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## Flexural Strength of Glass Fiber Post Following Conditioning with Various Chemical Solutions

### Running Title: Flexural Strength of Fiber Post with Different Chemical Treatments

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#### Abstract

The aim of this study was to assess various chemical solutions on the surfaces of glass fiber post could affect its flexural strength. A total of 80 Glass Fiber posts were divided into eight groups (n=10) based on surface treatment, untreated surface (control), 10% hydrofluoric acid (HF) for 1 minutes, 10% HF for 2 min, 24% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) for 4 min, 24% H<sub>2</sub>O<sub>2</sub> for 10 min, 35% H<sub>2</sub>O<sub>2</sub> for 4 min, 35% H<sub>2</sub>O<sub>2</sub> for 10 min, and chloroform solution for 1 hour. Fiber posts were immersed in the solutions for the required amount of time and then rinsed and dried. Fiber posts were loaded in three-point bending test using a mechanical testing machine, data recorded and Flexural strength ( $\sigma$ ) was measured. Statistical analysis revealed that the highest value of flexural strength was shown in the group treated with 24% H<sub>2</sub>O<sub>2</sub> for 4min followed by Chloroform group, so they have no significant effect on flexural strength. While the least value was shown in group treated with 10% HF for 2min. Treatment with HF significantly decrease flexural strength for both application times. Treatment with H<sub>2</sub>O<sub>2</sub> showed significant decrease only in group treated with 35% H<sub>2</sub>O<sub>2</sub> for 10 min. It was concluded that strong acid etching of HF and long application time of higher concentration of H<sub>2</sub>O<sub>2</sub> had adverse effect on fiber post flexural strength.

## Introduction

The glass fiber post can be used to increase the retention of coronal restorations. It offers a number of benefits over other types, including ease of usage, adequate mechanical strength, and elasticity modules that are more similar to those found in tooth dentin (1).

Glass fiber posts are made up of glass fibers in which a polymer resin matrix bonding them together. Most of the fiber reinforce posts have epoxy resin or bisphenol A- glycidyl methacrylate (bis-GMA) accompanied by some fillers (2).

One of the most common failure longevity of fiber posts restoration it is potentially depend on an adequate adhesive between luting cement and fiber post surface. Fiber posts core restoration decementation can occur between fiber posts and luting resin cement because the inadequate bonding strength (3), this is because the non-treated fiber surface is smooth and there is no mechanical interlock may form when resin cements are used (4).

To overcome this problem the glass fiber post subjected to variety of surface therapies with varied success, which include mechanical and chemical. Micromechanical surface treatment roughens the surface, increasing surface energy (5). Sandblasting is one of the mechanical techniques that shown to improve bond strength. This process may involve a complex laboratory treatment and may affect the post's properties (6). Chemical solution application on post surfaces is a less aggressive and less expensive way of post surface treatment that can lead to a preferred interaction with resin cement (7). Among the suggested chemical treatment to enhance bond strength are etching with hydrofluoric acid, hydrogen peroxide, at different concentration and durations (8). Although each treatment may enhance the adhesive process between the luting resin cement and exposed fiber, little information is known on whether the changes in the post structure would influence their mechanical characteristics. Such effect may be different with the type, concentration, and length of exposure to solutions (2). Flexural strength is one of the main properties of the glass fiber post because it will determine the capability of materials to bend before their fracture (9). The effectiveness of fiber post restoration etched with chemical solution to enhance their adhesion inside root canal system might affected when the occlusal force transferred by core build up material deep to the post. Few studies examined the effect of conditioning methods on the mechanical properties of fiber post. Braga et al, revealed that mechanical properties and bond strength of glass fiber post were not altered by surface treatment with

Hydrogen Peroxide (10). Aksornmuang et al, found that fiber post pretreatment with hydrofluoric acid and hydrogen peroxide had no adverse effect on its flexural properties, but high concentration of hydrofluoric acid cause more surface irregularities (11). The objective of the present research was to evaluate flexural strength of glass fiber post using a three-point bending assessment exposed to different chemical treatment of varies concentration and times.

### **Materials And Methods:**

The materials and parameters that used in the study are presented in Table (1). Eighty glass fiber post (Innodental CO., LTD, Seoul, Korea) were used in the study. Fiber post has parallel cylindrical shape, smooth surface with length of 20 mm and cross-sectional diameter of 1.3 mm. each post was washed with ethanol and dried well before treatment. They were haphazardly classified into eight groups depending in to the post surface treatment (n=10). Three chemical agents were used for surface treatment: Hydrofluoric acid (HF), Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>), and chloroform solution. There are no standard recommended parameters (concentration and application time) for using HF and HP, so different ratios were chosen depending on associated researches (11-13). Surface treatments were made as follow: Group 1-was left without treatment as control group. Group 2-surface treated with 10% hydrofluoric acid for about one minute (HF 10% -1 min). Group 3-surface treated with 10% hydrofluoric acid for two minutes (HF 10% -2 min). Group 4- surface treated with 24% hydrogen peroxide for about 4 minutes (H<sub>2</sub>O<sub>2</sub> 24%- 4 min). Group 5-surface treated with 24% H<sub>2</sub>O<sub>2</sub> for about 10 min (H<sub>2</sub>O<sub>2</sub> 24%- 10min). Group 6-surface treated with 35% H<sub>2</sub>O<sub>2</sub> for about 4 minutes (H<sub>2</sub>O<sub>2</sub> 35%- 4min). Group 7-surface treated with 35% H<sub>2</sub>O<sub>2</sub> for about 10 minutes (H<sub>2</sub>O<sub>2</sub> 35%-10 min). Group 8-surface treated with chloroform solution for about 1 hour. For all groups, surface treatments were performed by immersion of the fiber posts totally in each chemical

agent for the specified time for each group. After surface treatment, all fiber posts were cleaned using ultrasonic cleaner for about 5 minutes to remove any solution remnants and dried well using oil free compressed air spray for about 30 sec to be ready for flexural strength test.

### Three points flexural strength test

A three-point bending test was done to assess the flexural strength of all samples using a mechanical testing device (EMIC DL2000; EMIC, São José dos Pinhais, PR, Brazil). It has a 500 Newton load cell which is a head apply force and convert it to electrical signal so it can be measured, with crosshead speed of 0.5 mm/min following standard specification for this test (10, 14). The mechanical test was carried out by placing the specimen on two supports and the midpoint of sample positioned exactly in the middle of the space between them which is 9 mm (span distance of the test 9 mm).

Crosshead position was in the midpoint of the distance between supports so the load applied perpendicular to the long axis of the post at its midpoint as shown in Fig. (1). The maximum load for each post was measured until fracture. The flexural strength ( $\sigma$ ) was determined according to next equations : (10, 15)

$$\sigma = 8F_{Max} L / \pi d^3 \text{ (in MPa)}$$

Where F Max is the highest force (in Newtons) necessary to breaking the post, L is the distances length in (mm), d is the diameter of the fiber post in (mm). Sample before and after treatment and testing is shown in Fig. (2).

Data were collected and statistical analysis was accomplished by using one-way analysis of variance (ANOVA) test which performed to investigate if there is significant difference between groups, followed by multiple comparison Tukey HSD (Honestly Significant Difference) test to examine the differences by comparing between group means, this was performed by utilizing IBM SPSS statistical software program (24.0) at  $p$ -value  $<.005$ .

### Results:

Descriptive statistics including mean and standard deviation are presented in Table (2). The highest value of flexural strength between tested groups (after control) was shown in group treated with (H<sub>2</sub>O<sub>2</sub> 24%- 4 min), while the lowest value was shown in group treated with (HF10%-2 min). ANOVA test displayed that there were statistically significant differences between groups at  $p$ -value  $<.005$  as seen in Table (3). Multiple comparisons Tukey test as seen in Table (4) revealed significant decrease in the mean of flexural strength in both groups that treated with hydrofluoric acid (HF10% - 1 min and HF10%-2 min) and group treated with (H<sub>2</sub>O<sub>2</sub>35%- 10 min) as compared to control, treatment group of (HF10%-2 min) showed significant difference from all other groups. For the other surface treatment groups which include treatment with 24% H<sub>2</sub>O<sub>2</sub> for both application times (H<sub>2</sub>O<sub>2</sub> 24%- 4 min) (H<sub>2</sub>O<sub>2</sub> 24%- 10min), group of (H<sub>2</sub>O<sub>2</sub>35%- 4min), and chloroform solution they showed no significant effect on the flexural strength as there were no statistically difference between them and control group. Correlation between surface treatment and flexural strength is shown in Fig (3).

### Discussion:

Several treatments have been suggested to alter the surface of fiber posts in order to enhance their adhesion ability and clinical effectiveness. These compounds, on the other hand, may have a dual effect on the post surface. Although chemicals eliminate detritus from the surface, exposing the glass fibers and increasing contact with the silane coupling agent; but moreover, the chemicals have the potential to destroy the polymeric epoxy phase on the post surface. Regardless of the fact that many manufacturers advocate etching fiber posts before usage, there is no standard process or chemical for this process, and dentists often clean post surfaces with chemicals found in dental clinics (10).

The effect of these protocols on fiber post mechanical properties such as strength

should be considered to obtain optimum clinical success.

In clinical practice, broken posts or dislodgment of post from resin matrix were first related to restorative dislocation and failures, hence various studies employed the maximum load to determine fiber post flexural strength (11).

The fracture resistant is assessed by the flexure strength value, greater value mean that a material has high fracture resistance. The flexural strength influenced by the samples shape and is determined by the maximum load a sample can withstand (16).

Nowadays in dental clinic some of the most commonly used etching agents for glass fiber post are hydrofluoric acid and hydrogen peroxide. This study was performed to examine the effect of these agent and if the concentration or time of application may have negative effect on fiber post strength, organic solvent like chloroform solution also studied.

The research results showed that posts soak with (10%) hydrofluoric acid for both application periods (1 min and 2 min) lead to decrease flexural strength of fiber post, especially with application time (2 min) which showed the least value of flexural strength. This result may be attributed to the mechanism of action of HF on fiber post. The etching of fiber posts with hydrofluoric acid creates microscopic gaps between the exposing fiber, hence employing this acid at a high concentration might be harmful to the post fibers. The acid's action was shown to be time depending on and affected by post-composition, the quartz fibers seeming to be stronger than glass fibers. This is because hydrofluoric acid has very corrosive action on the matrix's glass phase (17). According to GULER et al., 2012, in spite the known increase in bond strength, etching with hydrofluoric acid may cause high alteration of the epoxy resin matrix, this can severely destroy fibers and compromise the posts' physical and mechanical characteristics (18). It was found that glass fiber was wiped out when post etched with 10% HF for 60 and 120 seconds, this acid had removed all fibers from surface and making grooves or crack lines of fibers on the particle filled resin

(11). On the other hand, some studies like D'arcangelo et al, Elnaghy and Elsaka found that surface treatment with HF had no effect on fiber post strength (19-20), this may be because the lower concentrations of HF or less time which they used in their studies. So, the use of aggressive acid solutions for long etching time should be avoided to reduce or prevent further damaging of the fibers. The results showed that H<sub>2</sub>O<sub>2</sub> had less negative effect on flexural strength especially when it used with less concentration (24%) for both application times (4 and 10 min). this may be due to etching of H<sub>2</sub>O<sub>2</sub> which work on epoxy resin matrix instead of post fibers. H<sub>2</sub>O<sub>2</sub> has the capacity to partly breakdown the resin matrix, destroying the epoxy resin connections and exposing a large number of glass fibers inside the post without causing damage, this allows chemical optimization by reaction of fibers with silane coupling agents. This mechanism represents H<sub>2</sub>O<sub>2</sub>'s selectivity. The mechanical characteristics of the posts are unaffected because the fibers are undamaged (10). However, there is no stander for both chemical concentration and application time. Flexural strength showed significant lower value only when fiber posts etched with high concentration 35% H<sub>2</sub>O<sub>2</sub> for long etching time of 10 min. According to Menezes et al, 2011 either 24 % or 50 % of H<sub>2</sub>O<sub>2</sub> were capable to partly degrade the epoxy resin and exposing the glass fibers after exposure time of a 1-minute. Although mild etching caused by 24 % H<sub>2</sub>O<sub>2</sub> after 1 min exposure, binding strength was comparable to that produced with greater concentrations or longer application durations (21). Chloroform is a strong organic solution that can improve adhering ability of the fiber post 20% according to S.E. Sultan et al as this solvent clean the post surfaces debris and forming a very well surface pattern with exposed fibers so increase their availability for micro retention with penetrating of bonding substance (13). In this present study chloroform solution was chosen to use as a surface treatment, and according to results it was found that chloroform had no significant effect on fiber post flexural

strength. This result agrees with Cheleux et al., 2007 outcome who evaluated the influence of several surface treatments on the bond strength and flexural strength of fiber posts. Chloroform was one of these treatments, and the surface treatment had no effect on the flexural characteristics (22).

**Conclusion:**

Within the limitation of the present study, it was found that Strong acid etching like

10% HF had adverse effect on flexural strength of glass fiber post for both application times. 24% H<sub>2</sub>O<sub>2</sub> and chloroform can be used for fiber post surface treatment without decreasing its flexural strength, but higher concentrations of H<sub>2</sub>O<sub>2</sub> (35%) require shorter times to avoid affecting its mechanical properties.



Fig. (1): Specimen under load in universal testing machine

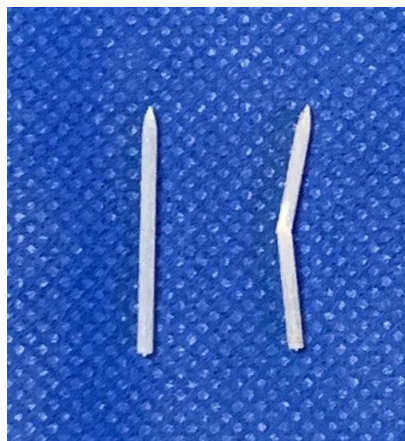


Fig (2): Specimen before and after flexural testing

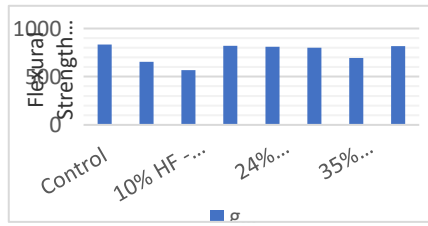


Fig. (3): Chart display the correlation between surface treatment groups and flexural strength

Table (1): Materials and parameters used in the study

Glass fiber post	Parallel, smooth, 20 mm length, 1.3 mm cross section	
Chemical solutions	concentration	Application time
Hydrofluoric acid (HF)	10%	1 min, 2 min
Hydrogen Peroxide (HP)	24%, 35 %	4 min, 10 min
Chloroform solution	Pure organic solvent	1 hour

Table (2): Descriptive statistics of flexural strength in MPa for all groups arranged from highest to lowest values

Group	N	Mean	Std. Deviation	Std. Error	Minimum	Maximum
Control	10	834.060	44.84	14.18	745.80	902.60
24% H <sub>2</sub> O <sub>2</sub> – 4 min	10	821.470	38.56	12.19	763.90	902.40
Chloroform	10	816.640	40.98	12.95	770.10	889.80
24% H <sub>2</sub> O <sub>2</sub> – 10 min	10	810.020	48.33	15.28	749.20	901.40
35% H <sub>2</sub> O <sub>2</sub> – 4 min	10	800.970	38.61	12.21	739.70	871.50
35% H <sub>2</sub> O <sub>2</sub> – 10 min	10	693.160	69.70	22.04	602.70	789.50
10% HF – 1 min	10	654.880	58.20	18.40	578.30	748.10
10% HF - 2 min	10	568.690	52.27	16.53	492.30	644.60
Total	80	749.986	104.30	11.66	492.30	902.60

Table (3): One Way - ANOVA test for all experimental groups

	Sum of Squares	df*	Mean Square	F-value	P-value**
Between Groups	679671.900	7	97095.986	38.868	.000
Within Groups	179864.315	72	2498.115		
Total	859536.215	79			

\* df= degree of freedom

\*\* P&lt; 0.05 mean significant difference exist

Table (4): Multiple Comparisons (Tukey HSD) analysis of the mean  $\pm$  standard deviation values of flexural strength in (MPa), subset for alpha = 0.05

Group	N	Mean $\pm$ SD (MPa)	Tukey grouping
Control	10	834.060 $\pm$ 44.8	A*
24% H <sub>2</sub> O <sub>2</sub> – 4 min	10	821.470 $\pm$ 38.5	A
Chloroform	10	816.640 $\pm$ 40.9	A
24% H <sub>2</sub> O <sub>2</sub> – 10 min	10	810.020 $\pm$ 48.3	A
35% H <sub>2</sub> O <sub>2</sub> – 4 min	10	800.970 $\pm$ 38.6	A
35% H <sub>2</sub> O <sub>2</sub> – 10 min	10	693.160 $\pm$ 69.7	B
10% HF – 1 min	10	654.880 $\pm$ 58.2	B
10% HF - 2 min	10	568.690 $\pm$ 52.2	C
Sig		0.000	

\* Mean values with the same letter indicate no significant differences (p &gt;0.05), while mean values with different letters indicate significant difference between them (p&lt;0.05)

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