



A simplified etching technique to improve the adhesion of fiber post

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PURPOSE. Numerous methods were used to etch the fiber posts to improve its bonding to root canal dentin. Our aim was to evaluate the efficacy of 37% phosphoric acid in etching fiber posts in comparison with 24% hydrogen peroxide. **MATERIALS AND METHODS.** Ninety human maxillary central incisors were taken and post space preparation was done. Ninety fiber posts were taken and divided into three groups (n=30) based on the surface treatment they received (H₃PO₄, H₂O₂, distilled water) and each group was further divided (n=10) based on the time period of application (15 seconds, 30 seconds, 60 seconds). All the posts were luted into canals using Rely X UniCem-2. Each tooth was then sectioned into six slices and subjected to push out test. Data obtained was subjected to statistical analysis at *P*<.05. The surface topography was evaluated using scanning electron microscopy. **RESULTS.** Highest bond strength values were noted in 15 seconds etched phosphoric acid group and 60 seconds etched hydrogen peroxide group with no significant difference between two groups. Surface topography revealed complete epoxy layer removal with no damage to its structural integrity in those groups. **CONCLUSION.** H₃PO₄ etching for a period of 15 seconds is an effective alternative in improving the adhesion of fiber post to root dentin. [J Adv Prosthodont 2014;6:295-301]

KEY WORDS: Bond strength; Fiber posts; Hydrogen peroxide etching; Phosphoric acid etching; Scanning electron microscopy

INTRODUCTION

Fiber posts and resin based materials can behave as a mechanically homogeneous complex with dentin; their clinical use introduced a new restorative concept, ensuring high resistance to occlusal loading.¹ This assumption has been recently supported by several *in vivo* and *in vitro* investigations.²⁻⁷

Formation of monoblock is essential for the success of

a post luted to root canal dentin.⁸ Improper bonding at dentin/cement, cement/post interface leads to increased stress to occlusal loading leading to failure of post retained restorations. Bonding between resin cement and fiber post is aided by micromechanical and chemical means.⁹⁻¹² Fiber post is covered by epoxy resin which is highly cross-linked and has a high degree of conversion.¹³ Therefore roughening the post to improve micromechanical bonding has been recommended.¹⁰

Many techniques like sandblasting, etching with hydrofluoric acid were used to improve bonding,^{11,14,15} but these methods caused damage to the structure of glass fibers and affected the integrity of posts.¹¹ Agents that dissolve only the epoxy matrix portion without interfering with fiber integrity were studied.^{12,16-18} Potassium permanganate, sodium ethoxide, and hydrogen peroxide were used to etch and expose the glass fibers.^{10,12,16-18}

Twenty-four percent hydrogen peroxide etching for 1 min proved to be effective in improving bond strength.¹⁹ Thirty-seven percent phosphoric acid is the commonly used agent to etch enamel and dentin that is readily avail-

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able. The objective of the present *in vitro* study was to evaluate the efficacy of 37% H₃PO₄ in comparison with 24% H₂O₂ to etch fiber posts in terms of push out bond strength at various time periods. In addition surface topography was evaluated by SEM and bond strengths at various root regions were compared.

MATERIALS AND METHODS

Approval for the study was taken from Human Rights and Research Committee, Mamata Dental College. Ninety freshly extracted human maxillary central incisors were taken and stored in sterile saline (0.9% NaCl). Each tooth was decoronated 1 to 2 mm below CEJ using safe-sided diamond disk under water coolant so that length was standardized to 16 mm. Patency was confirmed with a 10 K-file and root canals were enlarged using ProTaper rotary instruments (Maillefer Dentsply, Ballaigues, Switzerland) until file F3 reached the working length (1 mm from apical foramen). Irrigation was done with 5% NaOCl (Vishal Dento care Pvt. Ltd., Ahmedabad, India) and 17% EDTA (Canalarge, Ammdent, Chandigarh, India) between each instrument. Finally canals were dried with paper points and obturation was done using Gutta percha cones (Maillefer Dentsply, Ballaigues, Switzerland) and AH plus sealer (Dentsply, Delhi, India) by lateral condensation method. The specimens were stored 37°C, at 100% relative humidity for a period of 72 hours so as the resin sealer sets completely.

Following this the coronal gutta percha removal was done with peeso-reamers #1 to 3 (Mani Inc., Tochigi, Japan) leaving 4 mm apical GP. Final post space preparation was done with #3 drill provided by the manufacturer of the Glassix post system (Swiss dental products of disinfection, Nordin, Switzerland). Finally post spaces were irrigated with distilled water and canals were dried.

Ninety glass fiber reinforced parallel and smooth posts (Glassix posts, Nr3- Ø 1.35, Swiss dental products of disinfection, Nordin, Switzerland) were taken and divided into

three groups (n=30) according to the surface treatment they received (37% H₃PO₄, 24% H₂O₂, distilled water). Each group was further divided into 3 sub-groups (n=10) based on the time period of etchant application (15 seconds, 30 seconds, 60 seconds). In H₃PO₄ group, gel (Etch gel, DentoInc., St. Paul, MN, USA) was applied over entire surface of the post; in other two groups posts were immersed in respective solutions; all posts were then treated with distilled water and air dried. Etched posts are then luted into the root canals using Rely X Unicem-2 Clicker (3M ESPE AG, Seefeld, Germany) and light activated through cervical portion for 40 second (Blue phase C8, Ivoclar vivident, Schaan, Liechtenstein). The specimens were then stored for 24 hours at 37°C in distilled water.

Following storage, each specimen was sectioned into six slices of 2 mm each using diamond saw under water coolant. The first two represent the coronal; second two middle and last two represent the apical regions of the root. The apical side of each specimen was marked with an indelible marker and each section was subjected to push out test from apical to coronal side in Universal testing machine (Autograph AG 15, Shimadzu, Kyoto, Japan) where the loading was performed at a cross head speed of 0.5 mm/min until post dislodged from the root slice. Schematic representation of entire push-out test is shown in Fig. 1.

Comparison of bond strength values were done using Repeated measures of ANOVA (IBM SPSS statistics for Windows, version 21.0, Armonk, NY, USA) and pair wise comparison was done by Tukey multiple post hoc test at a significance level at P<.05.

Twenty seven fiber posts were taken and divided into 3 groups (n=9) based on the surface treatment (H₃PO₄, H₂O₂, distilled water). Each group was subdivided into three (n=3) based on the time period of etchant application (15, 30, 60 seconds). After etching all specimens were cleaned ultrasonically for 5 min. in deionized water, followed by immersion in 96% ethanol and gently air dried. The posts were then gold sputtered (model JFC-1600,

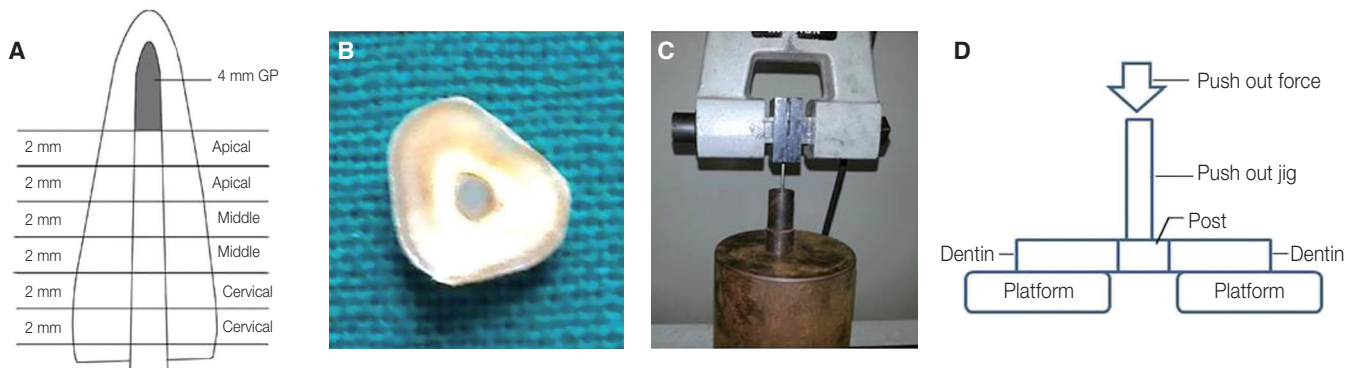


Fig. 1. (A) Sectioning of tooth into six 2 mm sections, (B) Thin slice (2 mm) showing the post at center adhered to dentin, (C) Specimen placement in Universal Testing Machine, (D) Pictorial representation of cross-section of push out test.

JEOL, Tokyo, Japan) and evaluated under SEM (model JSM-5600, JEOL, Tokyo, Japan).

Dislodged specimens after push out test were examined under stereomicroscope (Olympus Opto Systems, India Pvt Ltd., Noida, India) at $\times 4.5$ magnification and mode of failure was classified as adhesive between cement and dentin, adhesive between cement and post, cohesive within the cement.

RESULTS

The mean values of all the groups are shown in Fig. 2. Results showed highest bond strength values for H_3PO_4 group at 15 seconds and H_2O_2 group at 60 seconds at all the three root regions with no significant difference between those two groups ($P > .05$) (Table 1). Bond strength was greater in the coronal region followed by middle then apical irrespective of etchant and time period ($P < .05$).

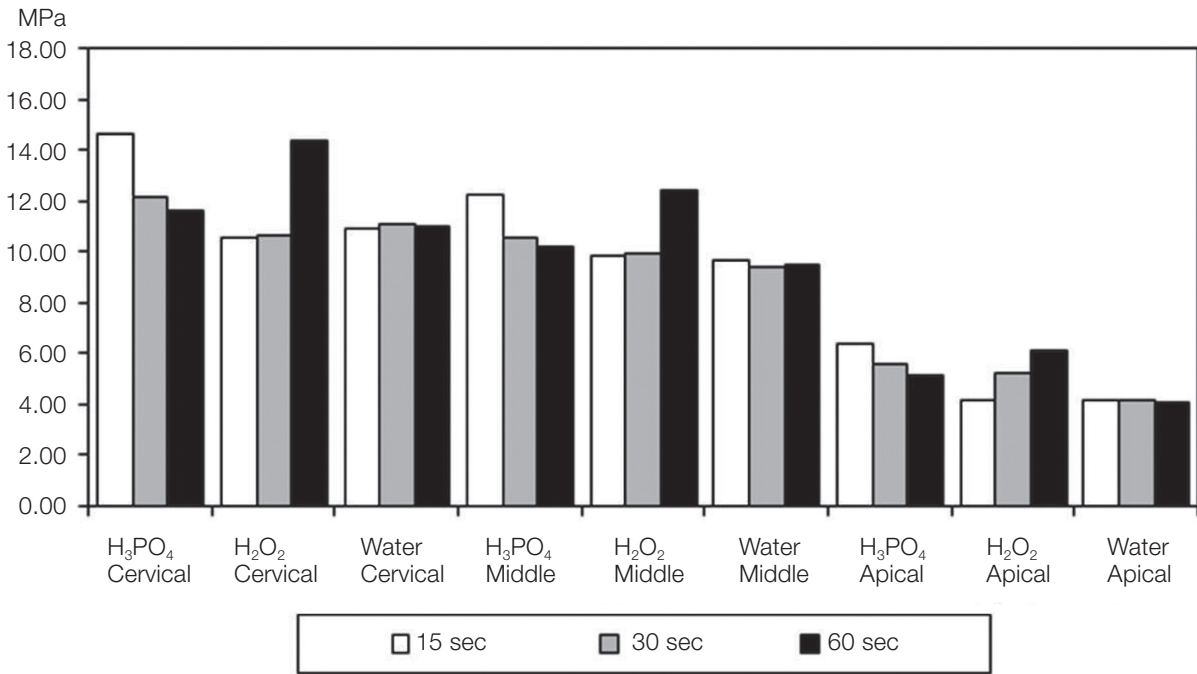


Fig. 2. Comparison of bond strength of different etchants at various time periods and at various root regions (Mean Values).

Table 1. Mean bond strength values of all groups in Mega Pascals

Etchant	Time period (Seconds)	Root region			Tukey*
		Cervical	Middle	Apical	
H_3PO_4	15	14.57 (0.62)	12.20 (0.24)	6.41 (0.32)	a
	30	12.14 (0.54)	10.57 (0.38)	5.60 (0.34)	b
	60	11.62 (0.24)	10.10 (0.65)	5.14 (0.30)	b
H_2O_2	15	10.57 (0.43)	9.86 (0.50)	4.17 (0.24)	c
	30	10.66 (0.34)	9.92 (0.58)	5.19 (0.22)	c
	60	14.38 (0.64)	12.40 (0.42)	6.07 (0.37)	a
Water	15	10.89 (0.80)	9.63 (0.46)	4.16 (0.24)	c
	30	11.09 (0.72)	9.38 (0.44)	4.15 (0.25)	c
	60	10.97 (0.51)	9.46 (0.38)	4.10 (0.26)	c
Tukey*		A	B	C	

* (Different letters - Capital letters - Comparison among columns, lower case letters - Comparison among rows) indicates statistical difference ($P < .05$).

SEM pictures of all the groups are shown in Fig. 3. Epoxy resin matrix was not removed in groups etched with distilled water at all time periods and in 15 seconds, 30 seconds H₂O₂ etched groups; complete removal of epoxy layer without damaging the fiber integrity was seen in 15 seconds H₃PO₄ etched group and 60 seconds H₂O₂ etched group. Some amount of damage to fiber integrity and complete damage to fiber integrity was noted in groups etched with H₃PO₄ for 30 and 60 seconds respectively.

The percentages of failures are shown in Table 2.

Adhesive failure between the cement and root canal dentin is the most common type of failure irrelevant to the type of surface treatment. Cohesive failure within the luting agent is the second most common type of failure in all the groups. Adhesive failure between cement and post are less compared to others. Fig. 4 shows various modes of failures. Among all groups, 60 seconds etched H₂O₂ group and 15 seconds etched H₃PO₄ group showed the least adhesive failures between cement and post, also highest number of cohesive failures within the cement.

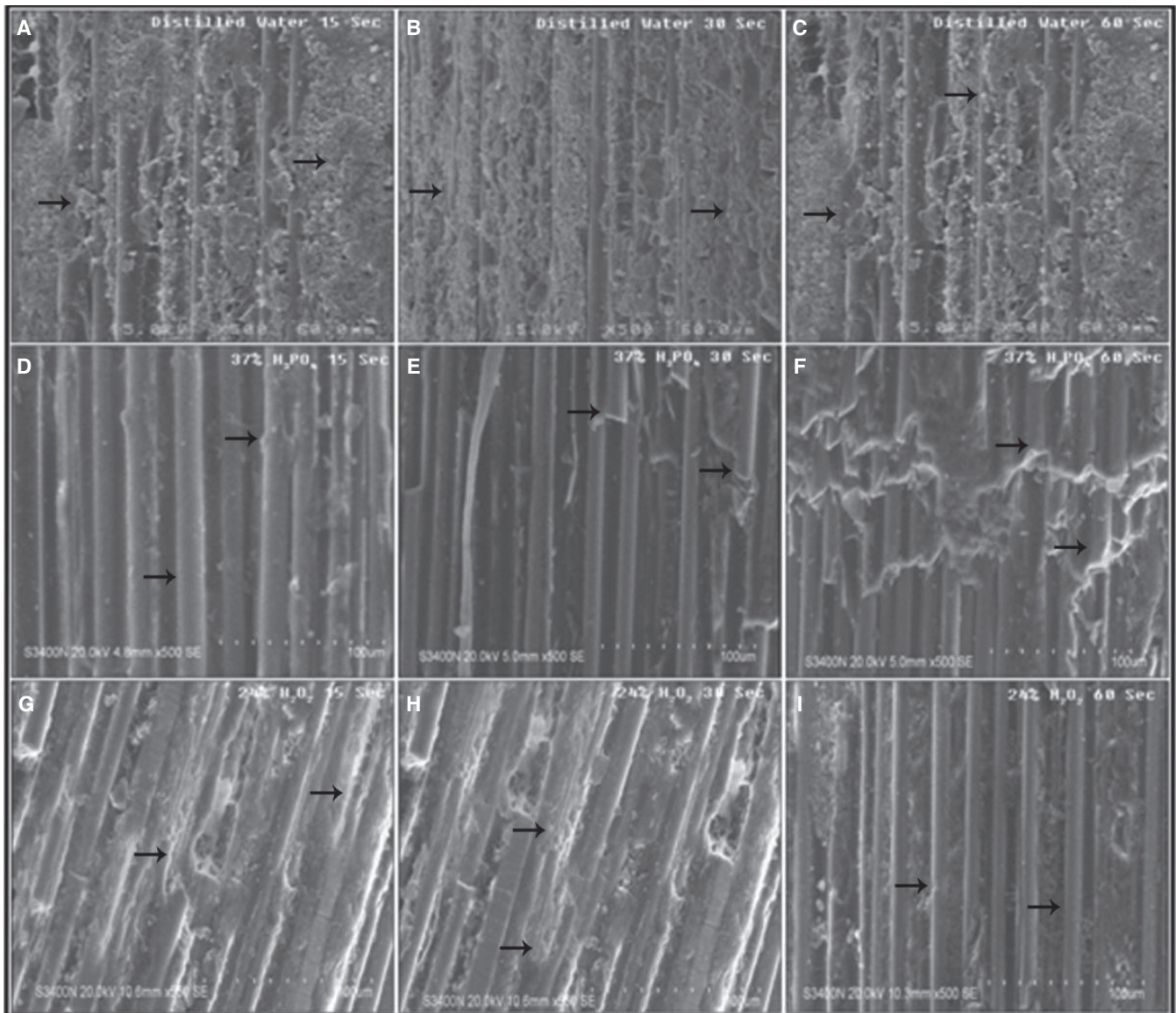


Fig. 3. SEM topographic images. (A) distilled water for 15 sec, (B) distilled water for 30 sec, (C) distilled water for 60 sec, (D) H₃PO₄ for 15 sec, (E) H₃PO₄ for 30 sec, (F) H₃PO₄ for 60 sec, (G) H₂O₂ for 15 sec, (H) H₂O₂ for 30 sec, (I) H₂O₂ for 60 sec. Arrows in image A, B, C, G, H shows the epoxy layer covering the fibers; arrows in image D, I show clearly exposed glass fibers with complete epoxy layer removal; arrows in image E, F shows the areas where damage to fiber integrity has occurred.

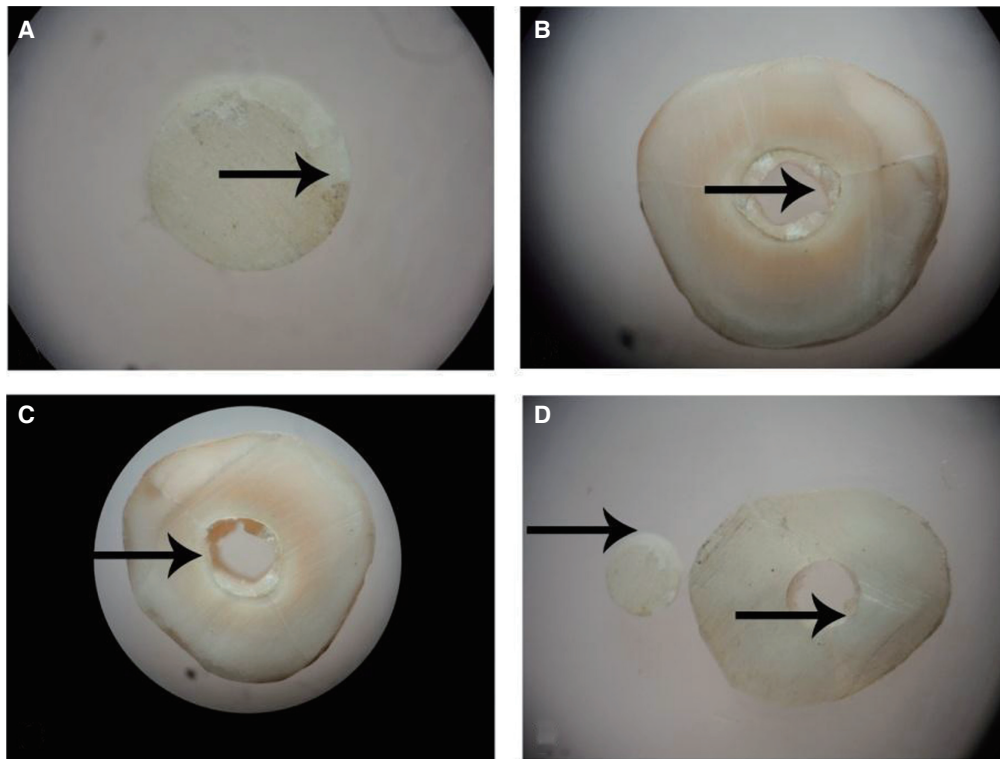


Fig. 4. Stereomicroscopic images (A) specimen before push out test, (B) adhesive failure between cement and post, (C) adhesive failure between luting agent and post, (D) cohesive failure within the cement. Arrows in image A shows the junction between luting agent and post, in image B shows the cement adhering to dentin, in image C represents dentin free of cement, in image D shows cement adhered both to the post and inner dentinal surface.

Table 2. Percentages of various modes of failures

Etchant	Time period (seconds)	Adhesive failure between cement and dentin (%)	Adhesive failure between cement and post (%)	Cohesive within the cement (%)
Distilled water	15	41.6	26.6	31.6
	30	43.3	28.3	28.3
	60	41.6	26.6	31.6
H ₂ O ₂	15	43.3	28.3	28.3
	30	41.6	26.6	31.6
	60	48.3	16.6	35.0
H ₃ PO ₄	15	46.6	18.3	35.0
	30	45.0	21.6	33.3
	60	41.6	26.6	31.6

DISCUSSION

Posts rely on strength of bond between different interfaces for their retention. Among them, the interface between root dentin and resin cement, post and resin cement, post and core has been the object of several studies involving bond strengths. Use of fiber posts has gained wide acceptance in the recent era. The main problem often encoun-

tered is incomplete adhesion of post to resin cement and also to the core. Many procedures have been put forward to enhance its adhesive effectiveness from simple etching procedures to sand blasting etc.

Results obtained in the present study clearly show the effectiveness of H₃PO₄ in etching fiber post and improving its adhesion to root canal dentin. In the present study H₂O₂ is used since it had been proven to be effective in etching

fiber post¹⁹ and improving the bond strength; H₃PO₄ is attempted to use in the present study since it is the commonly used chair side etchant.

Fiber posts are covered by epoxy resin, which has a high degree of conversion and few reactive sites to chemically bond to the resin cement.¹³ So to improve the bond between post and adhesive resin, removal of epoxy layer is essential without disturbing the fiber integrity. Unlike the use of corrosive forms of industrial epoxy resin etching techniques such as sodium ethoxide or potassium permanganate: H₂O₂ and H₃PO₄ etching provides clinically reliable methods to enhance micromechanical retention. Care should be taken not to etch for a longer time period since it affects the fiber integrity similarly to that of HF etching which had been proven to be an aggressive etching procedure.¹⁷ Dissolution of epoxy resin probably relies on an electrophilic attack of the H₂O₂ to the cured secondary amine. Thus the spaces created between the fibers provide conditions for the micro-mechanical interlocking of the resin with the post. Etching with H₂O₂ is a proved subject but studies are still under progress evaluating the efficacy of various concentrations and time periods in altering its etching capability.²⁰⁻²²

SEM images clearly depict the removal of epoxy layer with intact glass fibers in H₂O₂ and H₃PO₄ groups at 1 min and 15 seconds time intervals respectively. So the exposed glass fibers aid in improving the bond strength both by micro-mechanical interlocking and by chemical means due interaction between methacrylate group of luting agent and glass fibers.

Push out test was done since it produces shear stress at the interface between post and cement which mimics the stresses under clinical situation.²³

The use of adhesive resins have gained popularity from the past few years with their advantage of minimizing the number of steps required and ease of use, so we have selected a self adhesive resin cement. The adhesive properties are claimed to be based upon acidic monomers that demineralize and infiltrate the tooth substrate, and create micromechanical retention and chemical adhesion to hydroxyapatite by forming resin tags.

Our results are similar and support the study by Menezes *et al.*¹⁹ who proved H₂O₂ etching for one minute improves the bond strength of fiber post. Our results also showed highest bond strength values in the coronal followed by middle then apical root regions which is in accordance with previous studies.²³⁻²⁷ The reason thought for this might be the decrease in the tubule density from coronal to apical region as explained previously by Ferrari *et al.*²⁸ Most likely explanation for this higher resistance to dislodgement of post in coronal region and decreased resistance in apical region could be due to decreased effectiveness of curing light to penetrate from coronal region to the apex. Also the reduction in the bond strength may be related to the difficulties of moisture control in apical third of post space which may resist complete infiltration of the resin cement.

The type of failure modes reveal the least adhesive fail-

ures between the cement and post in 15 seconds H₃PO₄ group and 60 second H₂O₂ group indicating the possible improvement in bonding between post and cement after etching. Also due to increased adhesion of cement to the post there might be internal stress created within the cement at the time of loading causing increased number of cohesive failures within the cement in those groups.

CONCLUSION

Within the limitations of the present *in vitro* study it can be concluded that etching of fiber post is essential to improve adhesion. 37% H₃PO₄ etching for 15 second is a better and comfortable alternative to other methods in improving the adhesion of fiber post to root canal dentin.

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