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Effect of Methylene Chloride Surface Treatment on Surface Morphology and Flexural Properties of Glass Fibre-Reinforced Composite Post

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Abstract

Glass-fiber posts are nowadays routinely used in post-and-core systems. They have several advantages - improved light transmission throughout root canal, eradication of corrosive reactions that happen with metal alloy prefabricated posts, and simple removal if endodontic retreatment is required. The most important characteristic of fiber reinforced posts is their elastic modulus, which is closer to that of dentin, can provide the optimal stress distribution which reduces the possibility of root fracture. The limitations of bonding posts to root canal are usually related to polymerization effectiveness, difficult in creating a water-wet substrate, reduced number of dentinal tubules, deposition of cementum, and secondary dentin. In order to improve bond strength between fiber posts and the resin cement several surface treatment procedures have been investigated that involve use of mechanical, chemical or alternative etching techniques. Alternatively, many chemical solvents used for industrial applications (methylene chloride) have been introduced to improve adhesion. The etching effect of these chemicals depends on partial resinous matrix dissolution, exposing a greater surface area of fibers for silanization. All these surface treatments enhance the bond strength but, also changes post morphology. This leads to interference with fit, potentially reducing long term reliability of the restoration. Endodontic posts also need to withstand flexural loads that are applied during function. The various surface treatments may alter the chemical composition of the glass fiber post, leading to change in physical properties. The flexural strength property is checked to evaluate the resistance of the specimen to fracture. The purpose of this short-study is to evaluate the effect of methylene chloride surface treatment in comparison to different mechanical and chemical surface treatments on fiber-reinforced posts on its surface texture and flexural properties. 40 glass fiber-reinforced posts are randomly divided into 4 groups according to surface treatment: Group A (control; no treatment), Group B (silanization for 60sec), Group C (etching with 9% hydrofluoric acid for 1 min) and Group D (etching with dichloromethane for 10mins). The surface texture of the posts is characterized using scanning electron microscope (SEM). Flexural properties of the posts are assessed using a three-point bending test. The data is analysed using one-way ANOVA test.

Keywords: Bond strength, Elastic modulus, Flexural strength, Glass-fibre posts, Methylene chloride, Post-and-core, Surface texture.

Introduction

Endodontically treated teeth may be damaged by decay, excessive wear, or previous restorations, resulting in a lack of coronal tooth structure.[1] The restoration of endodontically treated teeth with a significant loss of coronal tooth structure may require the placement of a post to ensure adequate retention of a core foundation.[1-3] Cast metal posts and cores have been traditionally used in these situations to provide the necessary retention for the subsequent prosthodontic restoration.[1] Recently, the use of fiber posts in the restoration of endodontically treated teeth has increased in popularity.[4,5] Fiber posts are currently perceived as promising alternatives to cast metal posts, as their elastic moduli are similar to that of dentin, producing a favorable stress distribution and providing more esthetic outcomes for endodontically treated anterior teeth.[4,6] The use of tooth-colored fiber posts, together with the wide choice of composite resins available for core foundation restorations, allows better reproduction of the underlying natural tooth shade, resulting in a more esthetic solution.[7] In vitro and in vivo research indicates that failure of fiber post-and-core restorations often occurs because of debonding between the fiber post-resin and/or resin-root canal dentin interfaces as a result of inadequate bond strength.[2,4,8,9] The durability of a composite resin core restoration depends on the formation of a strong bond between the composite resin and the residual dentin, as well as between the composite resin and the fiber post, enabling the interface to effectively transfer stress under functional loading.[10] One difficulty with some of the available prefabricated fiber posts is that the polymer matrix between the post material fibers is highly cross-linked and, therefore, less reactive. This makes it difficult for these posts to bond to resin luting agents and tooth structure.[7,10,11] Amino-silane coupling agents are generally used as adhesion promoters in the presence of epoxy resin polymers. They provide a chemical bond between inorganic substrates of the post and the polymer. They also increase the surface wettability of the posts.[5,10] However, it was previously reported that silane does not bond well with the epoxy matrix.[4,6,10] In order to improve the bond strength between silane and the epoxy resin matrix, many chemical and mechanical surface treatment procedures for fiber posts have been investigated.[4,6,10,11] In previous studies, certain chemical solutions such as hydrogen peroxide (H_2O_2), potassium permanganate, hydrofluoric acid (HF), and silane coupling agents have been evaluated in an attempt to improve the bond strength between the fiber posts and the composite resin core materials.[2,7] An alternative approach for chemical surface treatment is the use of methylene chloride, which is reported to increase the bond strengths between acrylic resin denture base materials and acrylic resin repair materials.[12] This chemical agent changes the chemical features and surface morphology of denture base resins and increases their repair strength.[13] However, little is known of the physical and chemical effects of methylene chloride and hydrogen peroxide on quartz and glass fibers and the epoxy resin matrix of the fiber posts. Endodontic posts also need to withstand flexural loads that are applied during function. The various surface treatments may alter the chemical composition of the glass fiber post, leading to change in physical properties. The flexural strength property is checked to evaluate the resistance of the specimen to fracture. The purpose of this short-study is to evaluate the effect of methylene chloride surface treatment in comparison to different mechanical and chemical surface treatments on fiber-reinforced posts on its surface texture and flexural properties. 40 glass fiber-reinforced posts are randomly divided into 4 groups according to surface treatment: Group A (control; no treatment), Group B (silanization for 60sec), Group C (etching with 9% hydrofluoric acid for 1 min) and Group D (etching with dichloromethane for 10mins).

Materials and Methods

The present short study was conducted in Vokkaligara Sangha Dental College and Hospital, Bangaloe and all tests were conducted in Indian Institute of Science, Bangalore.

1.1. Material (Figure 1)

Glass fiber posts – 40 (AA Glass Fiber Posts)

Hydrogen Peroxide Solution

9% Hydrofluoric Acid (Porcelain Etch – Ultradent)

99% Dichloromethane

Universal Testing Machine (Mecmesin)

Optical Profilometer

Figure 1: Armamentarium used in the study

1.2. Sample Size Determination

Total no. of groups = 4

Max Mean Value (of the 4 groups) = 10.88

Min Mean Value (of the 4 groups) = 8.15

Mean Difference = 2.73

Pooled Standard Deviation = 1.47

α (Type I error) = 0.05

β (Type II error) = 0.20

1-β (Power of the test) = 0.80

Required sample size per group = 8 (rounded off to 10)

Total Sample Size = 10 x 4 = 40 (10 samples per group)

1.3. Methodology

40 glass fiber-reinforced posts are randomly divided into 4 groups according to surface treatment: (Figure 1)

Group A (control; no treatment)

Group B (etching with hydrogen peroxide for 60sec)

Group C (etching with 9% hydrofluoric acid for 1 min)

Group D (etching with dichloromethane for 10mins)

The preparation of the specimens were done in the following manner:

1.3.1. Control Group - No treatment

10 glass fibre-reinforced posts were left without any treatment

1.3.2. Etching with Hydrogen Peroxide

10 glass fibre-reinforced posts were taken and immersed in hydrogen peroxide solution for 60 seconds. They were removed after 60 seconds and dried with the help of a 3-way syringe. (Figure 2)

Figure 2: Treatment with hydrogen proxide.

1.3.3. Etching with 9% Hydrofluoric Acid

10 glass fibre-reinforced posts were taken and coated with 9% hydrofluoric acid for 30 seconds. They were rinsed with distilled water after 30 seconds and dried with the help of a 3-way syringe. (Figure 3).

Figure 3: Treatment with 9% hydrofluoric acid.

1.3.4. Etching with 99% Dichloromethane

10 glass fibre-reinforced posts were taken and immersed in 99% dichloromethane for 10 minutes. They were rinsed with distilled was after 10 minutes had elapsed and dried with the help of a 3-way syringe. (Figure 4)

Figure 4: Treatment with 99% Dichloromethane

1.3.5. Evaluation of Surface Roughness – Optical Profilometer

All the prepared specimens were then checked under the Optical Profilometer to generate a surface roughness quotient. (Figure 5-6) The surface roughness of each group was then calculated with the help of the following equation -

$$CLA = R_a = \frac{\sum A}{l} = \frac{A_1 + A_2 + \ldots + A_n}{l}$$

where,

Ra = mean roughness in micrometers

A= area under each crest and trough

l=length of the sample measured

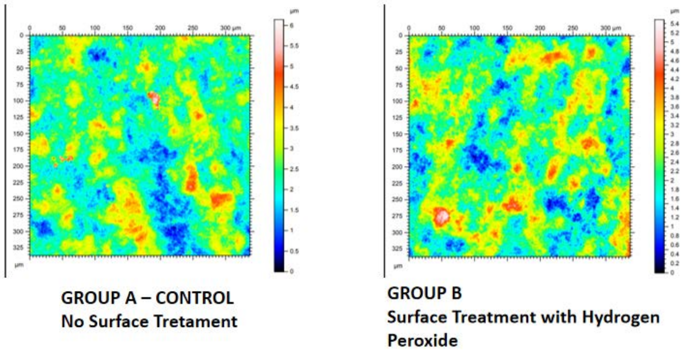


Figure 5: Surface Roughness of Group A and B

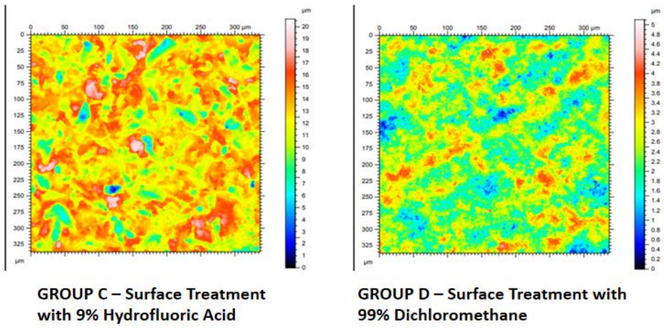


Figure 6: Surface roughness of Group C and D

1.3.6. Evaluation of Flexural Strength – 3 Point Bending Test

All the specimens were subjected to a three point bending test in a universal testing machine (Mecmesin) (Figure 7-8). This was done to calculate the flexural stregth of each specimen after being treated with various chemicals.

Figure 7: Universal Testing Machine (Mecmesin)

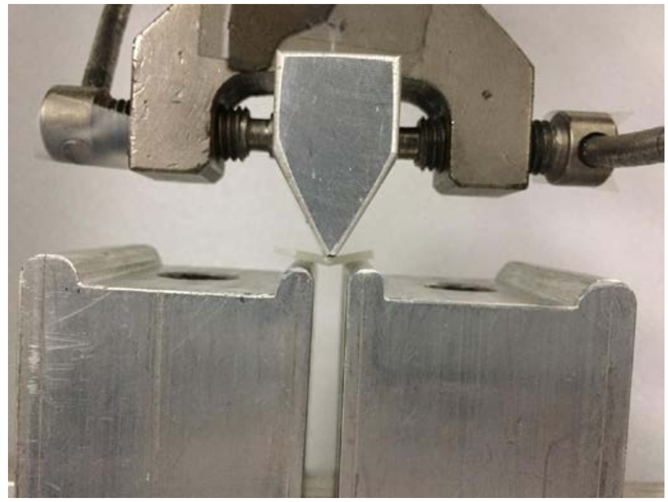


Figure 8: 3 point bending test

The results obtained from the 3 point bending test was tabulated as follows based on the values obtained.

Table 1: Average 3 point bending test measurements of each group.

Group A	186.83
Group B	196.17
Group C	152.30
Group D	203.22

Results

1.4. Statistical Analysis

Null Hypothesis: There is no significant difference in the mean flexural strength (N/mm2) recorded in the four groups i.e. $\mu1 = \mu2 = \mu3 = \mu4$

Alternate Hypothesis: There is a significant difference in the mean flexural strength (N/mm2) recorded in the four groups i.e. $\mu1 \neq \mu2 \neq \mu3 \neq \mu4$

Level of Significance: α=0.05

Statistical Technique Used: Analysis of Variance (ANOVA).

Decision Criterion: The decision criterion is to reject the null hypothesis if the p-value is less than 0.05. Otherwise we accept the null hypothesis. If there is a significant difference between the groups, we carry out multiple comparisons (post-hoc test) using Bonferroni test.

Computations: The following tables give us the results from ANOVA and the P-Value.

Table 2: Descriptives for Analysis

Group	n	Mean	Std Dev	SE of Mean	95% CI for Mean		Min	Max
					Lower Bound	Upper Bound		
Group A	10	186.83	5.74	1.81	182.73	190.94	176.64	194.38
Group B	10	196.17	15.10	4.78	185.36	206.97	174.76	218.43
Group C	10	152.30	20.47	6.47	137.66	166.94	112.97	188.67
Group D	10	203.22	12.67	4.01	194.15	212.28	182.95	223.17

Table 3: ANOVA (*denotes significant difference)

Source of Variation	df	Sum of Squares (SS)	M
Between Groups	3	15285.920	5(
Within Groups	36	7564.673	2
Total	39	22850.593	

In order to find out among which pair of groups there exist a significant difference, multiple comparisons were carried out using Bonferroni test.

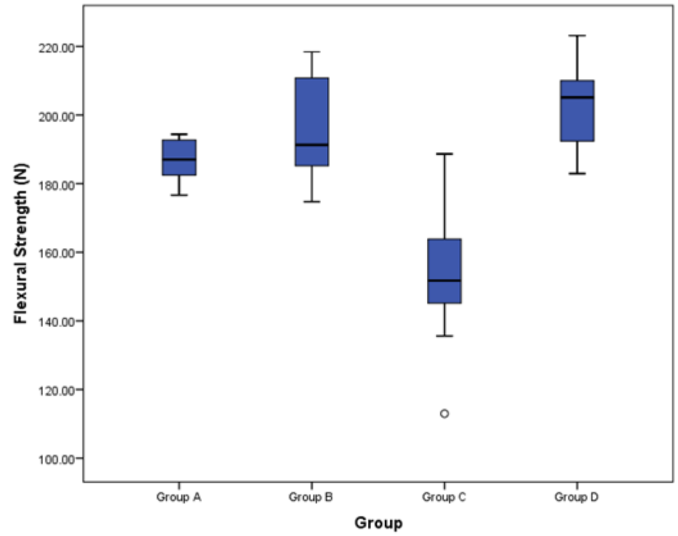
Table 4: Bonferroni Test

Group (I)	Group (J)	Mean Difference (I-J)	P-Value	95% CI for Mean Diff	
				Lower Bound	Upper Bound
Group A	Group B	-9.335	0.951	-27.43	8.76
	Group C	34.531	<0.001*	16.43	52.63
	Group D	-16.385	0.096	-34.48	1.71
Group B	Group A	9.335	0.951	-8.76	27.43
	Group C	43.866	<0.001*	25.77	61.97
	Group D	-7.050	1.000	-25.15	11.05
Group C	Group A	-34.531	<0.001*	-52.63	-16.43
	Group B	-43.866	<0.001*	-61.97	-25.77
	Group D	-50.916	<0.001*	-69.02	-32.82
Group D	Group A	16.385	0.096	-1.71	34.48
	Group B	7.050	1.000	-11.05	25.15
	Group C	50.916	<0.001*	32.82	69.02

Results

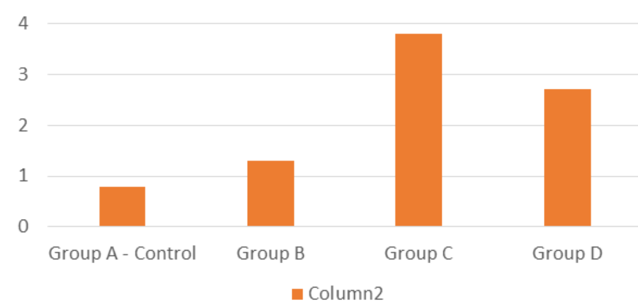
After all the statistical analysis was completed, it was found that, higher mean flexural strength was recorded in Group D (surface treatment with methylene chloride).

Graph 1 - Flexural Strength Result



The higher mean Surface roughness was recorded in Group C (surface treatment with 9% Hydrofluoric Acid)

Graph 2: Results of Surface Roughness



Discussion

Fiber posts are currently perceived as promising alternatives to cast metal posts, as their elastic moduli are similar to that of dentin, producing a favorable stress distribution and providing more esthetic outcomes for endodontically treated anterior teeth. In vitro and in vivo research indicates that failure of fiber post-and-core restorations often occurs because of debonding between the fibers post-resin and/or resin-root canal dentin interfaces as a result of inadequate bond strength.

One difficulty with some of the available prefabricated fiber posts is that the polymer matrix between the post material fibers is highly cross-linked and, therefore, less reactive. This makes it difficult for these posts to bond to resin luting agents and tooth structure.

Amino-silane coupling agents are used as adhesion promoters in the presence of epoxy resin polymers, by providing a chemical bond between inorganic substrates of the post and the polymer and increasing surface wettability. However, it has been reported that silane does not bond well with epoxy matrix.

Chemical treatments are aimed at roughening the post surface, thus enhancing the mechanical retention between the post and resin luting cement.The etching effect of hydrogen peroxide is to partially dissolve resin matrix, breaking epoxy resin bonds and exposing the surface of fibers to silanization through a mechanism of substrate oxidation.

All these surface treatments enhance bond strength but, changes post morphology. This leads to interference with fit, potentially reducing long-term reliability of the restoration. Endodontic posts also need to withstand flexural loads that are applied during function. The flexural strength property evaluates the resistance of a specimen to fracture.

Within the limitations of this study, it was found that, in the control group the flexural strength was found to be low (Graph 1) and the surface roughness was found to be the lowest (Graph 2). Etching with hydrogen peroxide showed high flexural strength (Graph 1) but low surface roughness (Graph 2). Etching with 9% hydrofluoric acid showed the lowest flexural strength (Graph 1) but highest surface roughness (Graph 2). The etching done with 99% Dichloromethane showed high flexural strength (Graph 1) and high surface roughness as well (Graph 2).

Further in-vitro and in-vivo studies should be conducted to prove the efficacy of the usage of 99% Dichloromethane as an ethcing agent in glass fibre-reinforced posts to improve bonding while not hampering its flexural capacity.

Conclusion

Within the limitations of this study, the following conclusions were drawn:-

- Surface pre-treatment methods showed significantly different ($p < 0.001$) flexural strength.
- The highest flexural strength values were obtained with 99% dichloromethane surface treatment, the lowest were obtained with 9% hydrofluoric acid surface treatment.
- The highest surface roughness was seen with 9% hydrofluoric acid and lowest with control group.
- The usage of 9% hydrofluoric acid as surface pretreatment of glass fiber posts should be avoided as it decreses the flexural properties.
- The use of dichloromethane can be advocated as it optimally increases surface roughness to aid in better bonding of fiber posts, but does not change the flexural properties of the posts.

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