Original Article

Effect of ceramic cooling protocols and zirconia coloring on fracture load of zirconia-based restorations

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ABSTRACT

Background: Residual thermal stresses in dental porcelains can cause clinical failure. Porcelain cooling protocols may affect the amount of residual stresses within porcelain and also porcelain–zirconia bond strength. The objective of this study was to assess the effect of cooling protocols on the fracture load of porcelain veneered zirconia restorations.

Materials and Methods: Forty zirconia bars (31 mm × 6.5 mm × 1.35 mm ± 0.1 mm) were fabricated by computer-aided design and computer-aided manufacturing technology. Half of the specimens were immersed in the coloring agent for 2 min before sintering (yellow group). Thus, the specimens were divided into two groups of white (W) and yellow (Y) samples (n = 20). Heat-pressed ceramic was applied to all bars. After pressing, half of the samples in each group were immediately removed from the oven (fast cooling) while the other specimens remained in the partially open door (30%) oven until the temperature reached to 500°C. Samples were thermocycled for 5000 cycles and subjected to modified four-point flexural strength test by a universal testing machine at a crosshead speed of 0.5 mm/min. Two-way ANOVA, One-way ANOVA followed by *post hoc* Tukey honest significant difference tests were used for data analysis ($\alpha = 0.05$).

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Address for correspondence: Dr. Mahsa Abbasi, Department of Prosthodontics, School of Dentistry, Shahrekord University of Medical Science, Shahrekord, Iran. E-mail: mahsa.abbs@yahoo. com **Results:** Fractures were cohesive in all samples (within the porcelain adjacent to the interface). Two-way ANOVA showed that the effect of cooling protocol on the fracture load of samples was statistically significant (P < 0.001). In addition, the fracture load of W and Y groups was significantly different (P < 0.001). The white slow group showed the highest fracture load (179.88 ± 23.43 N). **Conclusion:** Slow cooling protocol should be preferably applied for zirconia restorations. Coloring agent used in this study had a significant negative effect on fracture load.

Key Words: Computer-aided design, dental bonding, dental porcelain, zirconium oxide

INTRODUCTION

Zirconia restorations, fabricated by computer-aided design and computer-aided manufacturing (CAD/CAM) technology are becoming increasingly popular in dentistry. Zirconia ceramics were first introduced to dentistry in 1969.^[1]

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Website: www.drj.ir www.drjjournal.net www.ncbi.nlm.nih.gov/pmc/journals/1480 Zirconium dioxide is the most common ceramic core material^[2] with excellent mechanical properties such as high fracture toughness,^[3] excellent biocompatibility,^[4] and weak bacterial adhesion.^[5] In addition, it has higher modulus of elasticity than high-noble alloys.^[6]

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For esthetic reasons, zirconia frameworks are often veneered with porcelain.^[7] Despite high-survival rate of zirconia-based restorations, fracture of the veneering porcelain is a common occurrence. Porcelain fracture may include crumbling, chipping, spalling, fracturing, and delamination.^[8-10]

Review studies have reported that chipping and cracking of the veneering porcelain is the most common cause of technical failure of zirconia restorations.^[11-13] In the most cases, these defects occur completely within the porcelain layer but in some cases, they occur at the zirconia-porcelain interface.^[14-16] Several factors may explain the occurrence of these fractures such as geometric parameters (coping design, ratio of coping thickness to that of the veneering porcelain), thermal parameters (coefficients of thermal expansion, cooling protocol) and structural parameters (internal ceramic cracks, wettability of the coping, the bond strength between ceramic, and coping).^[17-19]

Residual stress within the porcelain due to thermal incompatibility can be a key factor in clinical problems related to porcelain restorations.^[16,20,21] Although porcelain designed for veneering of the zirconia framework may have a coefficient of thermal expansion compatible to that of zirconia, potential internal stresses may develop.^[7]

The most widely accepted explanation for the occurrence of high-tensile stresses in the porcelain layer is fast cooling of zirconia restorations.^[22] Thus, after introducing zirconia restorations to dentistry, the manufacturers proposed slow cooling, and firing programs to decrease the risk of chipping of porcelain.^[23]

Zirconia is a weak thermal conductor compared to metal alloys and the other all-ceramic cores.^[22] Thus, during the firing process, porcelain experiences temperature rise or fall faster than zirconia framework. In contrast to the metal-ceramic restorations, when zirconia-based restorations are rapidly cooled down, the internal surface of the porcelain layer next to the zirconia core remains in a temperature higher than that of glass transition temperature for a longer period. The thermal gradient between the outermost and innermost porcelain layers determines the type and amount of residual stresses.^[24] A consensus exists regarding the selection of a coping with a modulus of thermal expansion higher than that of the veneering porcelain to obtain a positive thermal mismatch.^[25] Such a mismatch can cause compressive stresses in the veneering porcelain and prevent crack propagation.^[26] It has been shown that this mismatch does not affect the bond strength of coping to zirconia because it cannot change the zirconia phase at the zirconia-porcelain interface; however, this hypothesis has been challenged by another study.^[16]

Taskonak *et al.*^[27] reported that fast cooling of zirconia-based restorations causes surface compressive stresses while slow cooling causes surface tensile stresses. However, several *in-vitro* studies have emphasized the association of residual tensile stresses in the veneering porcelain and fast cooling protocol of zirconia restorations.^[20,24,28] Guazzato *et al.*^[29] showed that the occurrence of cracks in the veneering porcelain increases due to the fast cooling. It seems that fast cooling of zirconia restoration of residual compressive stresses in the surface and residual tensile stresses at the center of the veneering porcelain and exposes the system to the risk of chipping due to crack formation.^[30,31]

Moreover, controversy exists regarding the effect of coloring on flexural strength of zirconia. Ardlin^[32] showed that colored zirconia framework had higher flexural strength than uncolored zirconia. Hjerppe et al.[33] concluded that long-term immersion of yttria-stabilized zirconia (Y-TZP) discs in coloring solutions significantly decreased their biaxial flexural strength while Pittayachawan et al.[34] found no significant effect of coloring agents on flexural strength of zirconia. Aboushelib et al.[35] indicated that type of zirconia had a significant effect on bond strength of core to the veneering porcelain, and bond strength of colored zirconia was found to be significantly lower than that of white zirconia. Another study reported that type of zirconia (white or colored) had no significant effect on tensile bond strength.^[36]

However, selection of the cooling protocol of ceramic can potentially increase or decrease the internal stresses of porcelain and zirconia. Previous studies mostly focused on the effects of residual stresses, thermal compatibility, and phases of firing and mainly used conventional shear or tensile tests; however, these tests are incapable of assessing the mode of failure at the fracture surfaces and have limitations with regard to the unequal stress distribution, incompatibility in the modulus of elasticity, cracks formed during the manufacturing process, complexity of the applied load, and fractures before testing. Caputo *et al.*^[37] designed a modified flexural strength test aiming to eliminate the problems related to previous tests. The efficacy of the modified flexural strength test has been previously confirmed.^[38]

The aim of this study was to evaluate the effect of cooling protocol and zirconia coloring on fracture load of zirconia-based restorations.

The first null hypothesis was that the cooling protocol of ceramic would have no significant effect on the fracture load of zirconia-based restorations. The second null hypothesis was that coloring of zirconia would have no significant effect on fracture load of these restorations.

MATERIALS AND METHODS

This was an *in vitro*, experimental study. Table 1 shows the materials used in this study. It was approved by the Institutional Ethics Committee of the dental school Isfahan University of Medical Sciences (NO:395661).

Fabrication of zirconia bars

Forty bar-shaped samples (31 mm × 6.5 mm × 1.35 \pm 0.1 mm) were fabricated of nonsintere white zirconia blocks by CAD/CAM technology (imes-icore GmbH, Eiterfeld, Germany). Sample size was determined according to the previous studies.^[7] Half of the specimens were immersed in the coloring liquid for 2 min before sintering according to manufacturer's instruction. After removal, excess coloring liquid was removed by a paper towel and samples were allowed to dry. Thus, zirconia bars were divided into

Table 1: The materials used

Material	Commercial name	Chemical composition
Y-TZP ceramic	Vita- Ceram YZ (Vita Zahnfabric, Bad Sackingen, Germany)	Zirconia powder: Al_2O_3 (67%), ZrO ₂ (3%), Ce-stabilized. Zirconia glass powder: Al_2O_3 (14-18%), SiO ₂ (14-18%), B ₂ O ₃ (11-15%), TiO ₂ (2-7%), La ₂ O ₃ (25-30%), CeO ₂ (6-10%), CaO (4-8%), ZrO ₂ (1-4%), Y ₂ O ₃ (2-6%)
Feldspathic ceramic	Vita PM9, Translucent 2M2P-T (Vita Zahnfabric)	SiO ₂ (62-67%), Al ₂ O ₃ (16-19%), K ₂ O (6-8%), Na ₂ O (5-8%), B ₂ O ₃ (1-3%)
Coloring liquid	VITA-In-Ceram YZ Coloring liquid, medium (Vita Zahnfabric)	

two groups of white (w) and yellow (y) (n = 20). The sintering process was performed according to the manufacturer's instructions for all samples. Final dimensions of the samples were measured by a digital caliper (0.01 mm). No surface pretreatment was done on zirconia bars before porcelain application. Zirconia bars in the two groups were randomly divided into two subgroups for slow and rapid cooling (W-slow, W-fast, Y-slow and Y-fast, n = 10). Samples with dimensional changes more than ± 0.1 mm from mentioned dimensions were excluded from the study.

Application of the veneering porcelain

Heat-pressed ceramic was used to obtain an equal thickness of porcelain on the entire surfaces of all zirconia bars and eliminate the stress due to porcelain surface adjustment. First, bars were ultrasonically cleaned with 10% isopropyl alcohol for 10 min. Then, a wax cube 6.5 mm \times 6.5 mm \times 2 mm was waxed up on the bars using a silicon index to reach same shape and size of porcelain cubes, and then, invested in a ring as recommended by the manufacturer (PM Investment Material, Vita Zahnfabric, Bad Sackingen, Germany). Before wax up, each zirconia bar was weighed. After wax up, wax sprues with 3-8 mm length and a minimum of 4 mm diameter were attached to wax cubes and weighed. The difference in weight of zirconia bars before and after wax up was used to determine the required amount of PM9 porcelain. Bars were placed in the ring in such a way to have 10 mm distance from the ring walls. Rings were placed in an oven (Kavo EWL type 5615, Elektrotechnisches Werk GmbH, Germany) to eliminate the wax. The temperature and time were adjusted based on the ring's weight and manufacturer's instructions. Rings were immediately transferred from the preheating furnace to the press oven. The ceramic was injected into the mold as recommended by the manufacturer. Then, the ceramic bars were cleaned with airborne-particle abrasion using 50 µm aluminum oxide particles. Final dimensions were measured by a digital caliper.

Cooling protocols

The final firing cycle of samples was performed in a press oven. For fast cooling, the investment rings were immediately removed from the oven and placed on a grid for a minimum of 1 h to reach room temperature as recommended by the manufacturer. For slow cooling, samples were placed in a furnace with its door open by 30% for 15 min to reach a temperature of 500°C. Then, they were removed from the furnace and were allowed to cool down to room temperature for 1 h.

Aging procedure

The specimens were subjected to thermocycling for 5000 cycles between 5° C and 55° C with a dwell time of 30 s.

Four-point flexural strength test

The modified four-point flexural strength test^[37] was used. In this test, porcelain is attached to the bar between two load application points (in areas where the load is greater). Based on previous studies, bond failure most probably occurs due to tensile loads.^[38] Samples were placed in a metal holder for conduction of four-point flexural strength test in such a way that the veneering porcelain was subjected to tensile stresses. For this purpose, 1000 kg load was applied by a universal testing machine) Electromechanical Universal Testing Machine, K21046, Walter + Bai, Switzerland) at a crosshead speed of 0.5 mm/min. The load at fracture (hearing the sound of it or observing a change in the load versus deflection curve) was recorded.

Assessment of the failure mode

The failure mode (adhesive/cohesive/mixed) was determined by a stereomicroscope with digital camera (Trinocular Zoom Stereomicroscope, SMP200, HP, USA).

Statistical analysis

Two-way and one-way ANOVA followed by Tukey honest significant difference test were used for data analysis (α =0.05).

RESULTS

Effect of cooling protocols

The mean fracture load values in the four subgroups are presented in Table 2. The W-slow and the Y-fast groups had the highest and lowest fracture load values, respectively.

Two-way ANOVA showed that cooling protocol, coloring of zirconia significantly affected the fracture load of the specimens (P < 0.001). Considering the significant interaction effect of cooling protocol and coloring of zirconia (P = 0.007) [Table 3], one-way ANOVA was applied, which showed a significant difference among the four subgroups (P < 0.001). Tukey's test was applied for pairwise comparison of the groups [Table 2]. There was significant difference between W-slow and the other groups (P < 0.001)

Table 2: Mean and standard deviation of Fracture load values (N)

Studied	Mean±(SD)*	95% CI		
groups		Lower bound	Upper bound	
White-Slow	179.88 (23.43)ª	163.12	196.65	
White-Fast	126.50 (15.94) ^{b,c}	115.09	137.9	
Yellow-Slow	142.05 (23.98) ^b	133.19	144.9	
Yellow-Fast	117.50 (14.63)°	107.03	127.96	

*Different lowercase letters show significant difference between groups (P<0.05)

Table 3: Two-way ANOVA for the effects of cooling protocol and zirconia coloring on the Fracture load values (N) of specimens

Source	Type III sum of squares	df	Mean square	F	Significant
Corrected model	22750.169	3	7583.390	29.347	0.000
Intercept	800717.379	1	800717.379	3098.657	0.000
Color	5484.730	1	5484.730	21.225	0.000
Cooling	15186.219	1	15186.219	58.768	0.000
Color cooling*	2079.220	1	2079.220	8.046	0.007

but there was no significant difference between slow-yellow (P = 0.153) and fast – white, also between fast – yellow and fast – white (P = 0.599).

Failure mode

In all samples, failure occurred in a thin layer of porcelain adjacent to the zirconia–porcelain interface. Thus, cohesive failure occurred in 100% of the cases (within the porcelain adjacent to the interface) [Figure 1]. Porcelain segments remained intact in all specimens except for three in which, the porcelain margin slightly chipped.

DISCUSSION

In this study, the cooling protocol and coloring of zirconia significantly affected the fracture load of samples. Thus, the both null hypotheses were rejected.

In this study, slow cooling protocol yielded significantly higher fracture load values. Göstemeyer *et al.*,^[39] reported a reduction in bond strength of zirconia core and the veneering porcelain due to slow cooling. Fast cooling may increase the flexural strength of all-ceramic restorations because residual compressive stress develops in the surface of the veneering porcelain.^[27,39] However, several studies have emphasized on the correlation of residual tensile stresses in the veneering porcelain and fast cooling protocol of zirconia restorations.^[20,24,28,40] Choi



Figure 1: Cohesive failure occurred in 100% of the cases.

et al.^[41] evaluated the effect of cooling speed on the residual stress in pressed ceramics onto zirconia. They concluded that fast cooled samples had smaller cracks because the residual compressive stresses developed in the surface compensated for the tensile stresses created in the system; whereas, slow cooling in almost all samples eliminated the residual stresses in the pressed ceramics. In addition, they showed that slow cooled samples (leucite-containing ceramics such as Vita PM9 used in the current study) showed higher toughness than leucite-free ceramics.

When an all-ceramic restoration is rapidly cooled down, the superficial layer rapidly loses temperature and cools down; however, due to low thermal conductivity of porcelain, the underlying layers are still warm and slightly viscous. Thus, smaller thermal gradient in slow cooling protocol results in formation smaller amounts of residual tensile stresses within the material, which are probably compensated by the surface compressive stresses.^[22] Due to equal thermal conductivity and thickness of samples in the current study, the thermal gradient was probably influenced exclusively by the cooling protocol.

The protocols used in this study were similar to the slow and fast protocols described by Tan *et al.*^[7] They reported that the strength of samples that received slow heating and cooling was twice the value in samples that underwent fast protocols. They recommended using slow heating and cooling protocols for zirconia-based restorations, which is in agreement with the current results. In contrast, Göstemeyer*et al.*^[39] used fast and slow cooling protocols and reported that slow cooling between the porcelain sintering temperature and glass transition

temperature may increase the risk of adhesive failure. This difference between our results and those of Tan *et al.*^[7] with the findings of Göstemeyer *et al.*^[39] may be due to the difference in cooling protocols and the materials used. In the slow cooling protocol by Göstemeyer *et al.*^[39] samples were cooled from the firing temperature to glass transition temperature within 5 min, which does not seem to differ much from the fast removal of samples from the oven.

In the current study, the four-point flexural strength test was used instead of the three-point test because the former provides a more controllable environment for assessment of the mechanical properties of the ceramics.^[42] The lower values obtained in the four-point test compared to the three-point test^[43] may be closer to the actual values, especially because the area under tension has increased, which increases the risk of localization of cracks in this area.^[44] In the modified four-point test in particular, the location of failure is predicted to be beneath the load application line. However, the most important drawback of this test is absence of a statistical method to calculate the bond strength and quantify the effect of basic properties of materials such as modulus of elasticity and Poisson's ratio on the results.^[38]

However, the mean bond strength values obtained in our study were higher than values obtained by Tan *et al.*^[7] Although their methodology was similar to ours, they did not perform thermocycling.

In previous studies, a significant reduction in flexural strength was noted in water compared to the dry environment,^[45-47] which may be due to ceramic corrosion by the water molecules.^[46] For this reason, we first performed thermocycling and then tested the samples.

Although it may seem that the slow cooling protocol is more time consuming, it is probably worth it since it decreases the risk of porcelain fracture and subsequent complications.

The results showed significant effect of coloring on fracture load of samples since white samples had a significantly higher fracture load than colored samples (P < 0.001). No consensus has been reached on the effect of coloring on bond strength. Lima *et al.*^[48] showed that use of surface agents to improve the color and enhance the bond between the zirconia framework and porcelain had no significant effect on flexural strength and failure mode of two-layer samples.

Another study reported that long-term immersion in coloring agents decreased the strength of Y-TZP ceramics.^[33] It seems that presence of water in surface agents results in higher phase transformation of zirconia in contact with the porcelain, which is responsible for future fractures.^[49] However, another study using energy dispersive X-ray spectroscopy showed no phase transformation after immersion of samples in the coloring solutions.^[50] One study even reported an increase in flexural strength after coloring.^[34] The controversy in this regard in the results of previous studies is probably due to the type of the layering porcelain and probably different coefficients of thermal expansion between layering porcelain used in these researches.

During flexural strength testing, all porcelain fragments were separated from the zirconia bars at a site close to the interface. A thin layer of porcelain remained on all bars. Thus, it seems that potential internal stress is present in the porcelain adjacent to the interface and the porcelain-zirconia bond is adequately strong. Porcelain segments remained sound in all except for three samples in which porcelain margin slightly chipped. It indicates stress accumulation in the relatively sharp margins. Rismanchian et al.[51] evaluate shear bond strength of two veneering ceramics to zirconia (Biodenta and Cercon specimens) and reported the fracture mode of these two systems were different. Since Biodenta fracture pattern was predominantly adhesive, it seems that maybe Biodenta porcelain was stronger than Cercon porcelain where as its adhesive bond was weaker. Therefore, it may be concluded that the type of failure is largely influenced by the type of material which is used.

In slow cooling protocol applied in the current study, temperature was decreased to 500°C by adjusting the oven temperature. However, this temperature does not necessarily indicate the temperature of the samples at all areas. Another limitation of this study was that the composition of coloring agent was not known. Modified flexural strength test was selected for this study due to its advantages and since it is a reliable test to compare the mechanical behavior of fragile materials. However, this test cannot completely simulate the clinical setting and long-term clinical studies on samples with anatomical forms must be performed.

CONCLUSION

Within the limitations of this study, these findings can be drawn:

- 1. Slow cooling protocol can improve the fracture load of zirconia-based restorations
- 2. Coloring of zirconia has an adversely effect on their fracture load.

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Conflicts of interest

There are no conflicts of interest.

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