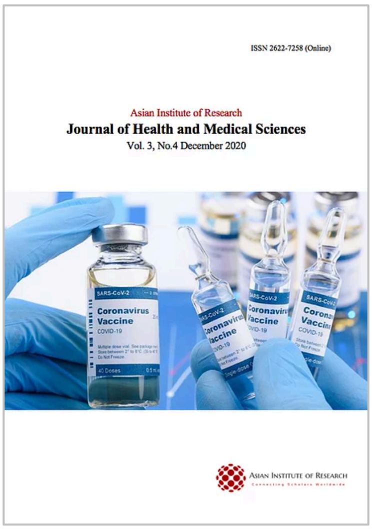

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Effects of Different Sintering Times on The Adaptation of Monolithic Zirconia Crowns

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Abstract
Changes to heat and time during the sintering process, which is one of the most critical stages in the production of monolithic zirconia crown restoration, can affect the physical properties of the material. The study was examined the effect of change in sintering time on the adaptation of monolithic zirconia crowns. Sixty monolithic crowns in upper first molar tooth form were produced from Y-TZP semi-sintered different blocks. Samples were divided into three groups according to brands and two subgroups according to sintering time (n=10). Marginal and internal gaps were photographed with a video microscope at x 180 magnification. The two-way ANOVA test was used for the effect of brand, and sintering time on adaptations were examined. There was no statistically significant difference in the overall gap values between the groups according to the long sintering times ($p > 0.05$). In crowns produced from zirCAD and Lava blocks, marginal gap values were not affected statistically significantly from sintering time ($p>0.05$). In crowns produced from katana blocks, shortening of sintering time caused a statistically significant increase in marginal and occlusal gap values ($p<0.05$). It can be said that the sintering time and temperature are an important factor in the biocompatibility of monolithic crowns.

Keywords: Yttria Stabilized Tetragonal Zirconia, Marginal Gap, Biological Adaptation, Dental Crown, Time

1. Introduction

Zirconium is a material with high biocompatibility and has no local or systemic side effects (Griffin, 2013). They also reduce the risk of pulp irritation that may occur due to their low thermal conductivity (Pereira et al., 2016). Since they are in opaque white colour, they are used as infrastructure material by veneering with feldspathic porcelains (Denry & Kelly, 2008). The biggest problem we encounter in these crowns and the reasons for being short-lived are cohesive breaks, especially in veneer porcelain (Suttor et al., 2001). For this, monolithic crowns produced with only one material, which are produced with CAD/CAM systems, do not need veneer porcelain, have been developed. Recently, the clinical indication of monolithic zirconium (MZ) has been expanding due to its superior mechanical properties (Marchack et al., 2011).

The term monolithic comes from the Greek words "mono: single" and "lithos: stone." It means that the materials have a regular feature throughout. Monolithic materials are two or more phases as microstructures. They still have fixed properties. MZ is partially stabilized with yttrium (0.01%). This process gives high bending resistance (1570 MPa) and high heat resistance (up to 2600 ºC). This resistance to heat, in particular, ensures that it is a material with high dimensional stability (Zhang et al., 2013). The atoms in the structure of these blocks, also called solid or translucent, are intertwined without any organic binder (Laboratories G. Bruxzir Solid Zirconia., n.d.). Moreover, their abrasion resistance is very close to the natural tooth. While the microstructure of the porcelain used as veneer ceramics causes abrasions and aging in the opposite natural tooth over time, MZ crowns do not have such an abrasion disadvantage (Batson et al., 2014). Their high resistance to breakage provides the advantage of use in cases where the interocclusal distance is insufficient. Even with an occlusal thickness of 0.5 mm, they can show sufficient resistance and durability, allowing them to be used in posterior restorations (Vafaee et al., 2017).

MZ restorations are prepared with CAD/CAM systems. For this reason, an appropriate closing relationship is obtained with the opposite teeth or restorations. Batson et al., in their study comparing the clinical properties of metal-ceramic, lithium disilicate, and MZ; They reported that crowns produced with CAD/CAM have acceptable high clinical properties. However, they reported that MZ showed no occlusal incompatibility with the antagonist region at 80%. Also; They reported that the gingival margin fit of MZ crowns is sufficient when evaluated according to the US health service criteria (Laboratories G. Bruxzir Solid Zirconia., n.d.).

One of the most important factors in long-term successful clinical results of the restorations is marginal fitting. Marginal misfit can cause plaque retention and bacterial microleakage, damaging both dental and supporting periodontal tissues (Kohorst et al., 2009). There is no definite consensus on the clinically acceptable full maximum range value in the scientific literature. Christensen proposed 34 to 119 µm as an acceptable, marginal range (Christensen, 1966). Currently, most authors use the criterion specified by McLean as 120 µm as the maximum acceptable, marginal gap for long-term success. (McLean & von, 1971) Besides marginal misfit, the restoration sits. Another factor affecting retention and survival is internal misfitting. Anadioti et al. The 25 µm thick cement spacer has shown to increase the casting seat and fit (Anadioti et al., 2015). Besides, an internal misfit can reduce the fracture resistance of all-ceramic restorations (Badran et al., 2019).

The most popular way to produce zirconia restorations now is to use the sintered zirconia blocks, which facilitates a smoother grinding process of the planned prosthesis. A specific sintering protocol is applied in order to reach the final density and maximum strength of the milled zirconia restoration, but together, the final sintering process is accompanied by a high shrinkage (about 20-30%) (Lankford et al., 1988). For compensate this shrinkage, the milled framework dimensions are enlarged by a single predefined percentage for all (Lankford et al., 1988).

High marginal fitting is an important factor for the clinical success of fixed restorations. Poor marginal adaptation causes the development of secondary caries and periodontal diseases (Felton et al., 1991). On the other hand, adjusting the prosthesis to ensure proper fit creates stress concentrations that can trigger the tetragonal to monoclinic (t → m) phase transformation of zirconium, which could potentially lead to disaster for the survival of the prosthesis (Piconi & Maccauro, 1999).

The sintering time and temperature change the grain size of the zirconia. The longer the sintering time and the higher the temperature, the larger the resulting grain size, which can lead to an increase in zirconia creep deformation and can, therefore, lead to distortion of the last crowns. As the particle size increases >1µm, zirconia becomes less stable and more prone to higher phase transformation (tetragonal to monoclinic phase). On the other hand, a smaller grain size <0.2 µm can be achieved fracture toughness (Stawarczyk et al., 2013). The most common sintering method for zirconia uses conventional furnaces at temperatures between 1350^0 and 1600^0 C and waiting times of 2 to 4 hours. An alternative sintering protocol proposed by manufacturers is a rapid sintering protocol that is claimed to save time and be more economical. The manufacturers 'in-house' testing claims similar compatibility, translucency, and colour stability of zirconia restorations with standard or rapid protocols. However, actual test data to support the claim are lacking. Several recent in vitro studies have shown that a combination of high sintering temperatures with short sintering times increases the flexural strength of the zirconia (Ersoy et al., 2015) and increases its optical properties (Ebeid et al., 2014). Also, a recent in vitro study has shown that changing sintering times does not affect marginal adaptation of zirconia copings (Khaledi et al., 2019).

2. Method

Acrylic right maxillary first molar was prepared according to the preparation rules for the full ceramic crown. The master resin die was doubled with silicone. Sixty resin die samples were fabricated. The samples obtained were randomly divided into three groups of twenty. A total of 60 monolithic zirconia crowns were produced, including twenty full ceramic restorations from three different zirconia blocks. Each group was divided into two subgroups, and one group was sintered in a short time, and the other group was sintered in a long time.
Three different monolithic zirconia materials were used in this study (Table 1). Digital designs of MZ crowns were made in Exocad Valetta 2.2 programs (exocad GmbH, Darmstadt, Germany) (Figure 1). After the design process on the computer, Y-TZP blocks were subjected to milling with five-axis machine. Yenadent D40 (Yenadent Ltd., Istanbul, Turkey) unit was used.

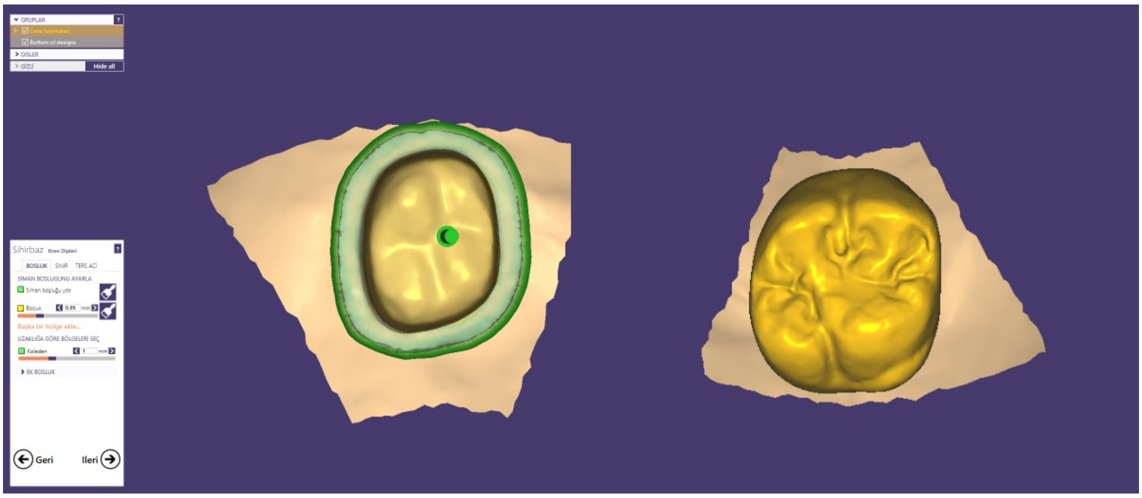

Figure 1. Digital desing on Exocad software

Table 1. Used blocks

Trade Name	Manufacturer
Lava™ Plus High Translucency Zirconia	3M, United Kingdom
IPS e.max ZirCAD Prime	Ivoclar Vivadent AG, Liechtenstein
KATANA™ HT	Kuraray Europe GmbH, Germany

Short and long sintering processes were applied to the samples in the 'Sirona inFire HTC speed' oven (Sirona Dental Systems GmbH, Bensheim, Germany). Time/temperature charts of short and long sintering processes are shown in Table 2.

Table 2. Sintering Processes

Short-term				Long-term		
	Heat rise	Heat	Waiting time	Heat rise	Heat	Waiting time
	°C/min.	°C	Min.	°C/min.	°C	Min.
Stage 1	10	0	0	12	0	0
Stage 2	10	0	0	70	1540	30
Stage 3	12	1540	120	70	1100	0
Stage 4	12	300	0	70	750	0

The silicone replica method was used to evaluate the compatibility of MZ crowns with resin abutments. Liquid petroleum jelly onto the resin die (Vazelin Blanc, Kalimed Medikal, Ankara, Turkey) was applied (Figure 2a,b). The polyvinylsiloxane material to be applied afterwards was prevented from deforming during detachment. The polyvinyl siloxane impression material (Xantopen VL Plus, Kulzer GmbH, Hanau, Germany) was squeezed into the inner surface of the samples with the help of a silicone dispenser gun and placed on the supports with 50 N load pressure. (Figure 2c) This load pressure was continued until the polymerization of the silicone material was completed.

After completing the polymerization of the silicon material, the samples were carefully removed from the resin dies. A different colour silicone material (president, Xtra light body, Coltène, West Sussex, UK.) was placed in the samples to support the thin silicon layer. After completing the polymerization of the silicone impression material applied for the support, the silicone replicates obtained were delicately separated from the supports. The presence of deformation on the surfaces of the separated silicone replicas was examined under the dental loop, and the same procedures were repeated for samples that did not preserve their integrity.

Silicon replica samples were cut with a scalpel to pass through the central occlusal groove in the mesiodistal direction. Markings were made on the silicone parts for the standardization of the measuring points. The adaptation of the copings was observed with at x 180 magnification with a video microscope (Lapsun Video Microscope, Lapsun, Hong Kong, China). (Figure 3c). All sample photos were taken for calibration using a precision micrometre with an interval of 0.01 mm. In twelve points determined by Holmes and et., the marginal and internal gap values were repeated five times for each point and averaged. Thus, 60 measurements were performed for each sample. All measurements were made with IC Measure software (The Imaging Source Europe GmbH, Bremen, Germany).

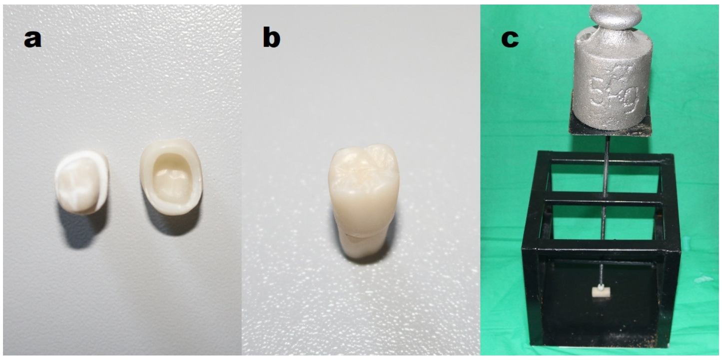

Figure 2. a: resin die, b: monolithic crown, c: applied light body silicon with 50 N force

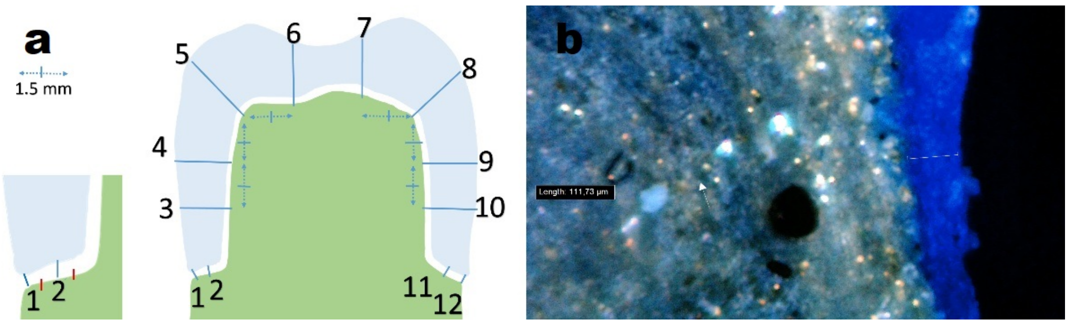

Figure 2. a:. marginal points (1,2,11,12), axial points (3,4,5,8,9,10); occlusal points (6,7) internal points (3,4,5,6,7,8,9,10), b: microscopic measurement image

Data analyzed with SPSS 22 program (SPSS Inc). The two-way ANOVA statistical analysis was used since the effects of brand and sintering time on the marginal and internal intervals were examined at the same time. The Leneve test was performed for control of the homogeneity of the group variances. The variations of the comparison groups were found to be similar. Due to the variance homogeneity, F statistics obtained as a result of the ANOVA test were accepted as reliable. Marginal, axial, and occlusal values were examined separately, and the results were evaluated within themselves. The presence/absence of statistically significant significance among the values obtained was evaluated with "T-test."

3. Results

There is no statistically significant difference between the marginal interval values of MZ crowns with long and short sintering times produced using Lava blocks ($p>0.05$). While average marginal interval values of samples sintered with zirCAD blocks in a long time were determined as 35.80±8.08 µm; Average marginal interval values of samples sintered with zirCAD blocks in a short time were found to be 32.20±10.92 µm. As a result, there is no statistically significant difference between the marginal interval values of MZ crowns with long and short sintering times produced using zirCAD blocks. While the average marginal interval values of the samples sintered with Katana blocks in a long time were determined as 39.20±15.72 µm; Average marginal interval values of samples sintered with Katana blocks in a short time were found to be 78.65±11.23 µm. The change in sintering time affected the marginal interval values of MZ crowns produced using Katana blocks. Marginal interval values in samples produced with long sintering time were found to be statistically significantly lower than in samples with short sintering time ($p<0.05$). As a result, there is no significant difference between the marginal interval values of samples produced with zirCAD and Lava type materials with short and long sintering times. However, in Katana blocks, a statistically significant difference was detected between the groups that were sintered in a long and short time ($p<0.05$).

In Lava and Katana type materials, there is no significant difference between the axial gap values of the samples produced with short and long sintering times. However, in zirCAD blocks, a statistically significant difference was detected between groups that were sintered in a long and short time ($p<0.05$). When the differences between the groups are evaluated, no difference between the groups can be detected in the samples produced by sintering in a long time; in samples produced by sintering in a short time between Lava blocks and zirCAD blocks ($p>0.05$); Statistically significant differences were found between Katana blocks and zirCAD blocks ($p>0.05$).

In Lava groups, no significant difference can be detected between the occlusal interval values of the samples produced with short and long sintering times. In contrast, in zirCAD and Katana blocks, there was a statistically significant difference between the groups that were sintered in a long and short time. There was no difference in overall internal interval values between the groups for long-term sintering. For the short term, the zirCAD group had the lowest internal interval value and was statistically different from the Katana group.

For Lava type materials, there is no significant difference between the cement range average values for all measurement points of samples produced with short and long sintering times. However, in zirCAD and Katana blocks, a statistically significant difference was detected between the groups that were sintered in a long and short time.

Table 3. Overall gap values

Groups	Sintering process	Marginal gap		Axial gap		Occlusal gap		General average gap		Overall Internal gap	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Lava™ Plus	Long	59.35	14.01	45.00	9.92	126.55	16.33	64.67	30.51	67.98	35.48
	Short	69.05	17.30	50.85	13.29	136.80	15.75	70.74	33.87	72.00	39.99
IPS e.max ZirCAD	Long	35.80	8.08	50.40	11.26	125.90	30.72	60.61	36.03	73.55	37.69
	Short	32.20	10.92	44.00	13.26	99.05	14.99	47.86	26.33	55.25	28.72
KATANA™ HT	Long	39.20	15.72	52.18	13.57	125.50	28.03	61.00	34.08	68.46	38.11
	Short	78.65	11.23	44.85	13.91	188.25	23.83	81.14	52.28	82.48	63.45

4. Discussion

Three different blocks and two different sintering times were evaluated in this study. The null hypothesis of the study was determined as "changes in sintering time do not affect the marginal and internal adaptation of MZ restorations. When the total cement film thickness evaluated, this hypothesis was accepted for Lava Plus group, while the hypothesis was rejected for Katana and zirCAD groups.

The marginal gap values of all the samples in this study remained within limits accepted in the literature (McLean & von, 1971). Crowns produced in a short period using the highest marginal range value with 78.65±11.23 µm, in Katana group. Crowns produced with the lowest marginal range value with 32.20±10.92 µm in zirCAD group. When the marginal interval values of crowns produced by sintering in a long time were examined, a statistically significant difference was found between the groups. Accordingly, zirCAD 35.80±8.08 µm, Katana 39.20±15.72 µm and Lava 59.01±14.01 µm marginal interval values were measured. This result contradicts the results of the study of three different zircon infrastructures conducted by Karataşlı et al. (Karataşli et al., 2011). In this study, the mean marginal interval value for Lava was reported as 24.6±14.0 µm, and the lowest marginal interval value was obtained in Lava infrastructures. However, unlike this study, they produced a non-anatomical infrastructure, not MZ crowns. Lee KH et al. reported the highest values for Lava (87.2±22.8 µm) in their studies evaluating the marginal compatibility of all-ceramic crowns (Lee et al., 2015).

Literature studies are showing that sintering time affects the mechanical properties of zirconia restorations. The sintering temperature and duration are thought to affect the particle size of the material. Increased grain size; "T-m" reduces the stability and strength of the material by stimulating the phase transformation. Studies are showing that high sintering temperature and long sintering time increase the particle size. For a natural "t-m" phase transformation, very fine particles are required (Lee et al., 2015). Lazar et al. stated that the presence of thick particles in the microstructure of zirconia ceramics stabilized by yttrium is an indication of the presence of the monoclinic form (Lazar et al., 2008). This contrasts with the results of Hjerppe et al.'s work on zirconia discs sintered at different times. In this study, there was no difference between samples in terms of micro strength. The reason for this is; It may be related to the length of the sintering time. In the study, discs produced by short sintering were applied for 1 hour 40 minutes increase and 1 hour waiting time. Although it was not statistically significant in the same study, it was observed that the particle size of the discs produced by short sintering decreased (Hjerppe et al., 2009).

Beuer et al. emphasized that milling is done mostly before sintering, and therefore, sintering may affect the compatibility of zirconia restorations (Beuer et al., 2008). In this study, shortening the sintering time did not cause a statistically significant difference in the marginal interval values of the samples produced with zirCAD and Lava brand blocks. In contrast, the samples produced with Katana brand blocks increased statistically significantly with the reduction of the sintering time ($p<0.05$). Although acceptable values (<120 µm) for the marginal range are defined in the literature, there is no consensus on what the ideal internal range value is (Tsirogiannis et al., 2016). Wettstein et al. reported that the axial pitch values of the restorations were between 9 microns and 38.3 microns (Wettstein et al., 2008). In a study performed on full ceramic restorations, it was shown that smaller spaces in the axial wall and marginal area were better against compressive forces (Badran et al., 2019). The optimal internal range value was determined as 73 µm against the compressive forces, and there was no significant difference in the compatibility of the restorations at the range values up to 122 µm; however, it is stated that there is a decrease in their durability. Internal range values; The cementation stage, the underlying material used, the magnification device used to make measurements, measurement points and numbers can be affected by many factors (Beschnidt & Strub, 1999).

The internal gap values of MZ produced by two different CAD/CAM systems (Zirkonzahn and Ceramill) were compared, and the internal gap values varied according to the areas measured. The highest internal range values were determined in the occlusal areas (213.40±19.57 µm) of the samples produced with the Ceramill system, and the lowest internal range values (42.90±6.84 µm) in the lingual axial walls (Ha & Cho, 2016).

The highest values were recorded in the occlusal range measurements (99.05±14.99 µm and 188.25±23.45 µm) in this study. Luthard et al. reported that the occlusal spacing values of the restorations produced using CAD/CAM systems might cause a higher resolution of the scanning device. In the same study, it was reported that the inability of the scanner to read the sharp edges thoroughly and the small details that could not be copied by the scraping unit might result in high occlusal spacing values (Kelvin Khng et al., 2016).

In MZ restorations, the cement spacing is set up during digital design. There are literature studies on the ideal cement range (Contrepois et al., 2013). In this study, the samples were not cemented, but they were designed with the cement spacing in mind. The cement gap value is set to 50 µm. Since cementation of zirconia restorations is not required to use an adhesive based cement, 50 µm cement interval value may be sufficient. Al-Rabab et al. .examined the effect of cement spacing values on marginal spacing in restoration designs in CAD/CAM systems and showed that marginal compatibility is better than designs produced with 100 µm cement spacing values in designs produced with a 50 µm cement spacing value (Contrepois et al., 2013).

Weaver et al. placed the restorations on the abutments with a non-standard press and said that the finger pressure was an average of 78.5 N. They also reported that pressure differences did not make a statistically significant difference on the silicone layer (Weaver et al., 1991), but standard pressure used in this study.

Conclusions

Within the limitations of this study, the following results were obtained:
1. No statistically significant difference was observed between the systems used in the study in terms of total cement film thickness values by the long sintering times.
2. In crowns produced from zirCAD and Lava blocks, marginal gap values were not statistically significantly affected by the sintering time.
3. The shortening of sintering time in crowns produced from Katana blocks caused a statistically significant increase in marginal and occlusal gap values.
The sintering times recommended by the manufacturer of the materials used in the production of monolithic crowns must be complied with.

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