

COMPARATIVE EVALUATION OF PULL-OUT BOND STRENGTH OF FIBER POST TO ROOT DENTIN PRETREATED WITH TWO SYNTHETIC AND TWO NATURAL ANTIOXIDANTS: AN IN-VITRO STUDY.

RUNNING TITLE : BOND STRENGTH OF FIBER POST TO ROOT DENTIN WITH ANTIOXIDANT PRETREATMENTS.

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Abstract

Objective: To evaluate the pull-out bond strength of fiber post to root dentin pretreated with two synthetic antioxidants (2% chlorhexidine (CHX) and 10% sodium ascorbate (SA)) and two natural antioxidants (6.5% proanthocyanidin (PAC) and 0.05% Epigallocatechin-3-gallate (EGCG)).

Methods: Forty extracted mandibular premolar teeth were decoronated to a length of 14 mm. Root canals were prepared using rotary NiTi files and obturated with gutta percha and resin sealer. Post space was prepared using peeso reamers, retaining 5 mm of apical gutta percha. Following smear layer removal and acid etching of the post space, samples were randomly assigned to 4 groups based on the dentine pretreatment, namely 10% SA (group I), 2% CHX (group II), 6.5% PAC (group III), and 0.05% EGCG (group IV). A glass fiber post was luted using a dual cure adhesive and luting cement. Pull out bond strength was evaluated using a Universal

testing machine. Data was analysed with one-way ANOVA 'F' test and followed by Turkey's post hoc test for pair wise intergroup comparison between each group.

Results: Among all the tested groups, 6.5% PAC showed the highest pull-out bond strength as compared to other groups. ($P < 0.05$)

Conclusion: Natural antioxidants performed better than synthetic antioxidants in increasing pull-out bond strength of fiber post to root dentin, with 6.5% PAC group showed the best results as compared to other tested groups.

Keywords: Collagen, dentin, glass fiber post, proanthocyanidin, pull-out bond strength

Introduction

Endodontically treated teeth are more prone to fracture than vital teeth.¹

Prefabricated posts, particularly glass fiber posts (GFPs), have gained popularity due to their elastic modulus similar to human root dentine, reducing the risk of vertical root fractures.^{2,3}

The aim of this study was to evaluate and compare the pull-out bond strength of fiber post to root dentin after surface treatment of radicular dentin with different antioxidants. The null hypothesis was that dentin pretreatment with SA, CHX, PAC and ECCG will not influence the bond strength of fiber post to root dentin.

Materials and Methods

Preparation of 10% SA solution: 10 gm of sodium ascorbate (Bio Balance) was weighed using digital weighing balance, then dissolved and mixed in 100ml of distilled water at room temperature.

Preparation of 6.5% PAC solution: 6.5 gm of grape seed derived PAC powder (INLIFE, Group since 1973) was weighed using digital weighing balance and dissolved in 100ml of distilled water.

Preparation of 0.05% EGCG solution: The EGCG solution was prepared at 0.05% w/v concentration by dissolving 0.05 gm of green tea powder (INLIFE, Group since 1973) in 100ml of distilled water.

Sample Preparation

A power analysis was established by G*Power version 3.0.1 (Franz Faul universitat, Kiel, Germany). Total minimum calculated sample size of 40 samples (10 samples per group; total 4 groups) would yield 80% power to detect significant differences, with effect size of 0.58 and significance level at 0.05.

Forty intact mandibular premolar human teeth, extracted for orthodontic or periodontal reasons, were collected and stored in 10% buffered formalin until use. The methodology was presented to the Institutional ethics committee and approval was obtained (BV(DU)MC&H/Sangli/IEC/Dissertation2021-22/D-69). The teeth were decoronated at the cemento-enamel junction and the root length was standardised to 14 mm using a diamond disc under water coolant. A size 10 K-file (Mani, Inc., Tochigi, Japan) was used to establish working length 1 mm short of the apex. Cleaning and shaping was done at the working length using rotary NiTi files (Protaper Universal, Dentsply, Maillefer, Ballaigues, Switzerland) following the sequence S1, S2, F1, F2 and F3. Canals were then irrigated with 2 mL of 3% sodium hypochlorite (NaOCl) (Sambhav agencies, Vita, India) between each change of instrument. Following instrumentation, the smear layer was removed using 5 mL of 17% EDTA (Waldent Innovations Pvt. Ltd) for 1 min and flushed with saline and then irrigation with 2 mL of 3% NaOCl for 30 sec followed by final flush with 0.9% Saline was done.

Obturation was completed using F3 gutta-percha cones (SureEndo, South Korea) and AH plus sealer (Dentsply, DeTrey, USA). After 12 hrs, post space preparation was created with up to #3 peeso reamer (Mani Medical Hanoi Co., Ltd., Thai Nguyen Province, Vietnam), retaining the apical 5 mm of gutta-percha. The smear layer was removed using 5 mL of 17% EDTA for 1 min and the post space was etched with 37% phosphoric acid (Prime Dental Products Pvt Ltd) for 30 sec. The etchant was rinsed off with water and the excess water was dried with paper points. The specimens were randomly assigned to four groups ($n=10$) based on the pretreatment

of root canal dentin and were irrigated with 5 mL of the respective experimental solutions using a side vented needle. The solution was left in place for 5 min, with intermittent agitation using a microbrush. At the end of the exposure time, the solutions were rinsed off and the canals were dried with paper points. All the prepared specimens were mounted upright on acrylic blocks. Then the fiber post was luted using a dual cure adhesive and luting cement (luxacore z DMG) and light cured for 20 sec. The samples were later stored in distilled water for 24 hours.

Pull Out Bond Strength Testing

Pull-out bond strength was evaluated using a Universal testing machine after 24 hours. The posts were fixed in a metallic grab attached to the superior base of the universal testing machine that moved upwards at 1 mm/min until displacement of the intracanal post. The maximum force for post displacement (in kN) was transformed into tension (in MPa) by dividing the force by the adhesive.

Statistical Analysis

Statistical analysis was performed using SPSS, expressing descriptive data as mean and standard deviation, with intergroup comparison conducted through One-way ANOVA and Turkey's post hoc test. Statistical analysis was carried out to compare the bond strengths among the groups, with significance set at a 95% confidence interval and a 5% level of significance.

Results

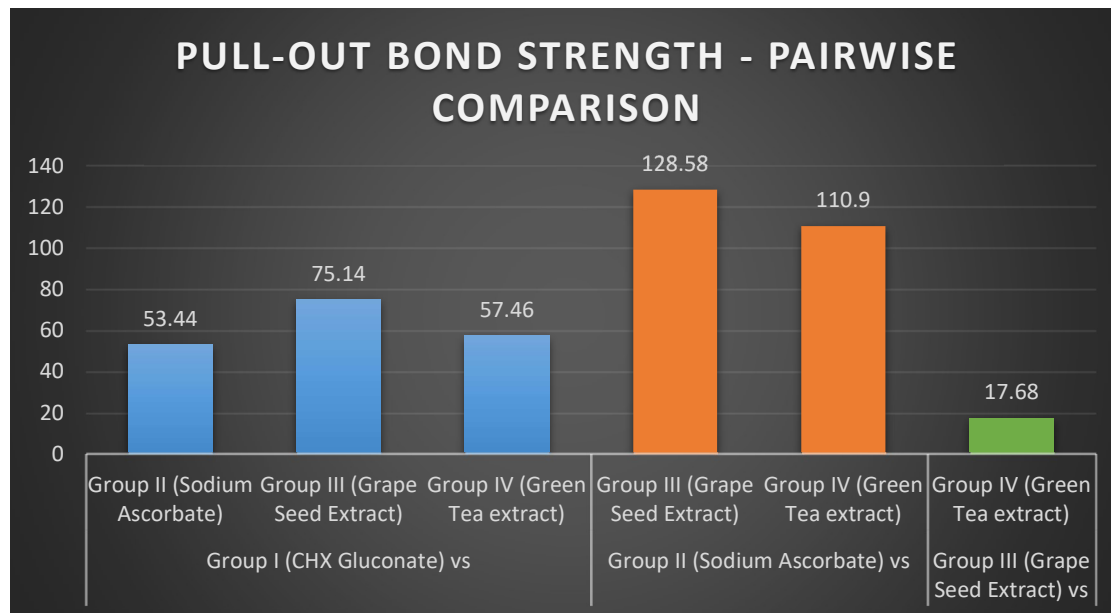
According to table no.1PAC (Group III) exhibited the highest pull-out bond strength, averaging 257.335, followed by EGCG (Group IV) at 239.65 and 2% CHX(Group I) at 182.19. SA (Group II) demonstrated the lowest bond strength with an average of 128.75.

Table 1: Descriptive statistics of pull-out bond strength of fiber post to root dentin pre-treated with two synthetic and two natural antioxidants in Newton (N)

	Mean	SD	SE	Minimum	Maximum
Group I (CHX)	182.1	18.0	5.69	138.5	201.4
Group II (SA)	128.7	29.41	9.3	51.9	154.8
Group III (PAC)	257.34	22.83	7.22	221.0	298.7
Group IV (EGCG)	239.65	17.19	5.43	209.7	265.9

According to fig no.1 in pairwise comparisons, Group III showed significantly higher bond strength than Groups I, II & IV. Conversely, Group IV displayed significantly higher bond strength compared to Groups II, I. However, there was no statistically significant difference between Group III and Group IV. Overall, the differences among Groups I, II, and III were highly statistically significant with a p-value of less than 0.001.

Fig 1: Intergroup group comparison evaluation of pull-out bond strength of fiber post to root dentin pre-treated with two synthetic and two natural antioxidants



Discussion

Endodontically treated teeth, prone to fracture, often require reinforcement with intracanal posts for stability during restoration. Glass fiber posts are favoured for their similar elastic modulus to dentine, reducing the risk of vertical root fracture.^{4,5}

Resin cement, applied with either total-etch or self-etch bonding systems, secures the post, forming a cohesive interface crucial for restoration longevity. Dual-cure or self-cure adhesives are preferred over light-curing systems to ensure polymerization quality, addressing concerns about light penetration. Contemporary resin luting cements chemically bond with the post and composite core, while also micromechanically adhering to dentine, creating a durable, unified structure, with the hybrid layer being its weakest link.⁶

The stability of adhesive dentin-cement interfaces diminishes over time due to hydrolytic degradation of resin within the hybrid layer and enzymatic degradation of collagen, catalysed by endogenous proteases like matrix metalloproteinases (MMPs) and cysteine cathepsins (CTs).⁷ To address this, recent recommendations suggest the use of antioxidants and reducing agents to enhance the longevity of resin-dentin bonds and reduce microleakage. These agents, derived from natural or synthetic sources, act as polymerization-facilitating agents and cross-linkers, effectively stabilizing the interface. Natural cross-linking agents such as tannic acid, genipin, ascorbic acid, and PACs have demonstrated promising results in crosslinking collagen and inhibiting enzymatic degradation, thus improving the durability of resin-dentin bonds.^{8,9}

In this study, mandibular first premolars were chosen due to their frequent extraction for orthodontic reasons, facilitating the study design. Standardization was achieved by decoronating all teeth to a root length of 14mm to minimize sample variability.

In this study, cleaning and shaping of the root canals were conducted up to F3 rotary instruments using a crown-down technique. The choice of instrumentation was based on observations by Baratto-Filho et al. (2009), who found that the cleaning ability of ProTaper™ instruments (F1, F2, and F3) correlated with their diameter. A 30-gauge needle with a tip diameter of 0.30 mm was employed, alongside canal instrumentation up to size F3 (Protaper Universal), corresponding to 0.30 mm.

Chemomechanical preparation involved the use of 3% NaOCl between each instrument change, effectively removing organic contents and smear layer. Sodium hypochlorite (NaOCl) was selected for its antimicrobial

and tissue-dissolving properties, ensuring thorough disinfection within the root canal system. Calt and Serper (2002) compared the effects of 17% EDTA on smear layer removal and on the structure of dentin, after 1 and 10 minutes of application. Their results showed that 1 minute EDTA irrigation is effective in removing the smear layer whereas a 10 minutes application caused excessive peritubular and intertubular dentinal erosion.¹⁰

Additionally, obturation was performed using the cold lateral compaction technique with F3 gutta percha cones, a widely adopted method due to its controlled placement and cost-effectiveness.¹¹ AH Plus resin sealer was utilized for its superior adaptability to root canal walls and recognized as a gold standard for its physical, chemical, and biological properties.^{12,13} Post space preparation followed, with 17% EDTA utilized for smear layer removal before etchant application on radicular dentin.

Various techniques have been proposed to preserve resin-dentin bond integrity and prevent collagen fibril degradation. Chlorhexidine (CHX) acts as a potent MMP-inhibitor, preventing hybrid layer degradation by inhibiting proteolytic activity in both sound and carious dentin. Crosslinking agents like tannic acid, glutaraldehyde (GA), and grape seed extract (GSE) increase collagen stability and bond strength by decreasing enzymatic degradation rates. Epigallocatechin gallate (EGCG), abundant in green tea, has been shown to reduce dentin demineralization possibly by inhibiting MMPs, offering further protection to the resin-dentin interface.¹⁴ In this study, PAC (Group III) exhibited the highest pull-out bond strength, averaging 257.335 Newtons (N), followed by EGCG (Group IV) and 2% CHX (Group I) with averages of 239.65N and 182.19N, respectively. Conversely, SA (Group II) demonstrated the lowest pull-out bond strength with an average of 128.75N. The observed efficacy of grape seed extract and green tea extract can be attributed to their polyphenolic bioflavonoid content, particularly proanthocyanidins (PACs), which serve as natural cross-linkers for stabilizing collagen matrices. PACs interact with collagen proteins, forming insoluble complexes and facilitating the activity of proline hydroxylase enzymes crucial for collagen biosynthesis.

Polyphenolic bioflavonoids, particularly proanthocyanidins (PACs), exert multifaceted effects on dentin collagen integrity and adhesion. PACs not only serve as cross-linkers, enhancing collagen network density and stability, but also inhibit enzymatic degradation by downregulating matrix metalloproteinases (MMPs) and cysteine cathepsins.^{2,15} Epasinghe et al. (2013) demonstrated PAC's ability to deactivate various MMPs and cysteine cathepsins, contributing to the preservation of dentin collagen within the hybrid layer.¹⁶ Cecchin et al. (2015) found that PAC-rich grape seed extract improved long-term bond strength between fiber posts and root dentin.¹⁷ Acid etching, commonly used in dentin treatment, exposes collagen fibers but may compromise bonding due to MMP activity. However, agents like chlorhexidine (CHX) and epigallocatechin gallate (EGCG) mitigate this by dampening dentin substrate, preventing collagen collapse, enhancing adhesive penetration, and promoting uniform hybrid layer formation.⁸

The study suggests that while sodium ascorbate (SA) did not impact immediate bond strength, it potentially reduces it due to competition with MDP monomers, forming stable complexes with calcium ions and hydroxyapatite. Despite SA's inhibitory effect on dentin MMP-2 activity, it showed no significant improvement in resin-dentin bond durability, possibly due to its lower MMP inhibitory potential and lack of cross-linking ability compared to other dentin biomodifiers.¹⁸ Similarly, 0.1% EGCG pre-treatment exhibited higher immediate bond strength compared to 10% SA, although not statistically significant, aligning with previous findings.¹⁹ Conversely, Monteiro et al. reported adverse effects on immediate bond strength with green tea solution, possibly attributed to higher concentration (2%) and impurities in the extract.²⁰

Overall, 6.5% proanthocyanidin (PAC) demonstrated the highest pull-out bond strength, indicating natural antioxidants potency over synthetic counterparts in enhancing fiber post-root dentin bond strength.

The present study evaluated only the short-term effect of SA, CHX, PAC and EGCG under in vitro conditions in root dentin. Future studies should focus on long-term effects of these agents and clinical trials of their usage in root dentine bonding. Various other concentrations of these antioxidants with different application times also needed to be tested.

Conclusion

Under the limitations of this study, it can be concluded that among all the tested groups, Natural antioxidants performed better than synthetic antioxidants in increasing pull-out bond strength of fiber post to root dentin,

with PAC (Group III) showed the best results as compared to other tested groups.

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