***	

Different letters indicate statistically significance difference. *; significant at $p \leq 0.05$

Table 1

- *Contrast Ratio (CR)-*

The results of the one-way ANOVA indicated that the sintering speed had a statistically significant effect on the mean value of CR. The high-speed cycle was found to have the highest mean CR values, and this difference was found to be statistically significant. Furthermore, there was a significant difference between the long, speed, and high-speed cycles, with the long cycle displaying the lowest mean CR values. **Table 2** summarizes these findings. **(Table 2)**

Sintering cycle	Contrast ratio	
	Mean	SD
Long cycle	0.663 c	0.012
Speed cycle	0.707 b	0.025
High-speed cycle	0.770 a	0.010
p-value	0.001*	

Different letters indicate statistically significance difference.; significant at $p \leq 0.05$*

Table 2

CORRELATION BETWEEN TP AND CR:

The results of the Pearson correlation analysis showed that there was a statistically significant negative correlation between TP and CR, with a correlation coefficient of -0.877 and a P-value of 0.002.

FRACTURE RESISTANCE RESULTS:

The results of the one-way ANOVA demonstrated that the sintering speed had a significant effect on the mean value of fracture resistance. Specifically, there was a significant difference observed between the three sintering cycles, with the high-speed cycle exhibiting the highest mean values and the long cycle displaying the lowest mean values. These differences were found to be statistically significant. A summary of these findings can be seen in **Table 3.**

Based on the SEM images and analysis, it appears that the sintering speed has an effect on the microstructure of zirconia. The sample with a longer sintering cycle showed larger and irregularly shaped grains, with a size ranging from 400-800 nm. These larger grains may have formed due to extended sintering times, allowing for grain growth and coarsening. Additionally, the presence of increased porosity in this sample suggests that it may not have sintered completely, which could be a result of the slower sintering rate.

Sintering cycle	Fracture resistance	
	Mean	SD
Long cycle	1407.040 c	13.231
Speed cycle	1676.227 b	21.510
High-speed cycle	1988.897 a	10.729
p-value	<0.001*	

Different letters indicate statistically significance difference. *; significant at $p \leq 0.05$

Table 3

It's important to note that SEM images can provide valuable insight into the microstructure of materials, which can help in understanding their properties and performance. In this case, the analysis of the images indicates that the sintering speed has a significant impact on the microstructure of zirconia, which may in turn affect its properties and potential applications.

In **Figure 2B**, the speed cycle sample exhibited grains that were slightly smaller than those found in the long cycle sample i.e **Figure 2A**. The grains in the speed cycle sample varied from 200-500 nm and had a more even distribution of grain sizes. They were irregularly angular in shape and had less porosity than the long cycle sample.

In contrast, the high-speed cycle sample in **Figure 2C,** had the smallest grain size, ranging from 100-300 nm. The small grains were dominant in certain areas and had a relatively regular rounded shape. The sample also had increased grain boundaries, which were more densely packed and had the least amount of porosity compared to the other samples.

DISCUSSION

The emergence of monolithic translucent zirconia as a promising option for dental crowns is due to its favorable optical and mechanical properties, as indicated by previous research[13,25]. In the current study, "NexxZr" crowns were chosen for evaluation as they have demonstrated high fracture strength and cause minimal wear to the opposing teeth when compared to other monolithic translucent zirconia crowns[8].

The purpose of this study was to address the need for a more efficient and cost-effective method of producing chair side CAD/CAM prostheses in a single visit. To achieve this goal, the study aimed to reduce the duration

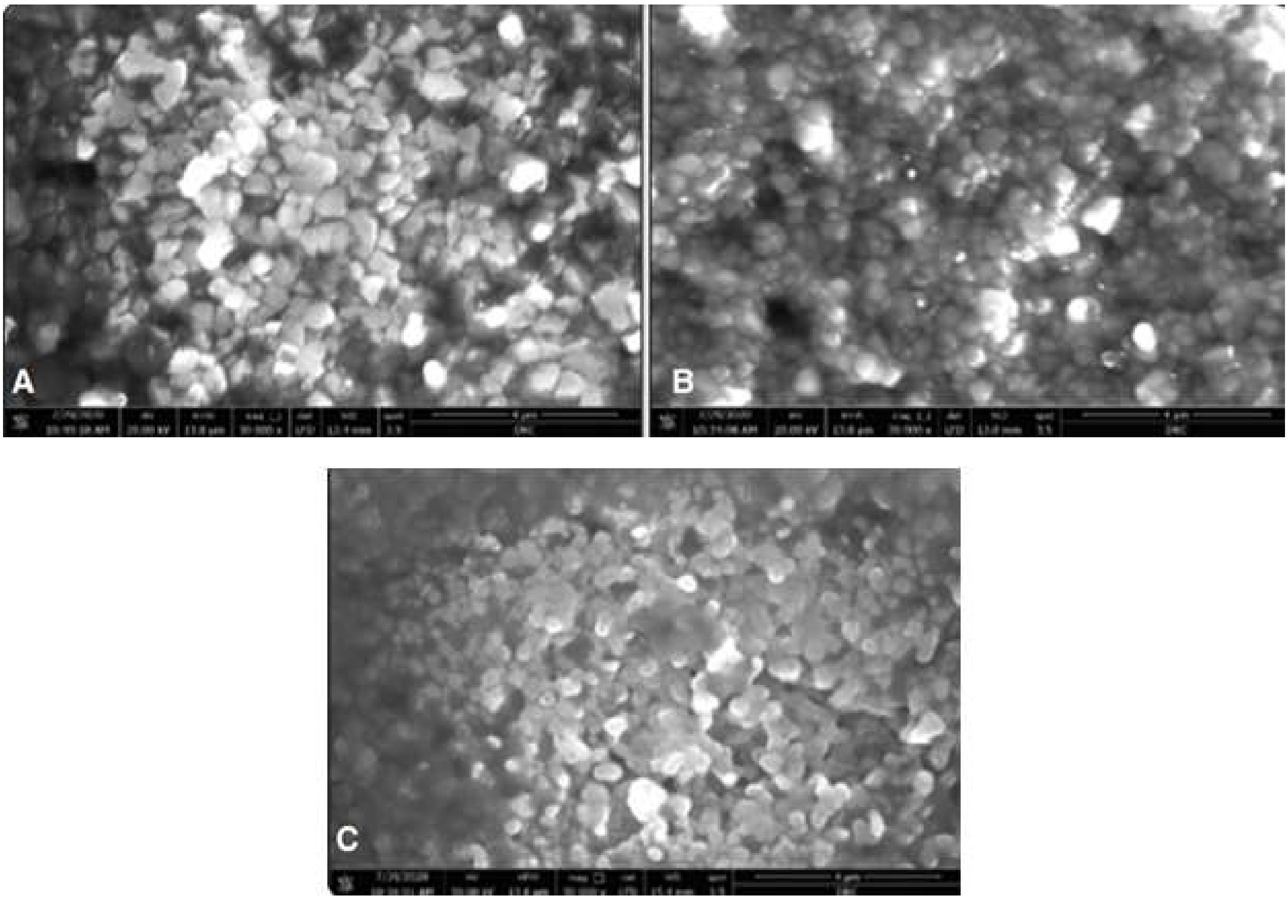

Fig. 2- A, B, C

of the sintering cycle, building on previous research that had investigated the effects of altering sintering parameters on the optical and mechanical properties of monolithic zirconia, as well as its clinical serviceability as fixed prostheses[7,18]. By shortening the sintering cycle duration, the study aimed to improve the overall efficiency of the chair side CAD/CAM prosthesis production process.

When it comes to esthetic ceramic prostheses, it is important that they match both the color and translucency of natural teeth. Translucency is the most important factor that influences the esthetic properties of ceramic restorations. There are different methods for evaluating the translucency and opacity of esthetic materials, including absolute translucency (direct light transmittance), as well as relative translucency measured by either **(TP)** or **(CR).**

In this study, both **(TP)** and **(CR)** were used to evaluate the translucency of monolithic zirconia crowns. A restoration is considered absolutely opaque if the **(TP)** is equal to zero and absolutely transparent if the **(TP)** is equal to “100”[24]. On the other hand, the **(CR)** value is “0” for a transparent material and “1” for a completely opaque material[26,27].

The results of this study were in agreement with previous research by **Della Bona et al.**[28], who found a statistical correlation between **(TP)** and **(CR)** results ($r = -0.877$, P-value = 0.002).

Previous research studies evaluating the translucency of ceramic materials have primarily used flat disc samples of standardized thickness. However, in this current study, monolithic full-anatomical zirconia crowns were selected instead of disc samples for measuring translucency in order to be more clinically relevant. This approach was chosen to better simulate the clinical situation where a monolithic zirconia crown is used to replace a natural tooth. By using full-anatomical zirconia crowns instead of disc samples, the study aimed to provide more accurate and clinically relevant translucency data.

The approach of measuring color and translucency through the buccal surface of various types of ceramic crowns, which was used in this study, is in accordance with some previous research studies[13,29-31]. This method is more clinically relevant as it simulates the way in which clinicians would evaluate the esthetics of ceramic restorations in a patient’s mouth.

In addition, using a spectrophotometer to analyze color has several benefits. The most important advantage is that it allows color to be handled as a numerical variable, which enables accurate comparison of different parameters without the interference of the effect of the surrounding light source or reflections[32]. By using a spectrophotometer, the study aimed to obtain objective and accurate color data for the monolithic zirconia crowns.

Fracture resistance is an important mechanical property of dental restorative materials, as it determines the material's ability to withstand mechanical stresses and prevent fractures under clinical conditions. Previous studies evaluating the influence of altering sintering protocols on the mechanical properties of zirconia have primarily focused on measuring flexural strength of flat samples[32-34].

In this study, fracture resistance of cemented translucent monolithic full anatomical zirconia crowns was evaluated to determine the influence of changing sintering parameters on their mechanical properties. This approach was chosen to ensure the reliability and suitability of the restorations on a clinical basis. Furthermore, zirconia crowns were cemented with resin cements, as studies have reported higher fracture strength in ceramic restorations cemented with adhesive resin cements[35]. The use of resin cement in cementing the zirconia crowns aimed to ensure adequate bonding and optimal fracture resistance of the restorations.

In conclusion, this study found that altering the sintering parameters of monolithic translucent zirconia crowns significantly affects both their translucency and fracture resistance. This suggests that careful consideration of sintering protocols is necessary in order to achieve optimal esthetic and mechanical outcomes in zirconia restorations. Additionally, SEM analysis was used to investigate the relationship between microstructure and translucency, highlighting the importance of understanding the underlying material properties that contribute to the overall performance of dental restorations.

CONCLUSION

These are the main conclusions of the study on the effect of different sintering protocols on the translucency and fracture resistance of monolithic zirconia crowns. The study found that shortening the sintering cycle had a significant impact on the translucency and fracture resistance of the crowns. Specifically, the crowns sintered using the speed cycle showed the highest fracture resistance, but also the lowest translucency. Based on the Translucency Perception Threshold, the study suggests that the speed cycle could be recommended for sintering "NexxZr" monolithic zirconia crowns. However, it is important to note that this recommendation may not necessarily apply to other types of monolithic zirconia crowns or other clinical situations.

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