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Effect of Physical and Physicochemical Surface Treatment Methods on the Tensile Strength of CAD/CAM-Fabricated Zirconia Posts and Cores Luted to Root Canals

Cumhur Sipahi, DDS, PhD/Fulya Toksoy, DDS, PhD***
*Simel Ayyıldız, DDS, PhD***/Mutlu Özcan, Dr Med Dent, PhD*****

This study investigated the effect of surface treatments on the retentive strength of zirconia posts and cores to root canals. Maxillary central incisors (n = 40) were treated endodontically, obturated, and post spaces were prepared. Zirconia posts and cores (n = 40) were obtained and assigned randomly to four groups (n = 10 per group; control, sandblasting, tribochemical silica coating, and tribochemical silica coating + silanization). Posts were cemented adhesively, and tensile force was applied. All treatment methods increased the tensile strength of zirconia posts and cores compared to the control group (P = .034). No significant difference was found between treatment methods. Failure types varied depending on the surface treatment method. (Int J Periodontics Restorative Dent 2011;31:e64–e70.)

*Associate Professor and Head, Department of Prosthodontics, Academic Center for Dental Sciences, Gülhane Military Medical Academy, Ankara, Turkey.
**Assistant Professor, Department of Endodontics, Academic Center for Dental Sciences, Gülhane Military Medical Academy, Ankara, Turkey.
***Assistant Professor, Department of Prosthodontics, Academic Center for Dental Sciences, Gülhane Military Medical Academy, Ankara, Turkey.
****Professor and Head of Dental Materials Unit, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Zurich, Switzerland.

Correspondance to: Dr Cumhur Sipahi, Department of Prosthodontics, Gülhane Military Medical Academy, Academic Center for Dental Sciences, Gn.Tevfik Sağlam Cad, Etlik, Ankara, Turkey; fax: +90 312.304 60 20; email: cumhursipahi@yahoo.com.

The increasing demand for more esthetic and biocompatible restorations has led to the development of tooth-colored, metal-free post and core systems. Recently, zirconium dioxide ceramics (zirconia) have become available and popular in the field of dental prosthetics to fulfill these demands.[1–3] Prefabricated zirconia posts are advocated to be used with heat-pressed glass ceramic (Empress Cosmo, Ivoclar) or with composite resin core materials.[4–6] For uniform and circular-shaped root canals with sufficient bulk of dentinal walls, prefabricated zirconia posts display intimate adaptation to prepared canal walls along their entire length. However, in wide, noncircular, or extremely tapered canals, prefabricated cylindric zirconia posts may not achieve such an intimate adaptation to the canal, possibly compromising the retention of the post and the crown. In such cases, custom-made ceramic posts and cores can be fabricated with presintered zirconia blocks using copy milling or computer-aided design/computer-assisted manufacturing (CAD/CAM) techniques.[7–10]

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Combined with CAD/CAM technology, the fabrication of complex restorations incorporating zirconia cores has become a relatively simple procedure. On the other hand, establishing a durable chemical or mechanical bond with zirconia has proven to be difficult. Since the material is acid resistant, it does not respond to acid etching and silanization procedures used for glass-containing ceramic materials that could react with silane coupling agents.[11–13]

Several surface-roughening and coating methods have been proposed to condition zirconia surfaces to optimize the adhesion of resin-based cements.[14,15] Unfortunately, significant improvement in adhesion has not been achieved to date. Besides mechanical retention, one other effective method to achieve chemical adhesion to zirconia is the use of a phosphate ester monomer (10-methacryloxydecyl dihydrogen phosphate [MDP]). Although it was claimed to provide a stable bond that resisted hydrolysis during different artificial aging procedures, this bond was not sufficient for the retention of zirconia restorations, resulting in debonding under function.[14,15] On the other hand, some studies have shown that silica coating followed by silanization could be an alternative conditioning method to improve the adhesion of resin-based luting agents to zirconia.[16–19] Özcan and Vallittu[17] demonstrated that tribochemical silica coating procedures significantly increased the bond strength for zirconia compared to that of airborne-particle abrasion using alumina. Bottino et al[18] reported that tribochemical silica coating systems increased the tensile bond strength of MDP containing resin cement (Panavia F2.0, Kuraray) and zirconia ceramic. Also, Atsu et al[19] found increased shear bond strength between zirconia and a resin luting agent after tribochemical silica coating in combination with an MDP-containing bonding/silane coupling agent mixture. In fact, the MDP monomer presents bifunctional groups and does not require the use of a silane coupling agent for luting. While the rationale for the use of air abrasion methods is to increase surface energy and roughness, in addition to its surface cleaning effect, silanes are suggested to be used to increase surface wettability and to form covalent bridges between the resin and hydroxyl groups on the zirconia. In that respect, it can be hypothesized that as a result of copolymerization between MDP containing resin cement and tribochemically coated and silaned surfaces,[19] improved adhesion and, thereby, retention of the zirconia posts in the root canals could be expected. It can also be hypothesized that silanization would increase adhesion of resin cements compared to air-abrasion procedures alone.

Although there are numerous studies evaluating the effect of tribochemical silica coating and silanization on zirconia, there is sparse data in the literature about CAD/CAM-fabricated zirconia posts and cores and their retentive strength to root canals after such procedures. The aim of the present in vitro study was to determine the effect of tribochemical silica coating followed by silanization and sandblasting on the retentive strength of CAD/CAM-fabricated zirconia posts and cores to prepared root canals compared to a control group.

Method and materials

The coronal aspects of 40 extracted maxillary central incisors were resected perpendicular to their long axes, instrumented endodontically, obturated, and embedded in acrylic resin blocks. Post spaces (1.5-mm diameter, 10-mm length) were prepared with progressively enlarging drills (Gates-Glidden). Final enlargements were accomplished using a 1.5-mm diameter drill (ParaPost no. 4, Coltene/Whaledent) to a depth of 10 mm.

Dimensionally stable autopolymerizing acrylic resin (Palavit G, Heraeus Kulzer) was injected into each post space. Canal reamers (no. 30) were introduced and centrally positioned into the post spaces filled with acrylic resin before the completion of polymerization. After polymerization, the outer portions of the reamers were cut transversally at a level 4 mm distant from the sectioned root surfaces so as to provide 4-mm projected tips.

To obtain standard cores, open-ended, semiconical, hollow brass matrices (6 mm high, 4-mm diameter at bottom, 5-mm diameter at

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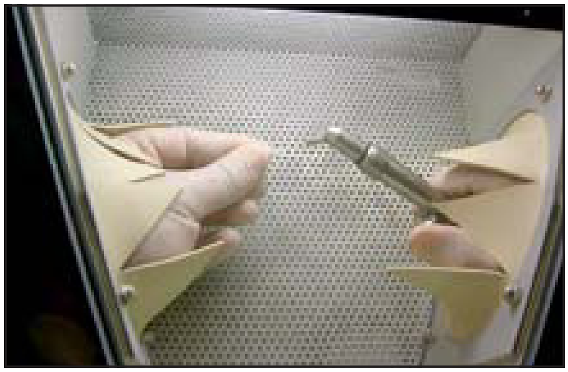

Fig 1 (left) *CAD/CAM-fabricated zirconia post and core.*

Fig 2 (right) *Particle abrasion of zirconia posts and cores in a closed chamber.*

top) were cast and fit flush to sectioned root surfaces so that the projected reamer tips remained at the center of the matrices secured onto sectioned root surfaces with sticky wax. Additional dimensonally stable autopolymerizing acrylic resin was injected into the matrices in a way that the projected reamer tips remained in the acrylic resin bulk. After polymerization, acrylic resin foundations were retrieved from the post spaces, the brass matrices were releaved from the acrylic surfaces, and 40 directly fabricated acrylic resin post and core patterns were obtained.

The 40 acrylic resin post and core patterns were transferred to a dental laboratory to be duplicated to zirconia post and core foundations with use of the CAD/CAM technique. A three-dimensional optical laser scanner (Dental Wings) was used to scan the patterns. Following scanning, presintered zirconia blocks (Whitepeaks) were placed into the computer numeric controlled milling machine (Yena-Dent, D-30), and the blocks were milled according to data provided by the scanner. After completion of milling, the specimens were subjected to a final sintering procedure, and 40 zirconia post and core specimens were obtained (Fig 1).

Obtained zirconia post and core specimens were assigned to four experimental groups (n = 10). Group CON served as the control and consisted of zirconia post and core specimens cemented without being subjected to any surface treatment procedure. Group SBL consisted of zirconia post and core specimens cemented after being subjected to sandblasting with alumina particles, group TSC consisted of zirconia post and core specimens cemented after being subjected to tribochemical silica coating, and group TSCS consisted of zirconia post and core specimens cemented after being subjected to tribochemical silica coating followed by silanization.

The inner surfaces of all prepared root canal post spaces were etched with 37% orthophosphoric acid (Preparator, Ivoclar Vivadent) for 20 seconds. Etched post space surfaces were rinsed with distilled water thoroughly and dried with air spray for 10 seconds.

Specimen surfaces from group SBL were subjected to sandblasting using 50-μm alumina particles. The procedure was applied perpendicular to the specimen surfaces from a distance of 10 mm under 2.5-bar pressure for 15 seconds. Specimens of groups TSC and TSCS were subjected to a tribochemical silica coating procedure with 30-μm silicon dioxide particles (CoJet Sand, 3M ESPE) using an airborne-particle abrasion device (CoJet System, 3M ESPE). Silica particles were de-

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Table 1 Descriptive statistics of tested groups

		Tensile strength (N)			95% confidence interval for mean			
	n	Mean	SD	SE	Lower bound	Upper bound	Minimum	Maximum
CON	10	70.5	31.9	10.1	47.6	93.3	36.0	118.0
SBL	10	79.2	9.8	3.1	72.1	86.2	67.0	91.0
TSC	10	97.3	24.2	7.6	79.9	114.6	62.0	134.0
TSCS	10	105.8	28.1	8.9	85.7	125.9	61.0	138.0
Total	40	88.3	27.9	4.4	79.3	97.2	36.0	138.0

SD = standard deviation; SE = standard error.

posited perpendicular to specimen surfaces from a distance of 10 mm under 2.5-bar pressure for 30 seconds (Fig 2). In Group TSCS, the silane coupling agent (Clearfil Porcelain Bond Activator, Kuraray Medical) was gently applied on specimen surfaces with cotton pellets, and silane was allowed to react on the posts for 5 minutes.

A phosphate monomer containing dual-polymerized self-adhesive resin cement (Clearfil SA Cement, Kuraray) was introduced into the post spaces in the canal using syringe endotips. Each zirconia post and core was inserted into its relevant canal post space, and excessive cement was removed with cotton rolls. A constant compressive force of 1 kg was applied onto zirconia specimens for 1 minute, and the resin luting agent was polymerized from mesial, distal, buccal, and palatal aspects of the roots for 40 seconds using a light source (Heliolux DLX, Ivoclar Vivadent) with a polymerization light intensity of 500 mW/cm^2.

Cemented specimens were subjected to thermocycling for 5,000 cycles between 5°C and 55°C, with a dwell time of 30 seconds at each temperature.

Following thermocycling, zirconia post and core specimens were mounted parallel along their long axes on a universal testing machine (Instron 1195, Instron), and axial tensile force was applied at a crosshead speed of 0.5 mm/min until dislodgement. Peak force values (N) obtained at the time of dislodgement were recorded. Failure types were classified as cohesive, adhesive between the dentin-resin interface, and adhesive between the resin-post interface.

Tensile force data (N) were analyzed using a statistical software program (SPSS 9.0 for Windows, IBM). The Kruskal-Wallis test was used to determine the probable difference between the control and experimental groups. The Bonferroni-corrected Mann-Whitney *U* test was then used to compare differences between experimental groups.

Results

Descriptive statistics (minimum, maximum, mean values, and standard deviations) of the tested groups are presented in Table 1 and Fig 3. The modes of failure are depicted in Table 2. According to the results of the Kruskal-Wallis test, significant differences were found between the experimental groups and the control group ($P = .034$). The Bonferroni-corrected Mann-Whitney *U* test showed no significant differences between the experimental groups (TSC-SBL, $P = .130$; TSC-TSCS, $P = .406$; TSCS-SBL, $P = .059$).

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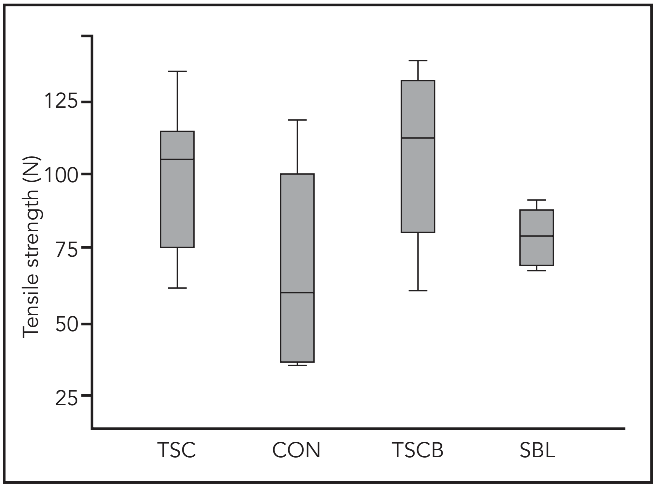

Fig 3 *Boxplot of mean tensile strength per experimental group.*

Table 2 Distribution of failure types observed per experimental group after tensile strength test

	Cohesive in the zirconia	Adhesive: Dentin-resin interface	Adhesive: Resin-post interface
CON (n = 10)	0	3	7
SBL (n = 10)	0	5	5
TSC (n = 10)	0	6	4
TSCS (n = 10)	2	7	1

Discussion

Since no significant differences were found between mean tensile strengths of zirconia posts after various conditioning methods, the hypothesis that tribochemical silica coating combined with silanization would increase the tensile strength of zirconia posts and cores to dentinal surfaces could be rejected. Since the air-abraded groups showed no significant difference with or without silanization, no effect of silanization could be verified, rejecting the second hypothesis. The TSCS group displayed significantly higher retentive force values compared to the untreated control group, supporting the findings of Özcan and Vallittu,[17] Bottino et al,[18] and Atsu et al.[19]

However, contrary to the findings of Özcan and Vallittu,[17] the sandblasting procedure alone (SBL) and the tribochemical silica coating procedure without silanization (TSC) were also significantly effective in increasing the tensile bond strength of zirconia posts and cores to root canal surfaces. This difference may originate from several factors. The first probable factor may be attributed to the variations in the test methods. In previous studies, only bond strength tests were employed to test the effect of conditioning methods on adhesion of resin cements where resin was adhered to flat zirconia surfaces.[17–19] In testing for tensile strength or retentive force required to detach the post from the root surface, in addition to frictional forces, the adhesion of the resin to the dentin walls comes into play. Conditioning root canal walls with 37% orthophosphoric acid may have provided a positive effect for increased tensile forces, regardless of the conditioning methods applied on the zirconia post surfaces.

In addition to the strength of the prosthetic material used, the cementation technique also is of importance for the clinical success of a restoration. Because of their high fracture resistance, zirconia-based restorations can be cemented using conventional luting agents.[20,21] However, resin bonding between tooth structure and the restoration is advocated for improving the retention, marginal adaptation, and fracture resistance of the restorations.[2] In the present study, a phosphate monomer containing dual-polymerized self-adhesive resin cement was used for the cementation of the posts. Since the self-adhesive resin cements have limited etching ability, the inner dentinal surfaces of prepared post spaces were subjected to acid etching to ensure increased surface area on dentin walls. Other clinical procedures than applied in this study, such as ethylenediaminetetraacetic acid sodium hypochloride application, may affect the outcome even further.

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The tensile strength values obtained in this study should be coupled with the failure types observed. The TSCS group was the only group that demonstrated cohesive failures in the post itself, with the lowest number of adhesive failures between the resin-post interface. Although the TSCS group did not show significantly high mean tensile strength compared to the SBL and TSC groups, the cohesive failure types indicate a strongly established physicochemical bond between the dentin/resin/zirconia interfaces exceeding the mechanical strength of the material. Seven adhesive failures between the dentin-resin interface in the TSCS group emphasizes improved adhesion between the resin-post interface exceeding that between the dentin-resin interface. When only failure types are considered, SBL and TSC showed similar failure types. Thus, it can be stated that additional application of silane in the TSCS group changed the failure types. Therefore, based on the failure types, the first hypothesis could be partially accepted.

Since chemical adhesion to zirconia is limited because of the inertness of the material, various surface treatment methods have been investigated, such as airborne-particle abrasion, acid etching, silica coating followed by silanization, and plasma spraying.[13,14,17] However, the main drawback of surface-roughening methods is that they may result in structural damage, material loss, grain pull-out,[5] and creation of sharp crack tips.[22,23] Thus, the bonded restoration may become more susceptible to radical cracking under functional loads.[22,23] The available current information on the possible aging effect of sandblasting protocols on zirconia surfaces is controversial. Although some studies clearly show the fatigue effect of sandblasting on zirconia in in vitro studies under chewing simulation devices,[22,23] others show no effect of aging.[24,25] Cyclic loading magnitudes, frequencies, and number of cycles applied in all of these studies are not possible to compare with one another. It should be noted, however, that the particle morphology shows great variability between ordinary alumina and silica-coated alumina.[26] This could be the reason why studies show controversial results related to the aging effect of sandblasting on zirconia. So far, no clinical study has proven that sandblasting yields to catastrophic fractures of zirconia frameworks. Nevertheless, a compromise needs to be found to achieve durable adhesion between the resin cements and zirconia through conditioning methods that do not impair the inherent strength of this material under chewing conditions.

Conclusion

All surface treatment methods increased the tensile bond strength of zirconia posts and cores luted to root canals. However, considering both the tensile strength results and the failure types, conditioning zirconia posts and cores with tribochemical silica coating followed by silanization showed more favorable results compared to silica coating or alumina sandblasting alone.

References

1. Hochman N, Zalkind M. New all-ceramic indirect post-and-core system. J Prosthet Dent 1999;81:625–629.
2. Koutayas SO, Kern M. All ceramic posts and cores: The state of the art. Quintessence Int 1999;30:383–392.
3. Michalakis KX, Hirayama H, Sfolkos J, Sfolkos K. Light transmission of posts and cores used for the anterior esthetic region. Int J Periodontics Restorative Dent 2004; 24:462–469.
4. Dilmener FT, Sipahi C, Dalkiz M. Resistance of three new esthetic post-and-core systems to compressive loading. J Prosthet Dent 2006;95:130–136.
5. Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. Biomaterials 1999;20: 1–25.
6. Jeong SM, Ludwig K, Kern M. Investigation of the fracture resistance of three types of zirconia posts in all-ceramic post-and-core restorations. Int J Prosthodont 2002;15:154–158.
7. Streacker AB, Geissberger M. The milled ceramic post and core: A functional and esthetic alternative. J Prosthet Dent 2007; 98:486–487.
8. Brittner N, Hill T, Randi A. Evaluation of a one-piece milled zirconia post and core with different post-and-core systems: An in vitro study. J Prosthet Dent 2010; 103:369–379.
9. Zhang YX, Zhang WH, Lu ZY, Wang KL. Fracture strength of custom-fabricated Celay all-ceramic post and core restored endodontically treated teeth. Chin Med J (Engl) 2006;19:1815–1820.
10. Awad MA, Marghalani TY. Fabrication of a custom-made ceramic post and core using CAD-CAM technology. J Prosthet Dent 2007;98:161–162.
11. Dérand P, Dérand T. Bond strength of luting cements to zirconium oxide ceramics. Int J Prosthodont 2000;13:131–135.
12. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: A review of the literature. J Prosthet Dent 2003;89:268–274.

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13. Aboushelib MN, Matinlinna JP, Salameh Z, Ounsi H. Innovations in bonding to zirconia-based materials: Part I. Dent Mater 2008;24:1268–1272.
14. Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. Int J Prosthodont 2002;15:267–272.
15. Amaral R, Özcan M, Bottino MA, Valandro LF. Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning. Dent Mater 2006;22:283–290.
16. Xible AA, de Jesus Tavarez RR, Araujo CRP, Bonachela WC. Effect of silica coating and silanization on flexural and composite-resin bond strengths of zirconia posts: An in vitro study. J Prosthet Dent 2006;95:224–229.
17. Özcan M, Vallittu PK. Effect of surface conditioning methods on the bond strength of luting cement to ceramics. Dent Mater 2003;19:725–731.
18. Bottino MA, Valandro LF, Scotti R, Buso L. Effect of surface treatments on the resin bond to zirconium-based ceramic. Int J Prosthodont 2005;18:60–65.
19. Atsu SS, Kılıçarslan MA, Küçükeşmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. J Prosthet Dent 2006;95:430–436.
20. Dündar M, Özcan M, Cömlekoğlu E, Güngör MA. A preliminary report on short-term clinical outcomes of three-unit resin-bonded fixed prostheses using two adhesive cements and surface conditioning combinations. Int J Prosthodont 2010;23:353–360.
21. Edelhoff D, Özcan M. To what extent does the longevity of fixed dental prostheses depend on the function of cement? Working Group 4: Cementation. Clin Oral Implants Res 2007;18(suppl 3):193–204.
22. Blatz MB, Phark JH, Ozer F, et al. In vitro comparative bond strength of contemporary self-adhesive resin cements to zirconium oxide ceramic with and without air-particle abrasion. Clin Oral Investig 2010;14:187–192.
23. Zhang Y, Lawn BR, Malament KA, Van Thompson P, Rekow ED. Damage accumulation and fatigue life of particle-abraded ceramics. Int J Prosthodont 2006;19:442–448.
24. Itinoche K, Özcan M, Bottino MA, Oyafuso DK. Effect of mechanical cycling on the flexural strength of densely sintered ceramics. Dent Mater 2006;22:1029–1034.
25. Scherrer SS, Cattani-Lorente M, Vittecoq E, de Mestral F, Griggs JA, Wiskott HW. Fatigue behavior in water of Y-TZP zirconia ceramics after abrasion with 30 μm silica-coated alumina particles. Dent Mater 2011;27:e28–e42.
26. Özcan M, Lassila LVL, Raadschelders J, Matinlinna JP, Vallittu PK. Effect of some parameters on silica-deposition on a zirconia ceramic [abstract 545]. J Dent Res 2005;(spec issue A):84.