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Comparative evaluation of the influence of various surface treatments on the immediate and delayed bond strength and microleakage of zirconia posts – An in-vitro study.

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#### Abstract

**Background:** Increasing demand for more aesthetically appealing and biocompatible restorations has led to the development of tooth coloured, metal free post core systems for endodontically treated teeth lacking coronal tooth structure. Zirconia posts exhibit excellent bio compatibility, corrosion resistance, favourable optical properties and mechanical strength. However, failures of zirconia posts, predominantly results from loss of retention at the post/cement interface. Various surface pre-treatment procedures were introduced to enhance the bonding of zirconia posts. Adhesion is important for the success of the post system, not just in terms of bond strength but also to prevent microleakage.

Though literature has various studies to measure the bond strength and microleakage between various surface treated zirconia posts and luting cement, there are no studies comparing the bond strength and microleakage of tribochemical silica coated and laser surface treated zirconia posts with conventional zirconia posts.

Aim of the study: The aim of this study is to evaluate and compare the effect of surface pre-treatments with

tribochemical silica coating and Nd: YAG laser irradiation on the immediate and delayed bond strength and microleakage of zirconia posts.

**Methodology:** Forty-five extracted maxillary second premolars were root canal treated followed by post space preparation. The specimens were divided into 3 main groups based on the surface treatments of the zirconia posts: untreated zirconia posts (group 1), tribochemical silica coated zirconia posts (group 2) and Nd: YAG laser treated zirconia posts (group 3). Posts were cemented into the root canals using dual cure resin cement. Specimens from each group was further divided into three sub groups based on testing parameters.

Five specimens each from all three groups were subjected to immediate pull-out bond strength (subgroup A), ten specimens each from all three groups were subjected to thermocycling. Delayed pull-out bond strength was done for five specimens each from all three groups (sub-group B). Five specimens each from all three groups were immersed in 2% methylene blue solution for 1 week and then tested for microleakage (sub - group C). The ratio of the length of the dentincement interface and length of the dye penetration was registered using stereomicroscope.

**Results:** Statistically analysis was done using the Kruskal -Wallis test and Mann-Whitney U test to evaluate immediate pull-out bond strength, delayed pull-out bond strength and microleakage. It was observed that there were statistically significant (P<0.002) differences between the immediate pull-out bond strength of all the three groups with Group 3A reporting the highest bond strength followed by group 2A and 1A.

It was observed that there were statistically significant (P<0.002) differences between the delayed pull-out bond strength of all the three groups with Group 3B reporting the highest delayed pull-out bond strength followed by

group 2B and 1B. Statistically significant (P<0.002) differences were observed between the groups for microleakage after thermocycling with group 3C reporting the least micro leakage followed by groups 2C and 1C.

**Conclusion:** Within the limitations of the present study it can be concluded that the Nd: YAG laser treated zirconia posts showed higher immediate and delayed pull-out bond strength when compared to tribochemical silica coated and untreated zirconia posts. Increased microleakage was observed in the untreated zirconia posts followed by the tribochemical silica coated and Nd: YAG laser treated zirconia posts.

**Keywords:** zirconia posts, tribochemical silica coating, laser, pull-out bond strength, microleakage

#### Introduction

A post and core system is required to improve the retention and support of endodontically treated teeth with a significant loss of coronal tooth structure<sup>1</sup>. Posts can be divided into two categories: custom/cast posts with a cast core and prefabricated posts, primarily with a composite core.

Metal alloys are generally used to fabricate custom/cast posts. Prefabricated posts are divided into two groups: metallic, such as titanium alloy posts, and non-metallic, such as zirconia ceramic, glass fibre-reinforced composite and glass-ceramic posts <sup>2</sup>. Metal posts and cores are commonly used because of their superior physical properties. Nevertheless, the increased use of all-ceramic restorations provides a rationale for tooth-coloured post and core systems <sup>3</sup>. When light is reflected through a metal post-and-core foundation, it may make a metal-free ceramic crown appear grey and discoloured. To ensure a successful Esthetic outcome, the post-and-core system would need to be tooth-coloured, reflecting

and transmitting light in a manner similar to a natural tooth  $^4$ .

In response to the demand for tooth-coloured posts, several non-metallic dowels were marketed. Among them, epoxy resin posts reinforced with carbon fibres, epoxy or methacrylate resin posts reinforced with quartz or glass fibres, zirconia posts and fibre-reinforced posts are quite popular <sup>5</sup>. Each one of the above system have their own advantages and disadvantages. Zirconia posts were first introduced by Meyenberg et al. in the year 1995, who reported that the flexural strength (900–1200 MPa) of these posts were comparable to that of cast gold or titanium posts and that it was possible to have the same post dimensions as high gold alloys or titanium. Currently in Prosthodontics, zirconia is a widely used material because of its good chemical stability, high mechanical strength, high toughness and a Young's modulus similar to that of stainless steel alloy. The high initial strength and fracture toughness of partially stabilized zirconia stems from a physical property known as trans formation toughening. Zirconia posts demonstrate high fracture resistance due to high flexural strengths, which is comparable to that of cast gold post and core or titanium posts <sup>6</sup>. Apart from its favourable chemical and physical properties, zirconia also wields the Esthetic advantage of having colour similar to that of natural teeth 7.

However, some authors have suggested that the zirconiabased post should be avoided due to the possibility of insufficient bond strength. Clinical reports have emphasised on the importance of post and core stability suggesting that it is one of the prime factors responsible for restoration failures. Several procedures to modify the surface of posts or dentin have been developed to enhance the bonding of the post to the luting cement and thereby increase the survival of intracanal posts. Examples of such methods to enhance the bond between dental materials include ultrasonic/sonic techniques, laser irradiation, silanization and/or adhesive application, acid etching, silica coating and sandblasting <sup>7</sup>.

Tribochemical silica coating consists of an airborne particle–abrasion pre-treatment with silica coated aluminium oxide powder followed by the application of silane. Some authors report that tribochemical silica coating provides durable bond between glass-infiltrated aluminium oxide ceramics and Bis-GMA-based composite resin cements <sup>4</sup>.

In addition to the current surface conditioning methods, another new treatment protocol to modify the zirconia surface and develop roughness is laser etching. The neodymium: yttrium-aluminium-garnet (Nd: YAG) laser is efficiently used in reducing tooth sensitivity, removing caries, bleaching and roughening high-strength ceramic surfaces prior to adhesive cementation. According to Usumez et al., when compared with control and airborne particle abrasion, Nd: YAG laser irradiation of the zirconia surface exhibited significantly higher bond strength values. Similar results were seen in the study by Akin et al. who demonstrated that bond strength values between zirconia and resin cement in surfaces treated with nd: YAG laser and Er: YAG laser were significantly higher than in control and sandblasted ones 8

Adhesion is important for the success of the post system, not just in terms of bond strength but also to prevent microleakage <sup>9</sup>. Microleakage is one of the factors that deter mines the longevity of indirect restorations. The chances of post and core failure due to coronal micro leakage is more likely than apical microleakage due to the efficacy of the apical 3 to 5mm of root filling. The type of luting agent used for post cementation is important in retention and prevention of microleakage.

Hence improving the bond between posts and dentin as well as the post and core will not just increase the stability but will also have a role to play in the reduction of microleakage<sup>10</sup>. Though literature has various studies to measure the bond strength and microleakage between various surface treated zirconia posts and luting cement, there are no studies comparing the bond strength and microleakage of tribochemical silica coated and laser surface treated zirconia posts with conventional zirconia posts. Therefore, the aim of this study is to evaluate and compare the effects of tribochemical silica coating and laser treatment on the immediate and delayed bond strength and microleakage of zirconia posts.

#### Materials and methodology

#### **Specimen preparation**

Forty-five extracted non-carious, human maxillary second premolar teeth with similar morphology were collected. The teeth were preserved in 10% formalin solution. Teeth were decoronated 2mm above the CEJ using a diamond disc.



Figure 1: Teeth stored in 10% formalin



Figure 2: Decoronation of the teeth using a diamond disc **Root canal preparation** 

Root canals were prepared using a step back technique with hand files (K files, Mani) followed by irrigation with 5.25% sodium hypochlorite (Hypo clean). Obturation was done with gutta-percha cones (Dentsply) using lateral condensation technique with AH plus sealer (dentsply). Root canal filling material was then removed leaving behind up to 5mm of material from the apex with peesoreamer.



Figure 3: Root canal preparation using hand files



Figure 4: Obturation with gutta percha cones

#### Post space preparation

A post drill (Ivoclar Vivadent) of size 1.4mm was used to prepare the root canals followed by irrigation with water. Root canals were rinsed with 5.25% sodium hypochlorite and dried with paper points. 37% phosphoric acid was used to etch the root canal for 10-15 secs, then rinsed and dried with paper points.



Figure 5: Post space preparation
Surface treatment method

1. Tribochemical silica coating (n=15):

The post surface (zirconia posts, Cosmo posts, ivoclar) was treated with 110µm aluminium oxide particles, followed by coating of silica modified aluminium oxide particles at a distance of 10mm for 15 seconds. This was followed by silane (Angelus) application with brush and allowed to air dry for 5 minutes.



Figure 6: Cosmo posts (Zirconia posts)



Figure 7: Tribochemical silica coating surface treatment 2. Laser treatment (n=15)

The post surface was subjected to Nd: YAG laser of 1064nm wavelength using a laser optical fibre of 300µm diameter at a distance of 1mm without contact in free-running pulse mode with air cooling for 20 sec.



# Figure 8: Nd: YAG laser surface treatment **Cementation**

Dual cure cement (Variolink N, Ivoclar) was mixed in the ratio of 1:1 and the mixture was applied to the post and post space with a lentulospiral. Post was then

inserted into the root canal by applying slight pressure to hold it in place. Light curing (Woodpecker) of the cement was done for 60 seconds.





Figure 9: Post cementation using resin cement



Figure 10: Light curing of the cement

#### Group 1: untreated zirconia posts (n=15)

**Sub-group A:** Tested for immediate pull-out bond strength after 24 hours of storage in distilled water (n=5).

Sub-group B: Tested for delayed pull out bond strength after 30 days of storage in distilled water followed by thermocycling (n=5).

Sub-group C: Tested for microleakage after 30 days of storage in distilled water followed by thermocycling (n=5).

Group 2: zirconia posts treated with tribochemical silica coating (n=15)

Sub-group A: Tested for immediate pull-out bond strength after 24 hours of storage in distilled water (n=5).

Sub-group B: Tested for delayed pull-out bond strength after 30 days of storage in distilled water followed by thermocycling (n=5).

Sub-group C: Tested for microleakage after 30 days of storage in distilled water followed by thermocycling (n=5).

## Group 3: zirconia posts treated with nd: yag laser (n=15)

Sub-group A: Tested for immediate pull-out bond strength after 24 hours of storage in distilled water (n=5).

Sub-group B: Tested for delayed pull-out bond strength after 30 days of storage in distilled water followed by thermocycling (n=5).

Sub-group C: Tested for microleakage after 30 days of storage in distilled water followed by thermocycling (n=5).



Figure 11: Forty-five teeth after zirconia post cementation

#### Mounting the specimens

Specimens in subgroup A and B of all groups were mounted individually in a mould of dimension 2.5cm×2cm×2cm using autopolymerising resin (DPI), parallel to the long axis of the posts.



Figure 12: Specimens mounted in autopolymerising resin

#### Testing for immediate pull-out bond strength

After the polymerization of the luting agent, five specimens of group 1, group 2 and group 3 was stored in distilled water at room temperature for 24 hours. In order to check the pull-out bond strength in universal testing machine (Instron), the lower part of the block was fixed to the jig of the machine from both sides and coronal part of the post was connected to the mandrel attached to upper compartment of the machine. Post head was grasped and dislodged by pulling the post along its long axis.

A constant load of 0.5mm/min was applied until debonding occurred. The maximum load value for each sample was recorded in Newton(N). The pull-out bond strength (MPa) was calculated using the load, diameter of the zirconia posts and length of the zirconia posts.

#### Artificial ageing

Ten specimens from groups 1, 2 and 3 were stored in water at 37°C for 30 days and then subjected to thermocycling (Schuler dental time temperature controller) for 5000 cycles between 5 °C to 55 °C for a dwell time of 30 seconds each and transfer time of 15 seconds between baths.



Figure 13: Thermocycling

Testing for delayed pull-out bond strength

Five specimens from group 1, group 2 and group 3 after artificial ageing was used to check the delayed pull-out bond strength in universal testing machine.



Figure 14: Testing for pull-out bond strength

#### Testing for microleakage

Five specimens from each group after artificial ageing was dried with absorbent paper tissue and then coated with clear nail varnish to prevent penetration of the dye into the tooth. Area of 1mm from the coronal surface was left uncoated. The specimens were immersed in 2% methylene blue (Anmol) solution for 1 week and rinsed under running tap water. The root was sectioned vertically with a diamond disc dividing the posts into halves.

Each section was inspected under stereomicroscope at 10x magnification to evaluate the extent of dye penetration in millimetre. Images of the specimens were obtained from a Nikon digital camera mounted to the stereomicroscope and the measurements of microleakage

were done using the Image J software. The software was calibrated to millimetre scale and the obtained images were analyzed for microleakage. The measurements of total post length (dentin-cement interface) and depth of dye penetration were calculated from which the ratio of both values were registered.



Figure 15: Immersion of specimens in 2% methylene blue & vertical sectioning of specimens using diamond disc



Figure 16: Stereomicroscope analysis

#### Results

Statistical analyses were performed using IBM SPSS Statistics for Windows, Version 25.0. Armonk, NY: IBM Corp. Results on continuous measurement are presented as Mean  $\pm$  SD; Median (IQR) and categorical as Frequency & percentage. Comparison for continuous variables between two groups was made using the Mann-Whitney U test and three groups using the Kruskal-Wallis test. A p-value less than 0.05 was considered statistically significant.

The overall comparison of the immediate pull-out bond strength across the three groups in the study are tabulated in Table 1. It is observed that there are statistically significant differences between the im mediate pull-out bond strength of all the three groups with Group 3A reporting the highest bond strength followed by group 2A and 1A.

Table 1: Overall comparison of Immediate pull-out bond strength (Mpa) between three groups

Group	N	Range	$Mean \pm SD$	Median (IQR)	P value
1A	5	155.45- 163.23	160.05±3.22	160.84(156.82- 162.89)	0.002*
2A	5	229.71- 242.39	236.04±5.44	235.57(230.81- 241.51)	
3A	5	292.83- 322.38	308.15±12.3 2	311.78(295.47- 319.03)	



#### Graph 1:

Intra group analysis by post-hoc comparison reveals that there are statistically significant differences between the groups as well as observed in Table 2. Group 3A is observed to have statistically superior immediate pullout bond strength than groups 2A and 1A while group 2A displayed a statistically superior immediate pull-out bond strength than group 1A.

Table 2: Post-hoc comparison of Immediate pull-outbond strength (Mpa) between two groups.

Group	Group	Mean Difference	P value
1A	2A	-75.99	0.001*
	3A	-148.10	0.001*
2A	3A	-72.11	0.001*

\*statistically significant (p<0.05) (Mann-Whitney U test) The overall comparison of the delayed pull-out bond strength obtained after thermocycling of the three groups in the study are tabulated in Table 3. It is observed that there are statistically significant differences between the delayed pull-out bond strength of all the three groups with Group 3B reporting the highest delayed pull-out bond strength followed by group 2B and 1B.

Table 3: Overall comparison of Delayed pull-out bond strength- after thermocycling (Mpa) between three groups

Group	N	Range	Mean ± SD	Median (IQR)	P value
1B	5	79.83- 92.12	84.03± 5.29	81(80.14- 89.43)	0.002*
2B	5	151.07- 163.81	158.74± 4.85	159.95(154.29- 162.59)	
3B	5	191.08- 225.14	208.24± 14.09	207.85(194.65- 222.03)	

\*Statistically significant (p<0.05) (Kruskal Wallis test).



Graph 2:

Intra group analysis of the delayed pull-out bond strength by post-hoc comparison reveals that there are statistically significant differences between the groups as observed in Table 4. Group 3B is observed to have statistically superior delayed pull-out bond strength than groups 2B and 1B while group 2B displayed a statistically superior delayed pull-out bond strength than group 1B.

Table 4: Post-hoc comparison of Delayed pull-out bondstrength- after thermocycling (Mpa) between two groups

Group	Group	Mean Difference	P value
1B	2B	-74.70	0.001*
	3B	-124.20	0.001*
2B	3B	-49.50	0.001*

\*statistically significant (p<0.05) (Mann-Whitney U test) The overall comparison of microleakage after thermo cycling of the samples between the three groups has been tabulated in Table 5. Statistically significant differences are observed between the groups for micro leakage after thermocycling with group 3C reporting the least micro leakage followed by groups 2C and 1C.

Table 5: Overall comparison of Microleakage-afterthermocycling between three groups

Group	N	Range	Mean ± SD	Median (IQR)	P value
1C	5	0.59- 0.66	0.63±0.02	0.63(0.60- 0.65)	0.002*
2C	5	0.33- 0.36	0.34±0.01	0.34(0.33- 0.35)	
3C	5	0.09- 0.17	0.12±0.03	0.12(0.09- 0.15)	

\*Statistically significant (p<0.05) (Kruskal Wallis test).

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Graph 3:

Intra group analysis of microleakage post thermocycling between the groups by post-hoc comparison reveals that there are statistically significant differences between the groups as observed in Table 6. Group 1C is reported to have significantly higher microleakage when compared to groups 2C and 3C while group 2C is reported to have significantly higher microleakage when compared to group 3C.

Table 6: Post-hoc comparison of Microleakage-afterthermocycling between two groups

Group	Group	Mean Difference	P value
1C	2C	0.28	0.001*
	3C	0.50	0.001*
2C	3C	0.22	0.001*

\*statistically significant (p<0.05) (Mann-Whitney U test) The immediate and delayed pull-out bond strength of all the three groups are compared by Mann Whitney U test and the results are tabulated in Table 7. Statistically significant differences are observed in the comparison between immediate and delayed pull out bond strength across all the groups with immediate pull-out bond strength found to be higher than the delayed pull-out bond strength. Thus it can be concluded that delayed pull-out bond strength after 5000 cycles of thermocycling is significantly lesser than the immediate pull-out bond strength across all the groups involved in the study.

Table 7: Comparison of immediate and delayed pull out

bond strength

Group	N	Range	Mean ± SD	Median (IQR)	P value
1A	5	155.45- 163.23	160.05± 3.22	160.84(156.82- 162.89)	0.001*
1B	5	79.83- 92.12	84.03±5. 29	81(80.14- 89.43)	
2A	5	229.71- 242.39	236.04± 5.44	235.57(230.81- 241.51)	0.001*
2B	5	151.07- 163.81	158.74± 4.85	159.95(154.29- 162.59)	
3A	5	292.83- 322.38	308.15± 12.32	311.78(295.47- 319.03)	0.001*
3B	5	191.08- 225.14	208.24± 14.09	207.85(194.65- 222.03)	

\*statistically significant (p<0.05) (Mann-Whitney U test)



#### Graph 4:

#### Discussion

A post provides retention and resistance for an artificial crown in conjugation with a core which lays the foundation of the restoration, restoring sufficient coronal anatomy of an endodontically treated tooth.<sup>7</sup> Prefabricated posts are divided into two groups - Metallic, such as Titanium alloy posts and non-metallic such as Zirconia ceramic, glass fiber-reinforced

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composite and glass ceramic posts. With the prefabricated post and core system, the root canal is enlarged to fit the configuration of the post and is luted to the canal following which the core is built on the same using appropriate restorative materials such as dental composites. Decision making and choice of materials of the posts depends multiple factors such asposition of teeth in the arch, amount of remaining tooth structure and Esthetic requirements. Zirconia posts were first introduced by Meyenberg et al, who reported that the flexural strengths of these posts were comparable to gold or titanium and that it was possible to have the same post dimensions as high gold alloys or titanium.<sup>6</sup> It has various favourable chemical and physical properties and also wields the Esthetic advantage of having a color similar to that of natural teeth.

However, some authors have suggested that the zirconiabased posts should be avoided due to the possibility of insufficient bond strength. Clinical reports have emphasised on the importance of post and core stability suggesting that it is one of the prime factors responsible for the success of the restoration. Several procedures to modify the surface of posts or dentin have been developed to enhance the bonding of the post to the luting cement and thereby increase the survival of intracanal posts.

Various surface treatment modalities have been proposed to enhance the bond strength of composite resins to ceramic surfaces, which either micromechanically or chemically facilitate improved resin-ceramic bonding. These surface treatments may be broadly classified under the following categories:

1. Treatments that result in roughening of the surface. (Mechanical bonding)

2. Treatments that intend to create a chemical bond between the luting cement and post (chemical bonding)

3. Treatments that include both roughening and chemical component by using a combination of the above-mentioned methods. (Combination of mechanical and chemical bonding)

There is still no consensus in the dental literature com paring the bond strength and microleakage of various surface treated zirconia posts with conventional untreated zirconia posts. Hence, this study was aimed to evaluate and compare the effects of tribochemical silica coating and laser treatment on the immediate and delayed bond strength and microleakage of zirconia posts.

Cosmo post zirconia post and core systems have been used in this study under the influence of various surface treatment modalities to test the immediate and delayed bond strength of these restorations. Cosmo posts are prefabricated, cylindro-conical posts made of Zirconium Oxide ceramic, a material which has been successfully used for medical appliances since many years. They possess good biocompatibility, corrosion resistance and excellent mechanical strength and are free of porosity as they are produced from high-quality ceramic material under high temperature.<sup>12</sup>

Tribochemical silica coating is known to increase the bond strength between composite resin and high-strength ceramics. Tribochemical silica coating involves the ceramic surface being airborne-particle abraded with aluminium-oxide particles modified with silica. The blasting pressure results in embedding of silica particles on the ceramic surface. Tribochemical silica coating involves the following steps:

1. An airborne particle–abrasion pre-treatment with Rocatec Pre powder (110 mm aluminium oxide) to clean the surface, followed by

2. A silica coating with Rocatec Plus powder (110 mm aluminium oxide, coated with silicon dioxide) and lastly

3. The application of silane.

The silica coating "energizes" the substrate surface, which allows the silica to adhere to it. Silane improves the bond between the silica adhered to the substrate and the resin matrix. Various short-term studies revealed that tribochemical silica coating and silanization improved the bonding to bis-GMA composite resins. Although the manufacturer of Cosmo post does not recommend altering post surfaces, in this study it was found that the alteration caused by tribochemical treatment followed by silanization improved the bond strength. Tribochemical silica coating combines micromechanical retention produced by airborne-particle abrasion and chemical bonding resulting from silanization of the silicated ceramic surface. However, it was found that this high initial bond strength was not stable and decreased over time. Studies have reported that tribochemical silica coating may also cause microcracks within the material, causing undesirable changes in their mechanical properties.6

Akin et al reported that surface treatment of zirconia posts with Nd-Yag and Er-Yag lasers resulted in an improved bond strength of the posts when compared to Sandblasting and CO<sub>2</sub> laser. Similar findings were also reported by Usumez et al that Nd: YAG laser irradiation of the zirconia surface exhibited significantly higher bond strength values.<sup>4</sup> There are various factors that can affect the variations in the bond strength of posts treated with lasers such as the energy, output power, pulse duration and distance of application of the laser. The laser parameters used in this study were based on the previous studies to ensure optimal results in the experimental groups. The application of Nd: YAG laser to the dentin surface causes morphological changes, including melting and resolidification, as well as the formation of small globules. Additionally, the Nd: YAG laser helps to reduce dentin permeability, remove smear layer, and provide melting and recrystallizing of the dentin tissue.

In this study single rooted premolar teeth were used to manage the root lengths and number of canals. Therefore, the present study utilized pull out tensile bond strength in premolars, in contrast to a sectional technique for bond strength assessment, which avoids the irregular bond strength outcomes in the apical specimens and allows for a bond strength assessment in an orally simulated technique.

For a laboratory study to produce clinically relevant data, artificial ageing methods, including water storage and thermocycling was included in the study design. To simulate a clinical environment, a clinically relevant thermocycling protocol, matching current literature guide lines, was used in this investigation. Samples were stored in water at 37°C for 30 days and then subjected to thermocycling for 5000 cycles between 5 °C to 55 °C for a dwell time of 30 seconds each and transfer time of 15 seconds between baths. This protocol represented an estimated maximum of 5 years of service.

Micro leakage is also an important factor to be considered as it influences the initiation of secondary caries, as well as the survival rate of post and cores and ultimately, the failure of endodontic treatment.

Clinically, micro leakage is caused by fatigue, a form of failure that occurs in structures subjected to dynamic stress. As a result of continuous occlusal stress, there can be microcracks and adhesive failure along the interface between the posts and tooth structure.<sup>12</sup>

The quantitative analysis of dynamic microleakage using an image analyzer was used in a study and then the ratio of dye penetration to the total root area was measured. The infiltration of colorants is a common technique involving the immersion of the root in dyes like

methylene blue, Prussian blue, eosin and India ink. Many dyes have been used as tracers in microleakage studies, but methylene blue at various concentrations is the most popular reported by Spanberg et al. in 1989. Methylene blue dye was used in this study as its molecular size is similar to bacterial by-products. Matloff et al. compared methylene blue dye with radioisotopes of carbon, calcium chloride and iodine and found that dye penetrated farther than any of the isotopes, and also com mented on its solubility in water and ease of use, thus concluding that dye penetration the most sensitive method for studying was microleakage. A study conducted by Jung et al compared the microleakage of various post systems and the results showed that there was a significantly higher level of microleakage in cast posts (6%) compared to fibre posts (3%) and zirconia posts (2.3%).<sup>11</sup>

The immediate bond strengths of the untreated zirconia posts, tribochemical silica coated zirconia posts and Nd: YAG laser treated zirconia posts are tabulated in Table 1. There was statistically significant difference between the immediate pull-out bond strength in all three groups (P<0.002). It was observed that laser surface treatment (Group 3A) produced the highest immediate bond strengths with the zirconia posts followed by tribochemical silica coated zirconia posts (Group 2A) and lastly the control groups (Group 1A). Group 3A was observed to have statistically superior immediate pullout bond strength than Groups 2A and 1A while Group 2A displayed higher immediate pull-out bond strength than control group which was statistically significant (P<0.001). In this study the zirconia posts treated with tribochemical silica coating showed improved immediate pull-out bond strength when compared to the untreated zirconia posts.

This may be attributed to two mechanisms: the irregularities created by the particles provided mechanical retention and the silane layer provided chemical bonding to silica layer. Tribochemical silica coating of the zirconia posts may not be highly effective due to the high strength of the material which allows only limited formation of undercuts and roughness on the surface. Higher surface roughness of the ceramic surfaces can be reached if larger size aluminium oxide particles are used in conjunction with the silica-coated particles. The mechanical interlocking achieved by airborne-particle abrasion and tribochemical coating is similar and it has been suggested that the chemical bonding between silane coupling agents or MDPcontaining primer/ silane mixture and tribochemical silica was superior to the chemical bonding between MDP-containing primer/silane mixture and airborne particle abraded surface. In this study the reported statistically low immediate pull-out bond strength of the control group may be attributed to the smooth surface of the zirconia posts which showed the least bonding to the resin.

Statistically significant differences were observed in the comparison between immediate and delayed pull out bond strength across all the groups with immediate pull-out bond strength found to be higher than the delayed pull-out bond strength (P<0.001). Under environmental conditions of moisture and stress, zirconia transforms spontaneously to monoclinic phase which leads to surface damage further degrading the mechanical properties of zirconia. Sato et al. postulated that the reaction of water with zirconia at the crack tip formed zirconium hydroxides which accelerated crack growth of pre-existing flaws and further unstable zirconia phase transformation. The increase in the monoclinic phase leads to reduction in strength followed by micro and

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macro cracks. Thermocycling adversely affects the micro morphologic interface between the post and the resin cement as well as between the resin cement and the root canal dentin. As a consequence of thermocycling, there is formation of cracks on the zirconia surface, resulting in an increased gap formation between the zirconia posts and resin cement interface. This causes a reduction in the bond strength of the posts over a period of time.

The delayed bond strengths of the zirconia posts exposed to different surface treatments are tabulated in Table 3. It was observed that the laser surface treated zirconia posts (Group 3B) showed statistically significant higher delayed pull-out bond strength followed by tribochemical silica coated zirconia posts (Group 2B) and control groups (Group 1B). Laser treated zirconia posts showed statistically significant decrease in the delayed pull-out bond strength compared to immediate pull-out strength in the present study (P<0.001). Though laser creates surface roughness to increase mechanical interlocking between zirconia and resin cement, during thermocycling the bond weakens due to difference in coefficient of thermal expansion.

One of the main mechanisms of the zirconia/resin interface degradation can be thermal fatigue that can result in stress affecting the bond interface, e.g., thermal expansion and contraction could lead to unequal changes in dimensions and eventually lead to bond failure. This study reports a statistically significant decrease in the delayed bond strength of the tribochemical silica coated posts (P<0.002) which is in agreement with studies conducted by Valandro LF et al and Matinlinna JP et al. This decrease in the bond strength could be attributed to the hydrolytic degradation of siloxane bonds as the chemical bond between MDP-containing primer/ silane mixture and silane coated zirconia posts was primarily

dependent on siloxane bonds rather than MDP-ZrO<sub>2</sub> bonds. Prolonged contact with moisture may result in the hydrolysis of siloxane bonds and may cause a reverse chemical reaction breaking the silanol groups. The control group reported low delayed pull-out bond strength which was statistically significant (P<0.002) which may be attributed to lack of surface roughness leading to insufficient bonding of zirconia posts.

Microleakage values of different surface treated zirconia posts after thermocycling was compared using the Kruskal Wallis test and tabulated in Table 5. The overall micro leakage scores of the control group was significantly higher than the tribochemical silica coated zirconia posts which was significantly higher than laser treated posts. Flexion of each post under dynamic mechanical loading simulations plays a key role in dye penetration through the microcracks created between the core and cervical dentin. Luting materials used to cement the posts acts as a mediator and the surface treatment of the posts improves the bond strength between the posts and dentin. Thus, effective surface treatment of the post can reduce the microleakage of the zirconia posts after cementation using appropriate luting agents. The results of this study reveals the relationship between bond strength and microleakage of the zirconia posts under different surface treatment modalities. It is observed that higher bond strength of zirconia posts corresponds to lower dye penetration and microleakage in the specimens.

The present study has certain limitations such as the lack of comparative evaluation of bond strength of zirconia post against various post systems made of other material which includes fibre reinforced composite, metal, carbon firbre and epoxy fibre posts.

Fracture mode of zirconia posts were not evaluated that could be used to analyse the different types of failure

(adhesive, cohesive or mixed) occurring between zirconia post/resin cement and resin cement/dentin interface. Surface morphology of the posts have not been evaluated post the surface treatment protocol. The focus of the current study was oriented towards analyzing the variation in the bond strength of zirconia posts treated with different surface treatment modalities over a period of time under dynamic oral loading.

Future scope in this area revolves around evaluation of the pull-out bond strength of posts fabricated with novel materials and techniques such as custom-made fibre posts and laser sintered ceramic posts to identify simpler, biomimetic post and core systems.

Further analysis of the adaptation of posts in the root canal anatomy could also be performed with advanced diagnostic modalities like micro-CT, optical computed tomography and Nano CT.

Technologies like atomic force microscopy and surface profilometry could also help elicit the variations in the surface morphology of the posts under different surface treatments and the interaction between the luting agents and the altered post surface during integration with dentin.

#### Conclusion

Within the limitation of this study, it can be concluded that

1. Zirconia posts surface treated by Nd: YAG laser showed significantly higher immediate pull-out bond strength values when compared to tribochemical silica coated and untreated zirconia posts.

2. Zirconia posts surface treated by Nd: YAG laser exhibited superior delayed pull-out strength values when compared to tribochemical silica coated and untreated zirconia posts. 3. A significant reduction in the delayed bond strength was seen in all the three groups when compared to immediate bond strength.

4. Untreated zirconia posts exhibited significantly higher microleakage when compared to tribochemical silica coated and Nd: YAG laser treated zirconia posts.

5. An inverse correlation was observed between bond strength of the posts and microleakage in all the groups.

6. Zirconia posts surface treated by Nd: YAG laser is better clinical alternative to untreated zirconia posts as they showed increased bond strength and reduced microleakage.

However, further clinical studies are required to demonstrate the long-term success of various surface treated zirconia posts.

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