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RESEARCH ARTICLE

RETENTIVE BOND STRENGTH OF DIFFERENT LUTING CEMENTS TO ZIRCONIUM OXIDE CERAMIC CROWNS: AN IN VITRO STUDY

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ABSTRACT

Aim: This study was undertaken to evaluate the bond strength of different luting cements to zirconium oxide ceramic crowns.

Materials and Methods: Thirty extracted human premolar teeth were prepared and used in the study for fabrication of zirconium oxide ceramic copings. These copings were then divided into three groups: - Group I (GC Gold Label), Group II (Calibra) and Group III (Panavia F 2.0), which consisted of ten copings each for cementation. The samples were then subjected to a crosshead speed of 0.5mm/min for dislodgement of copings along the apico-occlusal axis until failure on Universal testing machine. The data obtained was subjected to statistical analysis.

Statistical Analysis: The statistical tests used were ANOVA test for difference between the mean values and chi-square test for difference between the proportions. The level of significance was taken at 5% ($p<0.05$). The F-test is used for comparisons of the components of the total deviation Bonferroni test is used for multiple comparisons.

Result: The result showed that the mean retentive force for Group I cement was 126.9 ± 31.52, for Group II cement was 256 ± 58.04 and for Group III cement was 271.4 ± 51.15. Group I cement had the least retentive strength for retaining zirconium oxide copings.

Conclusion: It was concluded that Groups II and III are capable of retaining zirconium oxide copings successfully with no additional internal surface treatment other than airborne-particle abrasion with 50-µm aluminium oxide.

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INTRODUCTION

As art is the disposition of intelligible matter, regarding esthetics dentistry is no exception to it. The desire for fixed replacement of fractured, discoloured or missing teeth with esthetics simulating that of natural teeth has been influenced by advancements of material science and technology. Till date, dental profession is continuously facing the challenge to improve both the mechanical as well as the optical properties for ultimate esthetics and function. Situations like high demand for anterior esthetics, metal allergies and thin biotype constraints led to the discovery of metal free ceramics. Metal free ceramics provide superior esthetics because they allow diffuse transmission of light, reproducing a depth of colour and translucency, which minimizes gingival shadowing and yields an appearance of vitality (Tan and Dunne, 2004). Different types of ceramics have emerged in modern times withoptical properties like glass ceramics, silica based ceramics, lithium disilicate ceramics and most recently zirconium oxide ceramics. Zirconium oxide was identified in 1789 by German chemist Martin Heinrich Klaproth (Piconi and Maccauro, 1999). Zirconium oxide ceramics have several advantages over other ceramic materials as they undergo transformation toughening mechanism, operating in their microstructure which gives zirconium oxide ceramics its acceptable properties. Zirconium oxide ceramics provide unsurpassed esthetics, biocompatibility, high flexural strength, toughness, and desirable optical properties (Atsu *et al*., 2006). The success of fixed restoration not only depends on the type of ceramic or core material but also on the choice of cementing media. A strong durable bond is required to prevent microleakage, to increase the fracture resistance of the restored tooth or restoration, to provide good marginal adaptation and retention (Blatz *et al*., 2004). The selection of cementing media is a challenging task. Complete coverage zirconium oxide based crowns, due to high fracture resistance can be cemented using conventional methods (Atsu *et al*., 2006). The silica based ceramics can be etched by hydrofluoric acid (Blatz *et al*., 2004) and bonded by Bis-GMA based resin luting agents.

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However, it is difficult to modify the surface of zirconium oxide ceramic for the purpose of cementation because of its matrix being resistant to etching (Palacios *et al*., 2006). The aim of the study was to evaluate the retentive bond strength between three luting cements (GC Gold Label, Calibra and Panavia F 2.0) and their effect on the retention of zirconium oxide ceramic copings, to analyze whether resin luting agents are superior to conventional cements for retention of zirconium oxide ceramic copings and to determine the mode of failure for each cement group.

MATERIALS AND METHODS

The present in vitro study was conducted in the Department of Prosthodontics including Crown and Bridge and Implantology, Institute of Dental Studies and Technologies, Modinagar, Uttar Pradesh and in collaboration with Images Dental Private Limited, New Delhi and Department of Mechanical Engineering, I.T.S. College of Engineering, Greater Noida to evaluate the bond strength of different luting cements to zirconium oxide ceramic crown. Thirty human premolar teeth, indicated for extraction for orthodontic purposes were included in the study. Criteria for selection of extracted teeth for this study were: teeth free from caries or previous restoration and sound permanent premolars with normal crown anatomy and of almost similar size and shape as far as possible. The selected extracted teeth were used for the fabrication of zirconium oxide copings which were then divided into three Groups: - Group I, conventional glass ionomer cement (GC Gold Label), Group II, resin cement (Calibra) and Group III, resin cement containing adhesive phosphate monomer (Panavia F 2.0), which consisted of ten copings each for cementation.

The methods used for study was divided under following headings

Preparation of teeth for coping fabrication: Thirty freshly extracted non carious, human permanent premolar teeth were collected and were cleaned by placing them in 1% hydrogen peroxide and were subsequently washed with distilled water. The teeth were stored in normal saline at room temperature. The roots of teeth were then notched with diamond disc so that acrylic resin can bond mechanically to the teeth, resisting their removal from acrylic resin while tensile testing. Teeth were then embedded in splitted halves of PVC ring (2 inches in length and 1 inch in diameter) using autopolymerizing acrylic resin with the cementoenamel junction positioned 1mm above the PVC ring. The split halves of PVC ring facilitate their removal after auto polymerizing resin achieves dough stage. Then, the acrylic blocks were finished using acrylic trimming burs and polishing agents. The occlusal surface was sectioned flat 4 mm above the top of the acrylic resin. Gross reduction of teeth was done using air rotor handpiece. A high speed air motor hand piece was secured in a milling machine for final tooth preparation. The samples were then mounted in dental stone, using a base former. The mounted tooth assembly was kept on the platform of the milling machine (Figure 1) and was tilted at an angle of 10 degree which was determined using protractor. This was done so that a standardized angle of convergence of 20 degree was achieved (Figure 2). With the hand piece rigidly secured, the axial surface was prepared by rotating the platform against the diamond bur so that preparation can be standardized. Using

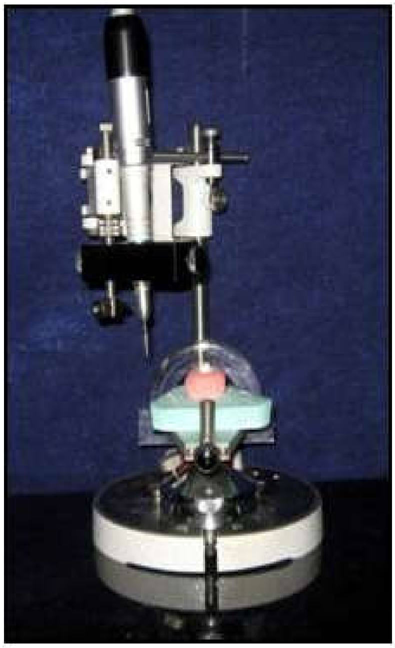

Figure 1. Milling machine attached with a hand piece and mounted tooth assembly for preparation of teeth

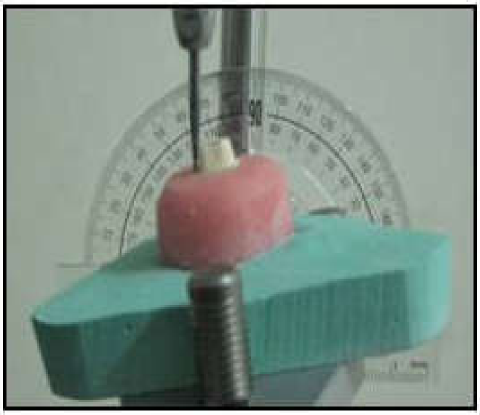

Figure 2. Mounted tooth assembly on platform of milling machine tilted to achieve convergence of 20 degrees

Figure 3. Wire passed through hole in the coping and attached to the upper apparatus of the Universal Testing Machine

water spray, the axial surface was reduced to a depth of 1mm. The preparation when viewed from the occlusal view was oval in shape, with a longitudinal diameter approximately 11 mm and transverse diameter 6 mm. A taper of 10 degree was automatically incorporated into the preparation. Finally, the preparation was refined using finishing burs.

Figure 4. Specimen with crosshead being moved

Figure 5. Debonded specimen

Preparation of zirconia copings: The prepared teeth were then used for the fabrication of zirconia copings in the dental laboratory. The teeth were placed in Cercon Eye (Dentsply, USA) and scanned. The data was transferred to Cercon Brain for milling through computer. The copings were milled from the commercially available zirconia blank using Cercon milling machine. The milled coping was then kept in Cercon Heat. The overall thickness of the zirconium oxide coping was 0.6 mm except at the occlusal aspect. The thickness of the zirconium oxide coping was increased occlusally to 4 mm so that a hole, 1mm in diameter was drilled. This hole served as a connection between the testing apparatus and the specimen.

Internal surface treatment of the zirconia copings: The intaglio surface of each coping was airborne-particle abraded with 50-µm aluminium oxide for a maximum of 15 seconds under 4- to 5- bar pressure using a sand blasting machine.

Cementation of zirconium oxide coping specimens: The liquid: powder ratio, mixing and manipulation of cements was carried out, according to the manufacturer's recommendations. The specimens were cemented under finger pressure (until the initial set of cement) to the prepared tooth surface with Group I, Group II and Group III cements. Excess cement was carefully removed. The full seating of the coping was confirmed by intimate contact of the coping to the tooth surface. After final setting of the cements, the cemented samples were stored in normal saline for 24 hours at room temperature.

Evaluation of retentive bond strength and mode of failure

After 24 hours the specimens were placed on the platform of Universal Testing Machine. An orthodontic wire of 23 gauge was passed through the hole drilled in the occlusal part of the cemented coping and this wire was attached to the opposing upper apparatus of the Universal Testing Machine with the help of especially fabricated wire locking device (Figure 3). The platform was stationary and the upper apparatus was moved at a constant speed for tensile testing. The specimens were subjected to a constant screw crosshead speed of 0.5 mm/min (Figure 4) for dislodgement of copings along the apico-occlusal axis until failure on Universal Testing Machine (Figure 5). The retentive force value of the specimens was directly obtained from the computer display attached to the machine in Newtons. The mode of failure of dislodged specimens was determined visually. For visual examination, the observations made were categorized into-

Adhesive failure - Cement principally on prepared tooth (>3/4 of axial surface) Cement principally on coping and tooth Cement principally on ceramic coping (>3/4 of axial surface)

Cohesive failure - Fracture of tooth or rootFracture of coping

Statistical analysis: The data was the statistical analysed using SPSS version 16.0. The values were represented in Number (n), Percentage (%) and Mean (υ). The statistical tests used were ANOVA test for difference between the mean values and chi-square test for difference between the proportions. The level of significance was taken at 5% ($p<0.05$). The F-test is used for comparisons of the components of the total deviation Bonferroni test is used for multiple comparisons.

RESULTS

Table 1 shows the mean value of retentive force (in Newtons) for each cement Group. The mean retentive force for Group I cement (GC Gold Label) was 126.9 ± 31.52, for Group II cement (Calibra) was 256 ± 58.04 and for Group III cement (Panavia F 2.0) was 271.4 ± 51.15. Table 2 shows one-way ANOVA results for retentive force between and within groups. The difference was found to be statistically significant since the p-value was < 0.05. Table 3 shows comparisons within the groups, which were done using Bonferroni Multiple Comparisons. The comparison of Group I (GC Gold Label) was done with Group II (Calibra) and Group III (Panavia F 2.0). The difference between Group I (GC Gold Label) and Group II (Calibra) was found to be statistically significant (p-value < 0.05) and the difference between Group I (GC Gold Label) and Group III (Panavia F 2.0) was also found to be statistically significant (p-value < 0.05). The comparison of Group II (Calibra) was done with Group I (GC Gold Label) and Group III (Panavia F 2.0). The difference between Group II (Calibra) and Group I (GC Gold Label) was found to be statistically significant (p-value < 0.05) but the difference

Table 1. Mean retentive force (in Newtons) for various cement Groups

Group No.	Cements	No. of pecimens	Mean	Standard deviation	Standard error
Group I	GC Gold Label	10	126.9	31.52	9.97
Group II	Calibra	10	256	58.04	18.36
Group III	Panavia F 2.0	10	271.4	51.15	16.17

Table 2. One-way ANOVA results for retentive force between and within cement Groups

Comparison	Sum of squares	df	Mean square	F	p-value
Between Groups	125947.400	2	62973.700	27.073	0.003*
Within Groups	62803.300	27	2326.048		
Total	188750.700	29			

* Significant

Table 3. Post Hoc Tests – Bonferroni Multiple Comparisons

Assigned Group	Group comparison	p-value significance
Group I	Group II	0.003*
(GC Gold Label)	Group III	0.003*
Group II (Calibra)	Group I	0.003*
	Group III	1.000
Group III	Group I	0.003*
(Panavia F 2.0)	Group II	1.000

* Significant

Table 4. Frequency of mode of failure with each cement Group

Mode of Failure	Group I	Group II	Group III	Total	Significance (p-value)
Cement principally on tooth	5 50.00%	1 10.00%	3 30.00%	9 30.00%	0.024*
Cement principally in coping	0 0.00%	6 60.00%	5 50.00%	11 36.70%	
Cement on tooth surface and in coping	3 30.00%	0 0.00%	0 0.00%	3 10.00%	
Tooth fracture/coping fracture/root fracture	2 20.00%	3 30.00%	2 20.00%	7 23.30%	
Total	10 100.00%	10 100.00%	10 100.00%	30 100.00%	

* Significant

between Group II (Calibra) and Group III (Panavia F2.0) was found to be statistically non significant (p-value > 0.05). The comparison of Group III (Panavia F 2.0) was done with Group I (GC Gold Label) and Group II (Calibra).The difference between Group III (Panavia F2.0) and Group I (GC Gold Label) was found to be statistically significant (p-value < 0.05) but the difference between Group III (Panavia F 2.0) and Group II (Calibra) was found to be statistically non significant (p-value > 0.05). Table 4 shows the frequency of mode of failure with each Group. The frequency of mode of failure with Group I (GC Gold Label) for cement principally on tooth surface was 5 (50.00%), for cement principally in coping was 0 (0.00%), for cement on tooth surface and coping was 3 (30.00%) and for tooth fracture, tooth removal or coping fracture was 2 (20.00%). The frequency of mode of failure with Group II (Calibra) for cement principally on tooth surface was 1 (10.00%), for cement principally in coping was 6 (60.00%), for cement on tooth surface and coping was 0 (0.00%) and for tooth fracture, tooth removal or coping fracture was 3 (30.00%). The frequency of mode of failure with Group III (Panavia F 2.0) for cement principally on tooth surface was 3 (30.00%), for cement principally in coping was 5 (50.00%), for cement on tooth surface and coping was 0 (0.00%) and for tooth fracture, tooth removal or coping fracture was 2 (30.00%). The frequency of mode of failure with Group I (GC Gold Label), Group II (Calibra) and Group III (Panavia F 2.0) was compared using the chi-square test. The difference was found to be statistically significant (p-value < 0.05).

DISCUSSION

All ceramic restorations are popular due to their outstanding esthetic qualities and metal free structure (Ernst *et al*., 2005). The attractive properties of zirconium oxide ceramics such as high strength, excellent mechanical properties and biocompatibility allow several applications in restorative dentistry, one of which is as a core material for all ceramic crowns and fixed partial dentures (Palacios *et al*., 2006). But their extensive use as an all ceramic restoration requires a reliable bond between zirconia and tooth structure. Keeping these facts in view, the present study was undertaken in vitro to evaluate the bond strength of different luting cements to zirconium oxide ceramic copings (Cercon). In order to carry out the present study, three brands of luting cements were procured from the market (GC Gold Label, Calibra and Panavia F 2.0). However, lack of retention is a common cause of fixed prosthesis failure. A reliable luting agent would therefore enhance fixed prosthodontic treatment (Rosenstiel *et al*., 1998). Therefore, retentive strength data are crucial for obtaining information about the potential clinical performance of luting cements for all-ceramic restorations (Ernst *et al*., 2005). In the study mean retentive force for Group I (GC Gold Label) and Group II (Calibra) was 126.9 ± 31.52 Newton and 256 ± 58.04 Newton respectively and the mean retentive force for Group III (Panavia F 2.0) was 271.4 ± 51.15 Newton. The results showed that the Group I (GC Gold Label) showed the least retentive strength values as compared to Group II (Calibra) and Group III (Panavia F 2.0) with Cercon zirconia ceramic. Heintze SD, reviewed various studies done to evaluate the effectiveness of luting agents on the retention of crowns and stated that resin-based luting agents such as Panavia resulted in a higher failure stress than glass ionomer cements (Heintze, 2010). Ernst CP also showed that the retentive strength values of Ketac cem Aplicap (glass ionomer cement) to zirconium oxide ceramic crowns was lowest among all resin luting agents and adhesives tested. Superbond C&B Monomer resin cement demonstrated highest median retentive strength but was not significantly different from Panavia, RelyX luting, Dyract Cem Plus and Rely X Unicem (Ernst *et al*., 2005). In the present study, results showed that the retentive bond strength values of copings cemented with Group II (Calibra) and Group III (Panavia F 2.0) had no significant difference. Palacios RP, also found that there was no significant difference in the retentive strength of the resin cements (Panavia F2.0 and Rely X Unicem) used for cementation of zirconia copings (Palacios *et al*., 2006). In this study, the teeth were prepared using dental rotary instruments, diamond burs. The teeth were prepared with flat occlusal surface and had an axial height of 4mm. The axial preparation was done to a depth of 1mm. The prepared teeth were then used for the fabrication of zirconium oxide ceramic copings. Palacios RP, determined the retention of zirconium oxide ceramic crowns with three luting agents and for the fabrication of these crowns, the occlusal surface of teeth was prepared flat with axial height 4mm however, the axial surface was reduced to a depth of 1-1.5 mm (Palacios *et al*., 2006). Although in the study done by Ernst CP, the occlusal surface of teeth was kept flat but the axial height was 3mm. The teeth were prepared for the fabrication of zirconia crowns to determine their retentive strength using different luting agents (Ernst *et al*., 2005).

Heintze SD, reviewed various studies on retentive strength of both all-ceramic and metal ceramic crowns and showed that the crown height varied between 3 mm and 6 mm and in almost all studies, the occlusal surface was trimmed flat (Heintze, 2010). In the present study, the convergence angle was kept 20 degrees. This degree of convergence was selected because a lower angle of draw may increase the retention and resistance to crown removal regardless of the type of cement, since it has been shown that retention increases exponentially as the taper decreases from 10 degrees (Palacios *et al*., 2006). The contribution of cement retention is better assessed with a 20 degrees convergence (Johnson *et al*., 2004) and was found to be a common clinical finding (Palacios *et al*., 2006). Palacios RP, in his study kept the convergence angle 20 degrees (Palacios *et al*., 2006). However, in the study done by Ernst CP a convergence angle of 10 degrees was used (Ernst *et al*., 2005). Heintze SD reviewed various studies done to evaluate the effectiveness of luting agents on the retention of crowns and found that the convergence angle varied between 4.8 degrees and 33 degrees (Heintze, 2010). In the present study, air-borne particle abrasion of the intaglio surface of the zirconium oxide copings was done with 50-µm aluminium oxide for 15 seconds under 4 bar pressure. Studies done by Blatz MB and Yang B, showed that air-abrasion significantly improved resin-zirconia ceramic bond strength and its durability by increasing the surface roughness, cleaning and activating the ceramic surface when combined with adhesive monomer containing primers such as 4-methacryloxyethyl-trimellitate-anhydride (4-META) or 10-methacryloxydecyl-dihydrogenphosphate (MDP) (10,11). Palacios RP, also used air-abrasion with 50-µm aluminium oxide for a maximum of 15 seconds under 4- to 5- bar pressure in his study (Palacios *et al*., 2006). Yang B, showed that regular pressure air-abrasion of zirconia ceramic at 0.25 MPa created greater surface roughness and larger bonding surface area than low pressure air-abrasion at 0.05 MPa or using no air-abrasion (Yang *et al*., 2010). In the present study, for cementation of zirconium copings to prepared teeth using different luting agents, finger tip pressure was applied. The pressure applied by Ernst CP during cementation was by hand (fingertip) without any kind of additional device (Ernst *et al*., 2005). Swift Jr EJ, in his study also applied finger tip pressure for cementation of silver-palladium alloy castings to prepared teeth (Swift *et al*., 1997). Tuntiprawon M also showed that the finger press was advocated for cementation of silver-palladium alloy crowns to prepared teeth in his study [Tuntiprawon, 1999]. Piemjai M concluded that the seating force had no influence on the failure stress when pulling off the crowns (Piemjai, 2001). In the present study, after cementation, a wire of 23 gauge was passed through the hole, drilled in the occlusal part of the coping. This was done to facilitate the tensile test. It served to provide a connection with the testing apparatus.

Zirconia has been shown to be a material with high fracture toughness so it did not result in fracture of the coping while testing. The crown retention test was more difficult for all ceramics because components allowing connection to the testing apparatus were not as easily integrated into all ceramic material. During testing, specimens were subjected to a tensile force at a crosshead speed of 0.5mm/min for dislodgement of the copings on Universal testing machine. Palacios RP also used a crosshead speed of 0.5mm/min for dislodgement of copings along the apico-occlusal axis until failure on Universal Testing Machine (Palacios *et al*., 2006). Johnson GH studied the effect of resin-based sealer on crown retention for three types of cement. In his study also for dislodgement of crowns, the crosshead speed used was 0.5 mm/min [Johnson *et al*., 2004]. Swift Jr EJ and Zidan O, in their studies also crowns were subjected to a crosshead speed of 0.5mm/min for dislodgement (Swift *et al*., 1997; Zidan and Ferguson, 2003). Heintze SD reviewed various studies in which crosshead speeds varied between 0.012 mm/min and 10 mm/min (Heintze, 2010). However, in the study done by Ernst CP, he used a crosshead speed of 10 mm/min[24] for dislodgement of zirconia crowns on Universal testing machine (Ernst *et al*., 2005). In the present study, we determined the mode of failure visually. Only the axial surfaces were evaluated to determine the type of failure, since the flat occlusal surface did not retain cement in most of the samples. Palacios RP also evaluated only the axial surfaces to determine the type of failure since no cement retained on the uniform occlusal surface in most specimens (Palacios *et al*., 2006). In the present study, visually for Group I (Gold Label), 50% of the specimens had cement principally on the tooth followed by 30% of the specimens with cement on both the tooth and in the copings. For Group II (Calibra), 60% of the specimens had cement principally in the copings followed by 10% specimens which had cement on the tooth surface. For the Group III (Panavia F 2.0), 50% of the specimens had cement principally in the copings, followed by 30% of the specimens with cement principally on the tooth.

However, in the study done by Palacios RP, he determined the failure type visually and showed that for the cement group, Panavia F 2.0, 54.5% of the specimens had cement in the copings followed by 36% of the specimens with cement principally on the tooth (Palacios *et al*., 2006). The crown pull off test was not only an intricate and time consuming test but also, a large number of parameters needed to be controlled and analyzed very carefully (Heintze, 2010). However, retention tests were important for determining clinical success of a fixed prosthesis. It has been documented that crown retention depends on many factors, such as taper, surface area of the preparations, internal surface roughness and type of cement. These factors may influence the stress distribution within the interposed cement layer, the efficiency of bonding of the cement to both surfaces being joined, and the durability of the cement including its long-term resistance to mechanical breakdown and/or dissolution. Luting cements are therefore, a critical but weak link between a fixed prosthesis (Ayad *et al*., 1998).

Conclusion

Within the limitations of this study, following conclusions were drawn: Group I (GC Gold Label) showed the least retentive strength values as compared to Group II (Calibra) and Group III (Panavia F 2.0) with Cercon zirconia ceramic. The retentive bond strength values of copings cemented with Group II (Calibra) and Group III (Panavia F 2.0) had no significant difference.

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