THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Effect of cementation and aging on the marginal fit of veneered and monolithic zirconia and metal-ceramic CAD-CAM crowns

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ABSTRACT

Statement of problem. Marginal fit of zirconia restorations is an important criterion for their long-term success. However, in spite of the wide use of zirconia in dentistry, the relationship between marginal fit and low-temperature degradation from aging is unclear.

Purpose. The purpose of this in vitro study was to compare the marginal adaptation of veneered and monolithic zirconia and metal-ceramic computer-aided design and computer-aided manufacturing (CAD-CAM) crowns before and after cementation and to evaluate the influence of artificial aging on the adaptation of zirconia crowns.

Material and methods. Seventy-two standardized dies were prepared to receive a posterior crown and randomly divided into 6 groups (n=12) as per the material and the presence or not of cement: metal-ceramic, veneered zirconia, and monolithic zirconia. The zirconia groups were subjected to accelerated low-temperature degradation through hydrothermal aging in an autoclave at 131 °C and 0.17 MPa for 5 and 20 hours. A scanning electron microscope with a magnification of ×1000 was used for marginal adaptation measurements, and X-ray diffraction (XRD) was used to characterize phase transformation degradation. The data were statistically analyzed using 2-way ANOVA, repeated measures ANOVA with Greenhouse-Geisser correction, and the *t* test (α=.05).

Results. No significant differences in the marginal discrepancy were recorded among the analyzed groups. The presence of cement did not influence marginal fit in any treatment group. No significant differences were observed in the marginal adaptation values before and after aging (P>.05). After 20 hours of aging, the monoclinic phase increase to 8.3% on veneered zirconia and to 3.1% on monolithic crowns.

Conclusions. Monolithic and bilayer CAD-CAM zirconia crowns showed marginal gaps that were within an acceptable range of clinical discrepancy, regardless of cementation. Marginal adaptation was not influenced by aging. Low-temperature degradation did not lead to a significant transformation from the tetragonal to monoclinic phase. (J Prosthet Dent 2021;125:323.e1-e7)

Marginal fit is one of the most important factors, together with fracture resistance and esthetics, for the long-term success of ceramic restorations.[1-3] Factors that influence marginal adaptation such as the tooth preparation design,[4-7] veneering process,[5-9] cement space parameters,[10,11] and cementation have been evaluated.[2] However, studies that evaluated the relation of cement with the marginal adaptation are sparse.[1,6,12-14] In addition, materials for computer-aided design and computer-aided manufacturing (CAD-CAM)[1,3,10,14-19] and the selection of the CAD-CAM processing workflow[20] also affect marginal accuracy.

Based on the study by McLean and von Fraunhofer,[21] a marginal gap between 100

Supported provided in part by a research grant between the University Complutense of Madrid and AVINENT Implant System SL. (No. 322-2014), through the last author.
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Clinical Implications

Accurate marginal fit is essential to avoid clinical complications. The results of this in vitro study demonstrated that cementation with an adequate cement space and aging will not affect the marginal fit of monolithic and veneered zirconia CAD-CAM crowns.

Table 1. Chemical composition and microstructure of zirconia restorations tested

Chemical Composition	Units	Z-CAD HD (Veneered)	Z-CAD HTL (Monolithic)
ZrO_2+HfO_2+ Y_2O_3	wt%	>99.5	>99.5
Y_2O_3	wt%	5.2	5.2
Al_2O_3	wt%	0.25	0.05
SiO_2	wt%	≤0.02	≤0.02
Density	g/cm^3	6.08	6.08
Grain size	μm	<0.4	<0.4

and 120 μm has been used as a clinically acceptable range and related to restoration longevity.[1,13,15,16,18,19] With CAD-CAM technology, marginal adaptation can be improved when compared with conventional techniques.[1,13,15] However, machining may contribute to the low-temperature degradation (LTD) of zirconia, increasing the monoclinic zirconia content.[22] The highest percentage of this phase has been reported in thin cervical margins of the copings, where machining conditions are more aggressive.[23] Therefore, the mechanical properties and the long-term stability of treated frameworks may be different from those of the bulk material.[22]

The effect of LTD remains unclear in spite of the clinical use of zirconia in different technical and biomedical applications.[24] Aging consists of a slow and spontaneous transformation from the tetragonal to the monoclinic phase (t→m), associated with the presence of water or water vapor, which begins in isolated particles on the surface of the zirconia by a stress corrosion type mechanism.[25] As the grains are transformed, a local volume increase occurs, which accentuates and disrupts the crystalline structure.[25]

Forecasts of transformation toughening at mouth temperature have been based on aging tests in an autoclave.[22,26-32] One hour of autoclave treatment at 134 °C has been reported to simulate 3 to 4 years of clinical service at 37 °C,[33] although that correlation has been disputed[28] and standardization regarding the use of the autoclave and the methods used to evaluate the sensitivity of the specimens is lacking. The International Organization for Standardization (ISO) standard 13356:2008[34] specifies using an autoclave at 134 °C and at 0.2 MPa for 5 hours.

Veneered zirconia restorations have been reported to have a higher rate of major chipping of the veneering ceramic than metal-ceramic (MC) restorations,[35] leading to the increased use of monolithic zirconia crowns. However, these zirconia restorations are in direct contact with moisture and subject to pH variations, mechanical loads, and temperature variations causing tetragonal phase instability, which could lead to aging.[27] Strategies to control the aging stability and to improve the translucency of monolithic zirconia restorations have been used, including lowering the alumina amount, increasing the yttria content, or adding 0.2 mol% lanthanum oxide.[36]

The purpose of this in vitro study was to compare the marginal adaptation of veneered zirconia (VZ), monolithic zirconia (MZ), and MC CAD-CAM crowns, as well as the influence of the cement on the fit of the restorations, and to analyze the influence of aging on the adaptation of zirconia crowns. The null hypotheses were that no differences would be found in marginal discrepancy among the restorations analyzed, with or without cement, and that aging would not influence the marginal adaptation of zirconia restorations.

MATERIAL AND METHODS

Seventy-two standardized dies simulating a first mandibular premolar were machined in stainless steel (Type 316L; Masteel). The dies were designed with a 1-mm-wide chamfer, 5 mm in height, and a 6-degree angle of convergence.[1,13,15,16,18,19] The specimens were randomly divided (www.alazarinfo.es) into 6 groups (n=12 each as per the results of power analysis) categorized as per the material and the presence or not of cement: MC, VZ, and MZ. The cemented crowns were identified with the letter C.

The MC crowns (n=24) were manufactured from a cobalt-chromium alloy (Coron; Institut Straumann AG). The specimens were scanned (Straumann CARES Scan CS2; Institut Straumann AG), and the data were entered into the system software program (Cares Visual 8.0; Institut Straumann AG). The thickness of the coping selected in the study was 0.5 mm. The frameworks were milled with a unit (Cares In-Lab; Institut Straumann AG), and the veneering ceramic (Vita VM 13; VITA Zahnfabrik) was applied as per the manufacturer's instructions.

The zirconia crowns were fabricated by the same dental laboratory technician, following the manufacturer's specifications. Details of both ceramics are presented in Table 1. To fabricate the VZ crowns, the abutments were scanned and digitized (Lava Scan ST; 3M ESPE). The frameworks were designed (Lava Design

Table 2. Sintering zirconia parameters

Material	Heating Rate °C/h	Maximum Temperature °C	Time/ h	Cooling
Z-CAD HD (Veneered)	600	1500	1	Slow cooling
Z-CAD HTL (Monolithic)	600	1450	2	Slow cooling

Table 3. Veneering and glaze firing procedures used

Main Feature	Monolithic Zirconia Glaze	Dentin Layers	Veneered Zirconia Glaze
Time to rise the temperature	6 min	8 min	3 min
Starting temperature	450 °C	450 °C	450 °C
°C/min	60 °C	40 °C	60 °C
Maximum temperature	770 °C	750 °C	725 °C
Cooling	Fast	Fast	Slow

Software; 3M ESPE), milled from presintered zirconia blocks (Z-CAD HD; Metoxit AG) in the milling unit (Lava Form; 3M ESPE), and sintered in a furnace (Programat S1; Ivoclar Vivadent AG). Details of the sintering process are presented in Table 2. The copings (n=24) were steam cleaned and veneered with hand-layered porcelain (IPS e.max Ceram; Ivoclar Vivadent AG). The ceramic was fired in a furnace (Vacumat 4000 Premium T; VITA Zahnfabrik) (Table 3). The manufacture of monolithic restorations (Z-CAD HTL; Metoxit AG) (n=24) was similar to that of the VZ group, but they were left unveneered and only a glaze layer was applied. A predefined cement space of 50 μm was selected for all crowns.

One of the crowns was waxed and duplicated with a silicone material (Express Penta Putty and Express Penta Ultra-Light Body; 3M ESPE) to serve as a guide for layering the copings and ensure the same porcelain thickness of the veneered crowns.[37,38] All crowns had identical final dimensions, which were verified with a digital micrometer (Mitutoyo).[1,16,38]

All the crowns were cemented with a resin cement (RelyX U200; 3M ESPE). The cement was applied to the axial surfaces of the restorations,[1,16,18,38] and a seating load of 10 N was applied for 10 minutes with a dynamometric key (820/70; USAG).[1,16,18,19,38] After cementation, the crowns were stored in a dry place at room temperature.

A scanning electron microscope (SEM) (JSM-6400; JEOL) at ×1000 magnification was used to evaluate the external vertical marginal gap of the crowns.[16,18,19] One location was marked (Lumocolor permanent pen 318; Staedtler) in the middle of the buccal and lingual surfaces of each crown to ensure the measurements at the same point for all crowns.[1,16,18,19] The specimens were positioned perpendicular to the optical axis of the microscope at an angle of 20 degrees to ensure repeatable projection angles.[16] The images obtained from the SEM, with the measurement in micrometers (INCA software v4.04; ETAS), were edited by using an imaging software program (ImageJ 1.49; NIH) to increase the number of measurements per specimen.[39] Therefore, 36 measurements (18 per surface) were recorded for each crown. The same experienced operator (M.P.) performed the SEM analysis.

Each zirconia group was then further subjected to accelerated LTD through hydrothermal aging in an autoclave (Microclave 2 4001404; JP Selecta) at 131 °C and 0.17 MPa for 5 and 20 hours. The marginal fit was measured after aging.

X-ray diffraction (XRD) was performed to assess the LTD by monitoring changes of the surface content of the monoclinic phase. The crowns were embedded in an acrylic resin (SamplKwick; Buehler), ground down, and polished (Phoenix Beta; Buehler) with diamond disks (Apex DGD; Buehler). The crystalline phases of the specimens were analyzed with a diffractometer (D8 ADVANCE; Bruker) with Cu-Kα radiation (K=1.5405981 Å), 40 kV, and 30 mA. Scans were performed in the 2θ range of 27 to 33 degrees at a step size of 0.01 degrees and a scan speed of 0.06 degrees/min. The mass fraction of the monoclinic phase (X_m) was evaluated by the Garvie and Nicholson equation[40]:

$$Xm = [\mathrm{Im}(-111) + \mathrm{Im}(111)] / [\mathrm{Im}(-111) + \mathrm{Im}(111) + It(111)],$$

where I_t and I_m represent the integrated intensities of the tetragonal and monoclinic and peaks. The monoclinic volume fraction (V_m) was evaluated by the equation reported by Toraya et al:[41]

$$Vm = 1.311 \times Xm / (1 + 0.311 \times Xm).$$

The mean and standard deviation was calculated for each group. The Shapiro-Wilk test confirmed that the data were normally distributed, and the Levene test verified the homogeneity of the variances. Two-way ANOVA was performed for comparisons among the groups and to analyze the influence of cement. Repeated measures ANOVA with Greenhouse-Geisser correction was used to analyze the influence of time of aging, Finally, a paired *t* test was used to compare the influence of cement in the groups. Statistical analysis was performed with a statistical software program (IBM SPSS Statistics, v22.0; IBM Corp) (α=.05).

RESULTS

The mean and standard deviation of the marginal fit values for each group are presented in Table 4. VZ, MZ, and MC crowns without aging and without cementing exhibited similar vertical marginal discrepancies (Fig. 1). The ANOVA revealed no significant differences in the

Table 4. Mean ±standard deviation (μm) of marginal discrepancies in each group (n=12)

Group	No Aging	Aging 5 h	Aging 20 h
MC	49.8 ±15	–	–
MCC	52.1 ±17	–	–
VZ	49.6 ±15	50.1 ±21	48.6 ±19
VZC	53.4 ±15	56.7 ±13	60.0 ±15
MZ	47.5 ±13	49.7 ±19	50.6 ±17
MZC	47.5 ±21	50.1 ±18	51.5 ±16

MC, metal-ceramic uncemented; MCC, metal-ceramic cemented; MZ, monolithic zirconia uncemented; MZC, monolithic zirconia cemented; VZ, veneered zirconia uncemented; VZC, veneered zirconia cemented.

marginal fit among the studied groups with (F=0.27; *P*<.59) or without cement (F=0.41; *P*<.66). Similarly, the paired *t* test showed no differences in the marginal fit within each material group.

When marginal adaptation was analyzed on zirconia crowns after aging, no significant differences were observed among the groups after 5 hours (F=0.43; *P*<.51) and 20 hours (F=0.42; *P*<.52). Likewise, no differences were shown within each zirconia group after 5 and 20 hours of aging, either before or after cementation (Fig. 2). When the time of aging was compared, no significant differences were observed in the marginal adaptation values for VZ (F=0.03; *P*<.93), VZC (F=1.28; *P*<.29), MZ (F=0.29; *P*<.70), or MZC groups (F=0.69; *P*<.45).

Figure 3 shows the XRD patterns. They were almost identical, revealing that the surfaces of both zirconia systems mainly consisted of tetragonal zirconia and a minor amount of monoclinic phase. The surfaces of MZ and VZ groups without aging contained 0.8% and 1.2% of monoclinic phase, respectively. The monoclinic content was 1.6% for the MZ group and 3.0% for the VZ group after 5 hours and 3.1% and 8.3% respectively after 20 hours. Therefore, a slight increase in monoclinic phase was observed in both zirconia groups after aging.

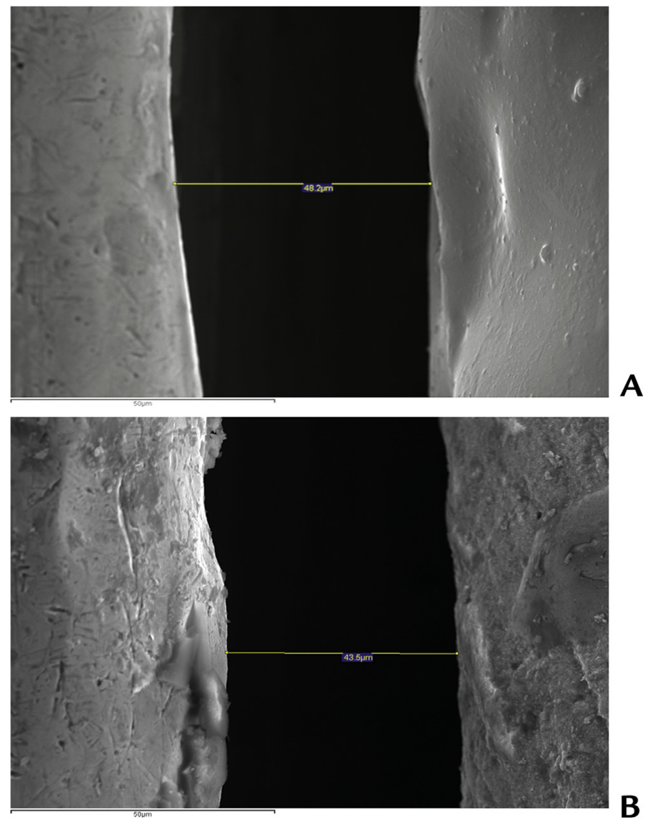

Figure 1. Representative scanning electron microscope image (original magnification ×1000) of marginal discrepancy before cementation. A, Veneered zirconia crown. B, Monolithic zirconia crown.

DISCUSSION

The results of this study support the acceptance of the null hypotheses that no differences would be found in marginal discrepancy among the restorations analyzed, with or without cement, and that aging would not influence the marginal adaptation of zirconia restorations.

The marginal adaptation of all tested groups was within the clinically acceptable range,[21] with values around 50 μm. The results indicate the precision of the CAD-CAM technology.[1,15]

In the present study, the analyzed groups exhibited similar vertical marginal discrepancies. The results were consistent with those of previous studies on crowns[10] and posterior fixed partial dentures.[19] However, Hamza and Sherif[3] reported slightly lower values for MZ crowns, and slightly higher values of marginal adaptation for ZV crowns have been reported in other studies.[5,7,8] One of the reasons for such differences is that the choice of a CAD-CAM system seems to significantly influence marginal adaptation.[3,10,15-17,20] In addition, methodological variables could explain such divergences.

Regarding the cement, no significant effect on marginal adaptation was observed in any treatment group. Few studies have compared the fit of the restorations before and after cementation. Consistent with the present study, previous studies[1,6,14] have also reported that the cement does not influence the marginal fit. Conversely, other studies[12,13] have reported an increase in the marginal discrepancy after cementation. The differences observed may be because of a lack of standardization regarding the type of cement used or the seating force of the restorations. In addition, the different values may be because of a difference in the cement space selected. In the present study, 50 μm was selected as per previous studies that reported that this space is adequate for the precision of fit of zirconia restorations.[1,11] The marginal fit values obtained were similar to those reported in previous studies for cemented MZ crowns[18] and VZ crowns.[16] Moreover, in the present study, similar adaptation values were found for zirconia and MC crowns. This result may be attributed to the fact

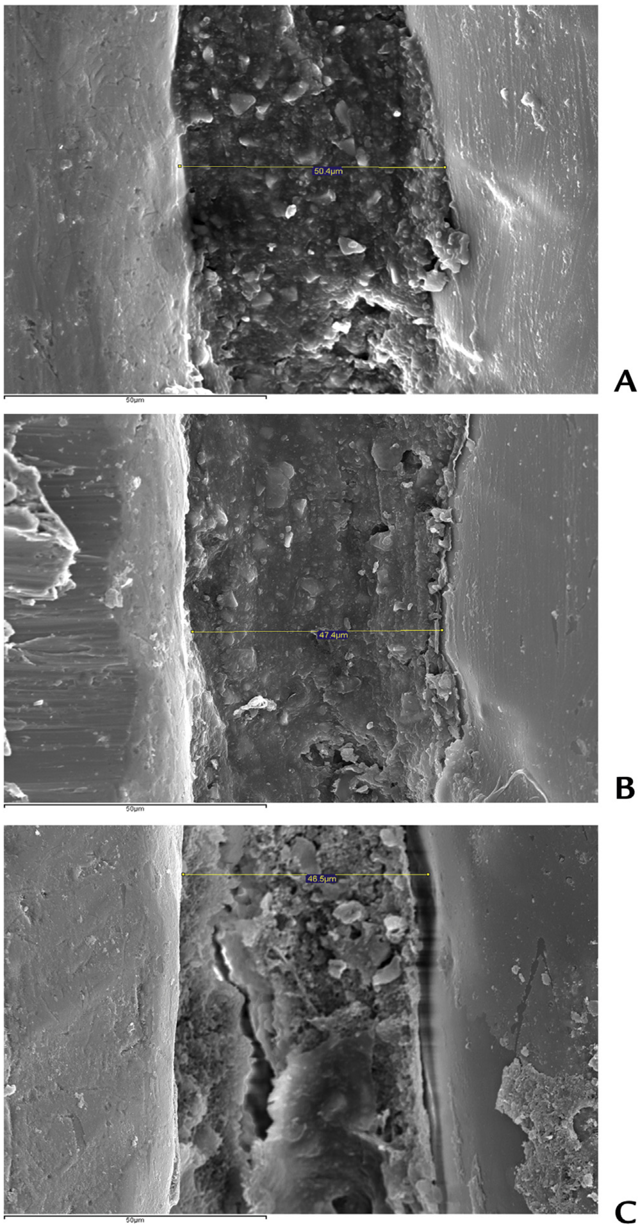

Figure 2. Representative scanning electron microscope image (original magnification ×1000) of marginal discrepancy in monolithic cemented crown. A, Before aging. B, After 5 hours of aging. C, After 20 hours of aging.

that CAD-CAM technology has better adjustment accuracy compared with conventional techniques.[13]

Regarding the effect of aging, the results suggest that the t→m phase transformation does not affect the marginal adaptation of MZ and VZ crowns. XRD was used for this assessment because it is the most commonly used technique.[26] The MC crowns were not subjected to such analysis because the main component of the ceramic coating is a vitreous matrix and the percentage of crystalline phase is very low.[35] The authors are unaware of previous studies evaluating the marginal adaptation of zirconia crowns after aging in an autoclave. Only 1 previous study evaluated the influence of aging to simulate oral conditions by cyclic fatigue on marginal adjustment, and no significant changes were reported after aging.[14] Before aging, the XRD analysis showed a small amount of monoclinic phase in the VZ and MZ crowns, which increased after 5 and 20 hours of aging. Sehgal et al[29] reported a high content of the monoclinic phase before aging in VZ disks, increasing the monoclinic content after aging at 134 °C for 5 hours. The detection of the monoclinic phase before aging could be attributed to the margin of error in the measurement or to damage produced by CAD-CAM machining.[22,23] However, the values found after aging for 5 and 20 hours were lower than the 25% allowed by the ISO 13356:2008[14] and were consistent with those of previous studies.[27-31]

A previous study reported that veneering firings did not enhance the influence of aging of zirconia core on phase transformation.[30] Conversely, in the present study, the VZ group aged more aggressively than the MZ group. Siarampi et al[31] analyzed the aging of 2 zirconia systems with bar-shaped specimens, and both had a monoclinic phase increase of 4% to 5% at 5 hours and about 15% at 10 hours. However, that study did not report the chemical composition of the ceramics, which could explain the discrepancies. Xie et al[30] reported that in zirconia bar-shaped specimens, the monoclinic content was between 9% and 15% for 10 hours and between 15% and 20% for 20 hours. Comparing the results with those of previous studies is difficult because the aging of zirconia is a phenomenon that is affected by different factors, including the grain size, type of stabilizing oxide, percentage and distribution of oxides, and processing workflow.[24] Furthermore, previous studies on aging and phase transformation have been performed on disk- or bar-shaped specimens, and the authors are unaware of studies that have analyzed the influence of aging on crowns.

In the present study, MZ crowns maintained hydrothermal stability compared with that of VZ crowns. Conversely, previous studies reported a reduction in hydrothermal stability and increased monoclinic phase [32,36] or lower than in the present study.[28] The differences may be because of a difference in the composition of materials and methodological parameters.

Limitations of the present study included its in vitro design, which may not replicate clinical conditions. However, metal dies were used, as in previous studies,[1,13,15,16,18,19] which provides standardization of the shape and dimension of the specimens[1,16] to avoid the influence of external factors. Another limitation of the study was that only vertical adaptation was evaluated, which may not reflect the overall adaptation. Future studies are recommended with other zirconia systems to

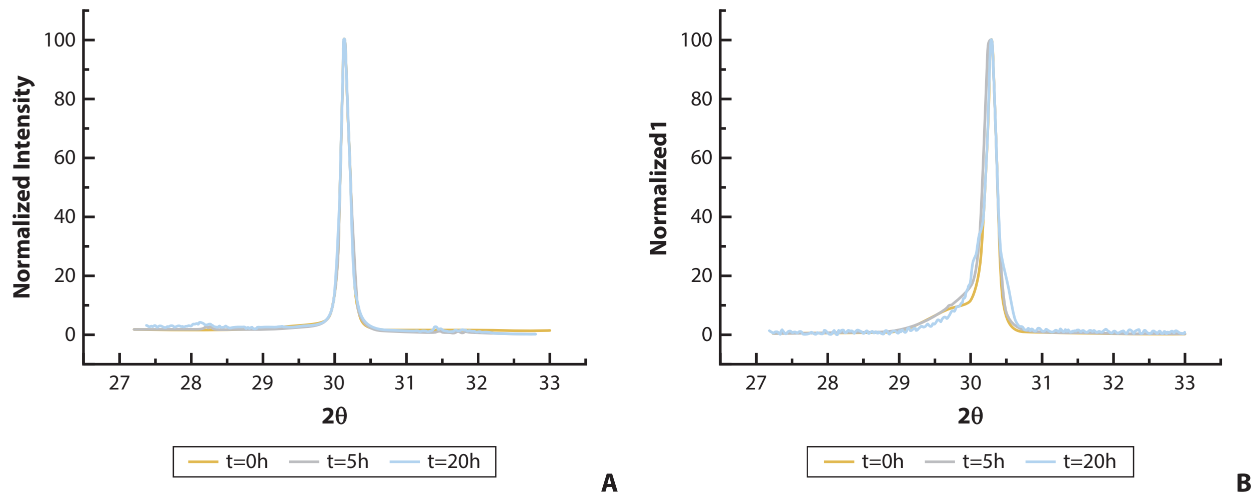

Figure 3. X-ray diffraction patterns (*black line*: surface before aging treatment; *red line*: surface after 5 hours of aging; *green line*: surface after 20 hours of aging). A, Veneered zirconia crown. B, Monolithic zirconia crown.

evaluate the effect of aging on the marginal fit, as well as clinical trials to assess the fit of zirconia restorations.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The marginal fit in all groups was within the clinically acceptable range, with values around 50 µm.
2. All tested groups showed similar marginal gaps with no differences among them.
3. Cementation had no influence on the marginal adaptation in any of the groups
4. Aging did not modify the marginal fit of the zirconia crowns.
5. XRD technique showed a slight increase of the monoclinic phase in both types of zirconia restorations after aging.

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Acknowledgments
The authors thank Dr Carmen Bravo, Center of Data Processing, Computing Service for Research Support, University Complutense of Madrid, for her assistance with the statistical analysis.

https://doi.org/10.1016/j.prosdent.2020.06.036