Research Article

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Comparative Evaluation of the Tensile Bond Strength of Cements Used for the Luting of Zirconia Crown Copings to Natural Teeth: An *In Vitro* Study

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ABSTRACT

Background: Bonding of zirconia crowns to the tooth and to the luting agent has been a topic of research since long time. Studies have been carried out in the past to evaluate the efficiency of conventional and adhesive luting agents for luting zirconia copings. But there was no unanimity in the conclusion. The present study was conducted in vitro for the comparative evaluation of the tensile bond strength of three cements namely zinc phosphate cement, glass ionomer cement and a self-adhesive resin cement used for the luting of zirconia copings to natural teeth and to characterize the mode of interfacial bond failure.

Materials and method: A total of thirty extracted human molar teeth were mounted in auto polymerizing acrylic resin blocks. They were divided into three groups of 10 samples each namely Group A- Zirconia copings to be luted with zinc phosphate cement, B- Zirconia copings to be luted with glass ionomer cement, C- Zirconia copings to be luted with self-adhesive resin cement. All the mounted teeth were prepared in a standardized manner to have a uniform configuration. Zirconia copings were then fabricated for each of the prepared tooth using ceramill CAD/CAM machine and luted with the respective luting agents. The samples were later subjected to tensile bond strength testing by universal testing machine. The results thus obtained were tabulated and subjected to statistical analysis by one-way analysis of variance (ANOVA) and post hoc tests. All the debonded samples were analyzed for their mode of failure using the criteria proposed by Johnson et al.

Results and Conclusion: On comparison of the mean tensile bond strength of the three different luting agents, it was revealed that self-adhesive resin cement Group C, mean 3.9676 ± 0.38 Mpa demonstrated statistically significant higher tensile bond strength than glass ionomer cement Group B, mean 1.04004 ± 0.19 Mpa and zinc phosphate cement, Group A, mean 0.69359 ± 0.18 Mpa. The mode of interfacial bond failure was found to be in accordance with the tensile bond strength values obtained for all the three groups. Self-adhesive resin cement showed a predominantly cohesive mode of failure which corresponds well with the highest bond strength obtained for that group. Within the limitations of the study, it was found that self-adhesive resin cement had the highest tensile bond strength and cohesive mode of bond failure.

Keywords: Luting of zirconia copings, Self-adhesive resin cement, Tensile bond strength, Mode of bond failure.

INTRODUCTION

Zirconia crowns can be cemented both conventionally and adhesively to the teeth by means of a number of luting agents available[1,2,3]. Zinc phosphate, glass ionomer cement are examples of the conventional luting agents whereas composite resin-based cements, self-adhesive resin cements

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are the adhesive ones. Shear, tensile, micro tensile and push-out tests have been advocated to evaluate the retentive capabilities of different luting agents on zirconia crowns[1,4]. While most them were laboratory studies involving bonding of flat zirconia and tooth surfaces, a few of them simulated clinical situations by testing full coverage zirconia crowns luted to extracted and prepared natural teeth. The results obtained from later type of studies involving full coverage crown are more indicative of the actual tensile bond strength exhibited by the luting agents under investigation[1]. Many investigators have employed various pre bonding surface treatments in an attempt to enhance the bond strength with conflicting results[5,6]. Most of these in-vitro investigations were done using various methodologies under different test conditions and there is no consensus regarding the choice of ideal luting material for zirconia crowns. Hence the present study was carried out with the objectives to evaluate the tensile bond strength of zinc phosphate cement, glass ionomer cement and self-adhesive resin cement used for the luting of zirconia copings to natural teeth and to characterize the mode of failure of zirconia copings luted to natural teeth with zinc phosphate cement, glass ionomer cement and self-adhesive resin cement.

MATERIALS AND METHOD

Freshly extracted maxillary or mandibular molar teeth with clinical crown height of minimum 6mm and diameter 10mm were selected and stored in distilled water at room temperature (Fig.1). Teeth with caries, restorations, hypoplasia and fracture were excluded. The teeth were randomly selected, numbered from 1 to 30 and divided into 3 groups of 10 each namely Group A, 1 to 10 - teeth designated for fabrication of zirconia copings to be luted with zinc phosphate cement, Group B, 11 to 20 - teeth designated for fabrication of zirconia copings to be luted with glass ionomer cement and Group C, 21 to 30 - teeth designated for fabrication of zirconia copings to be luted with self-adhesive resin cement. A customized hollow two-piece re-mountable stainless-steel cubical mold was fabricated for mounting the extracted teeth in auto polymerizing resin (DPI-RR cold cure) (Fig.2). The mounting was done with the help of dental surveyor and spirit level indicator (JON bharandi tools, Chennai). Total 30 of such mounted blocks were obtained in a similar manner (Fig.3) and stored in distilled water at 37±1^{0}C.

Fig 1: Extracted human tooth.

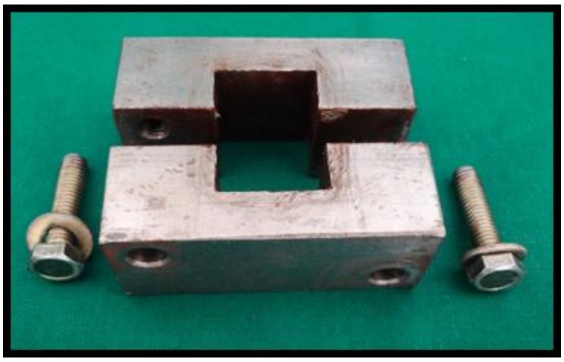


Fig 2: Stainless steel mold for mounting.

Fig 3: Mounting of teeth in auto polymerizing resin.

Fig 4: Preparation of natural tooth using precision milling lathe.

Fig 5: Prepared natural tooth.

Fig 6: Fabrication of special tray.

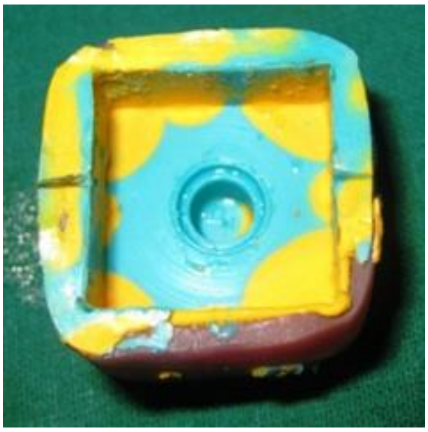


Fig 7: Making of impression.

Fig 8: Pouring of dies.

Fig 9: Fabrication of Zirconia coping.

Fig 10: Fabrication of Zirconia copings for all the samples.

Fig 11: Luting with zinc Phosphate Cement.

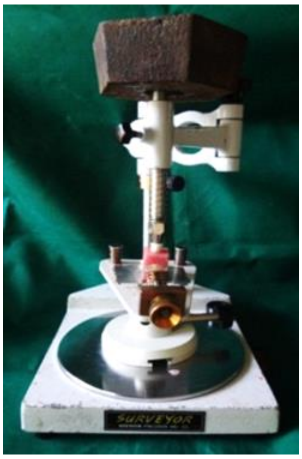


Fig 12: Luting with glass ionomer Cement.

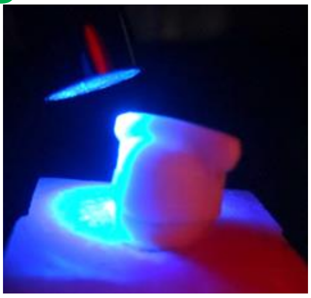


Fig 13: Luting with self adhesive resin cement.

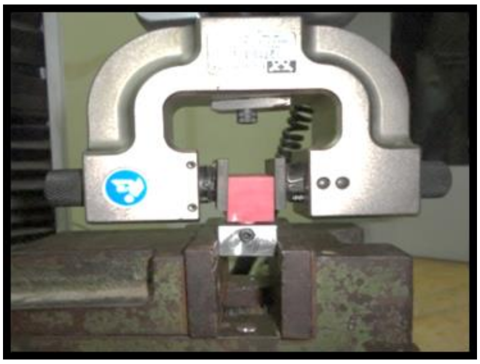


Fig 14: Tensile bond strength testing of the sample.

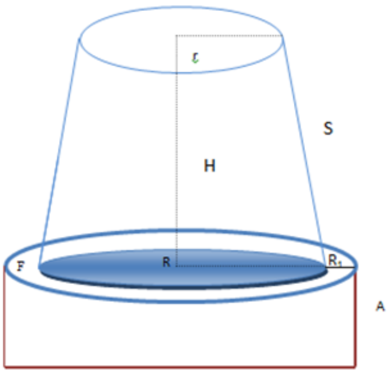


Fig 15: Schematic diagram of prepared natural tooth.

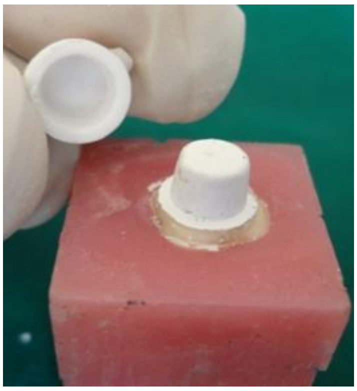


Fig 16: Debonded Sample -Group A.

Fig 17: Debonded Sample -Group B.

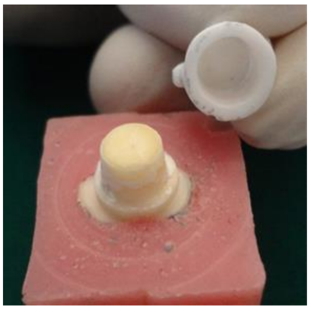


Fig 18: Debonded Sample -Group C.

The teeth were prepared by using a precision milling lathe machine (Medium cutting lathe, ANIL GROUP) (Fig.4) to have a uniform configuration of flat occlusal surface, 4mm axial length, 8 mm outer diameter, 6 mm inner diameter, 12-degree axial wall taper and shoulder finish line of 1mm width (Fig.5). After final preparation, the teeth were checked using a magnifying glass for any crack, fracture and pulp exposure. A special tray was fabricated using visible light cure resin material (DELTA, Polytray) for each of the prepared natural tooth (Fig.6). A spaced master die of the prepared tooth along with the acrylic block was obtained for this purpose using an addition silicone putty index (Aquasil, Dentsply) and die stone (Amann Girrbach, Alpenrock). A putty wash impression was made for each of the tooth with addition silicone putty and light body impression material (Aquasil, Dentsply) using above mentioned special tray after making vent holes and application of tray adhesive (Virtual, Ivoclar vivadent) (Fig.7). Impressions were poured using vacuum mixed type 4 stone (Amann Girrbach, Alpenrock). The dies were assigned the same number as the prepared natural teeth from which they were fabricated and divided into same three groups- Group A, Group B and Group C (Fig.8). The zirconia copings were fabricated using partially

Table 1: Crown dislodgement force, Tensile bond strength, Mean, Standard deviation for Group A.

Sample No.	Crown dislodgement force (Newton)	Tensile bond strength (Mpa)
1	62.31	0.5406
2	80.19	0.6957
3	83.41	0.7236
4	80.69	0.7000
5	81.92	0.7107
6	82.66	0.7171
7	77.44	0.6718
8	42.63	0.3698
9	80.00	0.6940
10	127.52	1.1063
Mean and Standard deviation		0.69359 , 0.18

Table 2: Crown dislodgement force, Tensile bond strength, Mean, Standard deviation for Group B.

Sample No	Crown dislodgement force in Newton	Tensile bond strength (Mpa)
11	113.66	0.9861
12	103.73	0.8999
13	103.23	0.8956
14	100.00	0.8676
15	129.19	1.1208
16	169.65	1.4545
17	127.18	1.1034
18	135.03	1.1715
19	97.04	0.8419
20	122.08	1.1059
Mean and Standard deviation		1.04004 , 0.19

Table 3: Crown dislodgement force, Tensile bond strength, Mean, Standard deviation for Group C.

Sample No	Crown dislodgement force in Newton	Tensile bond strength (Mpa)
21	490.22	4.2531
22	470.82	4.084
23	395.50	3.4313
24	440.78	3.8242
25	388.25	3.3684
26	420.03	3.6441
27	489.99	4.2511
28	510.47	4.4288
29	468.83	4.0675
30	498.30	4.3232
Mean and Standard deviation		3.9676 , 0.38

Table 4: Comparison of mean and standard deviation of tensile bond strength of Group A, B, C using One way analysis of variance (ANOVA).

Group	n	Mean	Standard deviation	F value	'p' value
Group A	10	.6929	.1829	456.017	.000**
Group B	10	1.045	.1874		
Group C	10	3.968	.3795		

Table 5: Multiple comparison of mean and standard deviation of tensile bond strength within Groups A, B, C using Post-hoc Tukey HSD analysis.

Group	N	Mean	Standard deviation	Mean Difference	'p' value	Level of significance
Group A	10	.6929	.1829	0.3521	.017*	0.05
Group B	10	1.045	.1874			
Group A	10	.6929	.1829	3.2751	.000**	0.05
Group C	10	3.968	.3795			
Group B	10	1.045	.1874	2.923	.000**	0.05
Group C	10	3.968	.3795			

Table 6: Type of failure mode for zirconia copings luted to natural teeth in Group A.

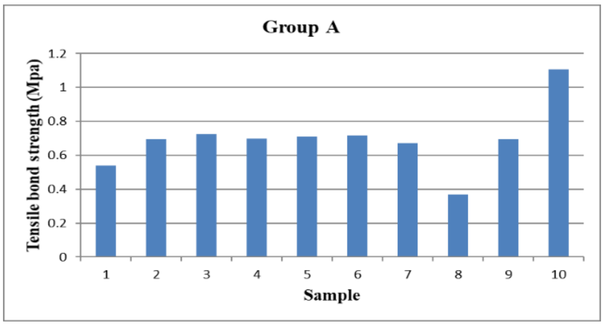
Sample No	Type of failure
1	Type -1
2	Type -1
3	Type -1
4	Type -1
5	Type -1
6	Type -1
7	Type -1
8	Type -1
9	Type -1
10	Type -1

Table 7: Type of failure mode for zirconia copings luted to natural teeth in Group B.

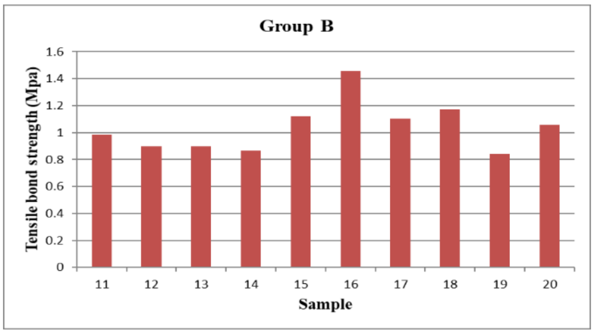
Sample No	Type of failure
11	Type- 2
12	Type -2
13	Type -2
14	Type -2
15	Type -3
16	Type -2
17	Type -2
18	Type -2
19	Type -3
20	Type -3

Table 8: Type of failure mode for zirconia copings luted to natural teeth in Group C.

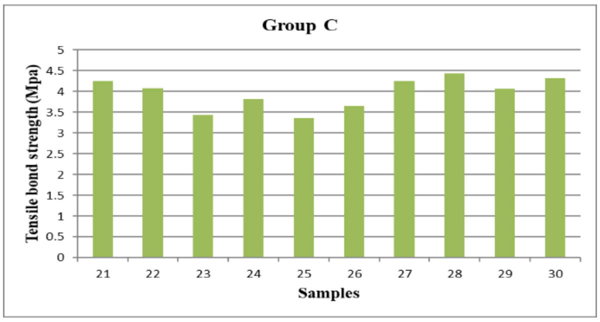
Sample No	Type of failure
21	Type- 4
22	Type -4
23	Type -4
24	Type -3
25	Type -4
26	Type -4
27	Type -4
28	Type -3
29	Type -3
30	Type -4



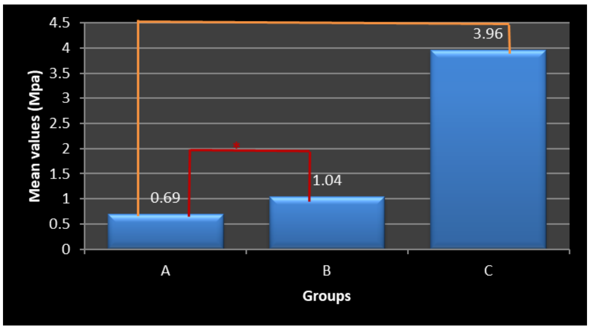
Graph 1: Basic values of tensile bond strength (Mpa) obtained for Group A.



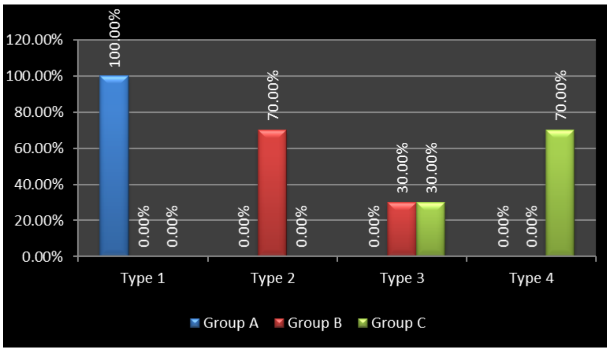
Graph 2: Basic values of tensile bond strength (Mpa) obtained for Group B.



Graph 3: Basic values of tensile bond strength (Mpa) obtained for Group.



Graph 4: Mean values of tensile bond strength (Mpa) for Group A, B, C.



Graph 5: Comparison of mode of failure of zirconia copings luted to natural teeth in Group A, B, C.

sintered zirconia (Ceramill ZI) which has got high strength and rigidity. Ceramill system (AMANN GIRRBACH, ceramill MOTION) was used for this purpose which consists of a scanning system (Ceramill map 300 work station), CAD software (Ceramill mind CAD), a CAM machining system (Ceramill CAM) and a sintering furnace (Ceramill therm high temperature furnace). The thickness of the zirconia coping was set uniformly as 1mm throughout with an occlusal bar on the external surface to facilitate tensile bond strength testing (Fig.9, 10). The thickness of die spacer was incorporated virtually in the coping design. Spacer thickness of 25 micron was designed for Group A, B and 15 microns for Group C which is the recommended spacer thickness for zinc phosphate, glass ionomer and self-adhesive resin cement respectively. The copings were fitted on the prepared teeth without the need of adjustments of intaglio surface. The intaglio surface of each zirconia coping was sandblasted with 50µm aluminum oxide powder for 15seconds using a sandblaster at a distance of 10 mm with 15psi pressure. Cementation of the samples was carried out following manufacturer's instructions as per their assigned luting agent under finger pressure for 10 minutes followed by under 2 Kg weight for 10 minutes by placing them on surveying platform under the surveying arm with the weight (Fig.11, 12, 13). All the prepared test samples were stored in distilled water at room temperature for 3 days before tensile bond strength testing. A universal testing machine (Instron, Lloyd instruments U.K) was used to determine the tensile bond strength of test samples (Fig.14). Tensile force was applied along the long axis of the sample. Each coping was pulled at a crosshead speed of 5mm/minute, until the test coping debonded from the prepared natural tooth. The computer attached to the testing machine recorded the force value at which this debonding occurred in Newton and were converted into Megapascal (MPa) by the formula given:

Tensile bond strength (MPa) = Force applied (N)/surface area (mm^2).

Total surface area of the prepared natural tooth = Area of the axial surfaces + Area of the occlusal surface + Area of the finish line.

Area of the axial surfaces = p (R+r) L (curved surface area of a cone frustum)

Area of the occlusal surface = πr^2

Area of finish line = $\pi R_1^2 - \pi R^2$

A schematic diagram of the prepared tooth has been given where R = Radius at base 3mm, r = Radius at top 2.65mm, R_1= Outer radius 4mm, H = Vertical height of the preparation 4mm, S = Slant height 4.015mm, F = Shoulder finish line 1mm, A = Acrylic resin block (Fig.15).The surface area was uniform for all the samples and was calculated to be 115.26 mm^2.Tensile bond strength values were tabulated for each group and subjected to statistical analysis using single factor analysis of variance-ANOVA and pairwise comparison of mean values by post-hoc test-Tukey's HSD analysis. After tensile bond strength testing, the mode of bond failure was determined by visual examination (Fig. 16, 17, 18) by a single operator employing the criteria proposed by Johnson et al[2]as discussed in results.

RESULTS

The results obtained were tabulated and subjected to statistical analysis. The mean tensile bond strength, standard deviation for Group A was 0.69359Mpa, 0.18 with maximum and minimum values being 1.1063Mpa and 0.3698Mpa respectively (Table 1, Graph 1).For Group B, the mean tensile bond strength, standard deviation was 1.04004 Mpa, 0.19 with maximum and minimum values being 1.4545Mpa and 0.8419Mpa respectively(Table 2, Graph 2). For Group C, the mean tensile bond strength, standard deviation was 3.9676Mpa, 0.38 with maximum and minimum values being 4.4288Mpa and 3.4313Mpa respectively (Table 3, Graph 3).

The overall comparison of mean value of tensile bond strength (Mpa) of the three test groups showed Group C exhibited the highest tensile bond strength followed by Group B and least by Group A, Group C > Group B > Group A. One-way analysis of variance-ANOVA yielded a 'p' value < 0.05 ($P = 0.000$). Hence, there was a statistically significant difference in the tensile bond strength among the three test groups (Table 4, Graph 4). Post- hoc test showed statistically significant difference in the tensile bond strength within the three groups (Table 5).

All the debonded test samples were then examined and characterized for mode of failure using the criteria proposed by Johnson et al[2]as given below:

The results were tabulated as given in Table 6, 7, 8. The mode of failure for all 10 samples in Group A was type 1 failure - cement principally on prepared teeth. Group B showed mixed mode of failure with predominant mode being Type 2 - cement both on prepared teeth and zirconia coping. 7 samples in Group B showed Type 2 failure and 3 samples showed Type 3 failure – cement principally on zirconia coping. In Group C, mixed mode of failure was found predominant being Type 4 - fracture of prepared tooth. 7 samples showed Type 4 failure and 3 samples showed Type 3 failure - cement principally on zirconia coping. Comparison of mode of failure in Group A, B, C has been graphically represented in graph 5.

DISCUSSION

Two main areas of concern regarding the use of zirconia for fixed prostheses are the bond strength between zirconia and the natural tooth and the bond strength between zirconia and the veneering ceramic. Studies were designed in the past to evaluate the bond strength of various zirconia systems and luting agents available in the market. However, the results of these studies were conflicting and inconclusive. While authors like Claus-peter Ernst et al[4]report a median bond strength value of 1.7Mpa for a resin luting agent, Palacios et al[2]reported a mean bond strength value of 5.1 Mpa for a similar resin luting agent. Atsuet al[5]reported a marginal increase of shear bond strength with silanization and tribochemical treatment, whereas Wegner and Kern[7]reported that neither air borne abrasion nor silanization improved the resin bond adhesion on zirconia. On the contrary Shahin[8]in their study concluded that air abrasion increases the bonding of zirconia to teeth both in case of conventional luting with zinc phosphate cement, glass ionomer cement and adhesive resin cement. Palacios et al[2]concluded that there was no significant difference in the retention of zirconia crowns when luted with either resin cement or resin modified GIC. Hence it is obvious that though these studies can serve as valuable guide lines, one cannot draw any conclusive inference from these studies. With this background, present study was carried out.

Extracted natural teeth were prepared to receive the zirconia copings because investigation of retentive strength of luting agents is best studied using axial dislodgement forces with crowns cemented on extracted human teeth for better simulation of clinical conditions[2].Molar teeth were selected in order to obtain preparations of specific dimensions without inadvertent exposure of pulp chamber during preparation. Their relatively larger surface area and diverging roots resist fracture and removal from the embedding acrylic resin during testing.

Standardized preparation of specific dimensions was carried out using a precision industrial milling lathe in order to eliminate the operator variability. This led to establishing a uniform surface area of bonding for all the specimens and was easy to calculate using established mathematical principles[9].A 12-degree taper was selected as proposed by Goodacareet al[10]since it was clinically achievable and also offsets the increase in mechanical retention of crowns obtained with minimal tapers such as 2 to 5 degrees. The occluso-cervical height of the preparation was kept at 4mm in line with the previous studies[1,3,10,11].A shoulder finish line of width 1mm was selected which was in accordance with the authors such as Yuji kokubo[11]and Beuer[12]who recommended shoulder preparation to be the preferred finish line configuration for zirconia copings from both mechanical and periodontal point of view. Similar models were employed in studies by Peter Ernst[4] and Palacio[2].Thesurface area was calculated using the formula as suggested by Daniel Chan et al[9]. The zirconia copings were fabricated by obtaining a die for the scanning purpose to not only precludes the need for an optical impression but also to keep the sequence of work flow similar to that of a conventional FPD fabrication. The spacer thickness for each luting agent was selected according to the established guidelines found in the literature[13]. The spacer thickness was virtually incorporated during the milling of copings. In the present study, the zirconia coping was fabricated with an occlusal bar of height 1.5 mm to firmly secure the it to the testing jig and prevent its rotational movement during application of tensile stress.

Luting agents employed in this study were selected based on their properties and clinical applications. Conventional luting agents like zinc phosphate, zinc polycarboxylate and glass ionomer cements have the advantages such as ideal film thickness, adequate compressive and tensile strength, ease of manipulation etc. But luting agents like zinc phosphate cement fail to achieve good adhesive bond strength to tooth structure especially in situations like short clinical crowns, core build ups etc. Resin based cements on the other hand provide reliable adhesive bond with both the ceramic and tooth structure in addition to their superior mechanical and esthetic properties[1,5]. Composite resin cements containing an adhesive phosphate monomer provide good marginal seal along with adequate retention[7]. Resin cements are particularly helpful in enhancing the retention in cases with less preparation heights, more taper and a smaller surface area[3].

Various testing methods such as shear, tensile, micro tensile and push-out tests have been advocated to evaluate the retentive capabilities of a number of luting agents on zirconia crown[1,2,4]. Testing of properties like shear bond strength and micro tensile bond strength does not hold good for full coverage zirconia crowns. After an extensive review of various laboratory models, Heitnze et al has recommended tensile bond strength testing using full coverage crown pull off tests on extracted natural teeth as the most reasonable testing modality to evaluate the bonding effectiveness of luting agents[1].

The lower tensile bond strength values obtained with zinc phosphate cement in the present study can be attributed to many factors such as a smooth, milled intaglio surface, non-parallel axial walls of the preparation and a flat occlusal surface. This was in accordance with the fact that when using non-adhesive luting agents like zinc phosphate, retention is dependent on the geometric form of the tooth preparation that limits the paths of displacement of the cast restorations[14].

Zirconia copings luted using glass ionomer cement showed a mean tensile bond strength value higher than that of zinc phosphate cement. It is an established fact that the compressive strength of glass ionomer cement continues to increase for several weeks after luting which is attributed to the reconstruction of the silicate network[13]. This might lead to increased frictional resistance in addition to the adhesive retention thus yielding higher tensile bond strength values. Attempts to improve the adhesive properties of glass ionomer cement have led to the introduction of resin modified glass ionomer cement.

The higher bond strength achieved with self-adhesive resin cement can be attributed to the greater adhesive capability of such cements, especially those which contain an adhesive phosphate monomer. In addition, the formation of a hybrid layer due to the penetration of dentinal tubules by hydrophilic resins improves the bond between the cement and the tooth structure[5,6]. Efficient adhesion of the luting agent with both the zirconia surface and the prepared tooth surface may have reflected in the higher tensile bond strength values obtained in the present study. The film thickness of recently introduced resin cements are less 15-20µ when compared to the earlier ones which was around 25µ or more. Reduction in film thickness may have improved the tensile bond strength. Airborne particle abrasion is also said to improve bonding of resin cement to zirconia surface[5,15]. One should keep in mind that resin-based bonding requires a dry field and hence will be successful only in situations where high degree of isolation can be achieved. Maintenance of dry field during initial setting is also critical for glass ionomer cement. While comparing the rate of debonding of zirconia restorations over a period of 2 to 5 years, Heintzeet al[1]reported that zirconia copings luted with zinc phosphate cement had the highest rate of debonding. In the earlier studies carried out by Omar zidan[3], S. D. Heintze[1], Claus-peter Ernst et al[4]zirconia copings luted with self-adhesive resin cement exhibited the highest tensile bond strength.

The type of failure of the zirconia copings upon bond strength testing gives an idea regarding the adhesive properties of the luting agent. In group A, all of the samples showed type 1 failure in which the zinc phosphate cement largely remained on the tooth surface with little or no cement remaining on the intaglio surface of the coping which indicated an adhesive mode of failure. This might be due to the comparatively smooth milled surface of the coping which offers very little mechanical interlocking with the luting agent and possibly explains the lower bond strength values obtained with this group. In Group B, 70% of the samples showed type 2 failure followed by type 3 failure in 30% of the samples. This indicated a mixed mode of failure which is predominantly adhesive in nature which in turn is reflected in the moderate increase in tensile bond strength of Group B samples.

In Group C, 70% of the samples exhibited type 4 failure which is characterized by cohesive failure of the sample showing fracture of prepared tooth with the coping retaining the bond to the tooth. This indicated good tensile bond strength between the resin luting agent, the zirconia surface and the tooth. However, it has to be noted that the actual tensile bond strength values for Group C will be more than the values obtained at the point of test sample failure, since the bond between the zirconia coping and the tooth had not yet failed. This superior bonding ability of self-adhesive resin cement was reflected in the significantly high tensile bond strength values obtained for Group C. The other 30% of the samples showed type- 3 failure in which the resin cement was predominantly found on the intaglio surface of the zirconia coping. This might be due to the adhesive phosphate monomers incorporated in the cement and also due to the sandblasting done before luting. Resin cements are known to flow and heal minor cracks developed during sandblasting, thus strengthening the zirconia and improving the bond. In this study no dentine bonding agent was used since a self-adhesive resin cement was employed which requires no pretreatment of either the zirconia coping or the tooth. In general, the mode of failure exhibited by all the three groups in the present study was in accordance to those reported for the samples in the previous studies[2,3].

The present study was conducted under invitro conditions. The effects of long-term thermal degradation and cyclic mechanical stresses on the restorations were not considered. The tooth preparation was purposefully designed to minimize the mechanical retention so that the adhesive capacity of the luting agents can be measured. Further long-term clinical trials are needed to accurately evaluate the criteria measured in this study.

CONCLUSION

Within the limitations of present study, it was found that self-adhesive resin cement demonstrated statistically significant higher tensile bond strength than glass ionomer cement and zinc phosphate cement. The difference in the tensile bond strength of glass ionomer cement and zinc phosphate cement was also found to be statistically significant. The mode of interfacial bond failure was found to be in accordance with the tensile bond strength values obtained for all the three groups. Self-adhesive resin cement showed a predominantly cohesive mode of failure which corresponds well with the highest bond strength obtained for that group. Based on the findings of the present study, it can be recommended that self-adhesive resin cement can be used for the cementation of zirconia crowns in clinical situations in order to achieve a more durable bond with the tooth structure provided the isolation protocol and manufacturer's instructions are followed accurately.

CONFLICTS OF INTEREST

The authors declare they have no potential conflict of interests regarding this article.

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