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Effect of photosensitizers on modulating bond strength of different luting cements used for cementation of resin modified composite fiber posts

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Abstract. – **OBJECTIVE:** The aim of the study was to evaluate the tensile BS of FPs luted with conventional and self-adhesive resin modified GIC cement on disinfection of post-space radicular dentin with various photo-activated photosensitizer (chitosan, TB and Clp6).

MATERIALS AND METHODS: Sixty-six orthodontically extracted mandibular pre-molars were selected on specific exclusion/inclusion criteria, trailed by thorough debridement of plaque/calculus and disinfected by immersion in Chloramine T trihydrate solution for 48 hrs. The selected teeth were sectioned from the cement-enamel junction removing the crown. The radicular region of the samples was entrenched perpendicularly in heat cure resin modified acrylic. The pulpal tissue was detached using K-files trailed by canal shaping with ProTaper Next rotary NiTi (Dentsply Sirona, Gloucestershire, UK) files under continuous irrigation with NaOCl. The canals were dried using paper points trailed by obturation by Pro Taper Next Obturators gutta-percha points (Dentsply, UK) coated with ADSEAL (Meta Biomed, Chungcheongbuk-do, South Korea). After incubation for seven days in an environment mimicking oral cavity, post space was created using peso reamers. The space was disinfected by G1: chitosan 3 mg/mL; G2: 13-15 mg/mL TB and G3: Clp6. After photo-irridation the FPs were luted with conventional (n=11) and resin modified self-adhesive cement (n=11) for each group respectively. The specimens were incubated for 48 hrs prior to tensile BS testing. The type of bond fracture/failure was evaluated under 30x magnification. The tensile BS was statistically analyzed using ANOVA trailed by Tukey's test to respective groups.

RESULTS: FPs luted using self-adhesive resin modified cement with prior to disinfection by TBs displayed highest tensile BS 291.47 N ± 5.36. On the contrary, post-space disinfected by Clp6 displayed statistically significant ($p \leq 0.05$) lowest bond strength (276.62 N ± 4.31). Intra-group analysis displayed statistically significant difference in bond strength of the luting dental materials ($p \leq 0.05$).

CONCLUSIONS: The self-adhesive resin modified cement revealed utmost bond strength on pre-treatment of post-space with respective photo-activated photosensitizer (chitosan, TB & Clp6) in contrast to conventional GIC.

Key Words:
Fiber post, Chitosan, Toluidine blue, Chlorine p6.

Introduction

The sustained prevention in failure for endodontically restored tooth is reliant on the eminence of the restoration and adhesion capability of dental material and prosthesis[1,2]. A commendable endodontic therapy is reliant on strenuous eradication of necrotized dental pulp tissue and complex microbial biofilm *via* chemo-mechanical reduction[1]. In tur, it provides sustainable seal in the apical region and alleviation of the peri-apical/

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radicular tissues[2]. Decontamination of the canals is a subtle aim of endodontic therapy; however, multifarious endodontic anatomy, isthmus, and configuration of the dentinal tubules can act as a barrier for the diffusion of the endodontic irrigants and dismantling of microbial biofilm[1].

Micro-leakage has substantively inimical effect on prognosis of endodontic therapies. Endodontically treated teeth with extensive destruction of clinical crown require fiber posts (FP) for structural reconstruction, simulate as a natural tooth for restoring esthetic appearance and function[3]. Consequently, composite posts are reinforced with resin essential for solidification of root canal treated tooth as it enhances potential for bearing the vertical (occlusal) stress, esthetic appearance and modulus of elasticity of the tooth structure[4,5]. The attainment of FP is multi-factorial including the geographical configuration of dentinal tubules in radicular region, mechanical property of dentin and amount and type of collagen content. Additionally, adhesive property of dental cement, bond strength (BS) of luting agent, composition of luting dental cement, amount of luting cement to be used, preparation of radicular-post space and -dentin attribute for attainment of substantial bond between FP-dentin with enhanced coronal seal[6].

Prognosis and efficacy of bond between FP-dentin is linked to the reduction of microbial content, eradication of smear layer and disinfection of radicular post space with chemicals and photosensitizers, admittance of cement into the post radicular space for restoration with FPs[7-12]. In current era, photodynamic therapy is considered as an immaculate disinfecting technique to be used in endodontic therapies. It employs the principle of photo-activating the photosensitizer with appropriate intensity and wavelength which in-turn leads to excitation of molecular oxygen forming singlet oxygen. The photosensitizers in turn lead to the lysis of the bacterial cell wall disinfecting the root canals[2,13-17]. Chitosan, toluidine blue (TB) and Chlorine p6 (Clp6) have been considered as an effective photosensitizers for elimination of the microbial complex photo-oxidatively from the root canals during endodontic therapy which can escort to effective fixation of FPs in the post space[18]. The use of TB as an adjunct to conventional irrigant is considered to provide an effective sanitization against common endodontic microbial flora[19,20]. Intensification in time for light exposure of TB photosensitizer can enhance the anti-microbial potential. Chitosan nano-particles have been observed to have a strong potential in disinfection of endodontic microbial biofilm. Chitosan has impending effect of disrupting the bacterial biofilm and maintain anti-bacterial effect even after aging[21]. But till date the effect of TB, chitosan and Clp6on FPs on altering the tensile BS of luting cements is yet to be evaluated.

The dental cements used to lute FPs should encompass passable BS in order to evade dislocation of FPs under masticatory forces. Efficient luting cement should develop a uniform structo-mechanical complex of cementing material with FP and radicular dentin[22]. Till date no luting agent stood testified to attain ideal properties, thus detachment of FPs as a consequence of adhesion failure at the interface of dentin and cement luted FPs is considered as a communal factor[23]. GIC is widely used for luting prosthetic appliances[24].

Till date there is no data indicated in indexed databases evaluating BS of FP luted with dissimilar categories of dental cements with prior photo-irritation of radicular dentin by chitosan, TB and Clp6 photosensitizer. The present *in vitro* study intended to evaluate the tensile BS of self-cure resin modified *vs.* conventional GIC luted FPs to radicular dentin after disinfection with respective diode activated photosensitizers (chitosan, TB and Clp6). It can be hypothesized that FPs luting with self-cure resin modified cement could have comparable tensile BS to those coated with conventional glass ionomer cement on pre-treatment with photosensitizer chitosan, TB and Clp6.

Materials and Methods

The study was designed following the guidelines of Checklist Reporting *In Vitro* Study and conducted on approval from the Ethical Committee of Riyadh Elm University, Riyadh, Saudi Arabia (IRB Approval number: FRP/2021/427). Sixty-six orthodontically extracted mandibular pre-molars were selected based on a strict defined inclusion criterion. The inclusion criteria defined for selection of teeth enlisted non -traumatic, -carious, -fractured mandibular pre-molars with closed apices after validation on radiological analysis. Later, after through debridement of periodontal ligaments, plaque and calculus by the ultrasonic scaler the teeth were immersed in a disinfecting solution of Chloramine T trihydrate (Merck, Darmstadt, Germany) for 48 hr, refrigerated at a temperature of 4°C. As elaborated in a study by Qamar et al[2] each tooth was horizontally sectioned at the cement-enamel junction in order to preserve 14 mm

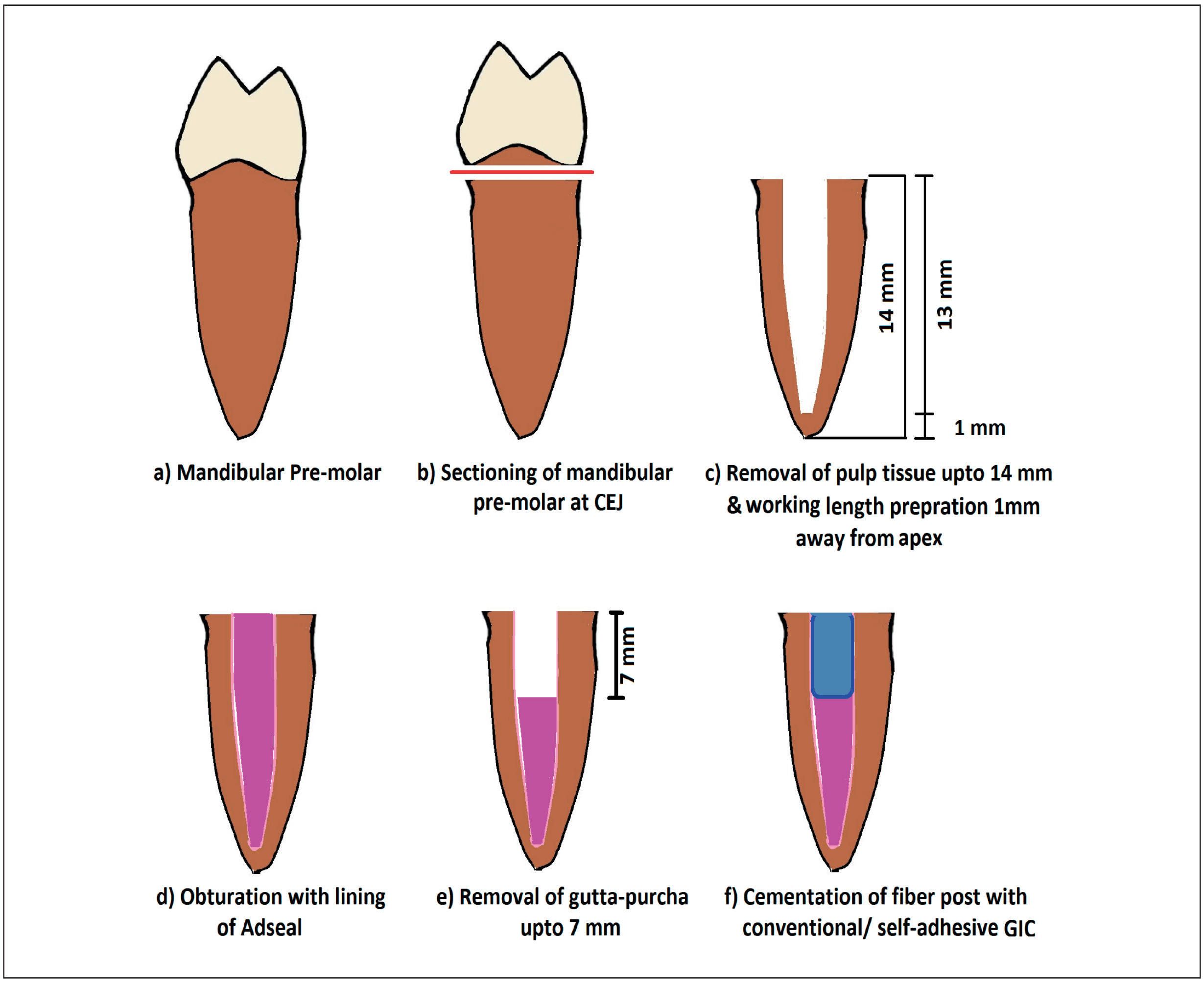

Figure 1. Stages for sample preparation; **a**) mandibular pre-molar; **b**) sectioning at cemento-enamel junction; **c**) removal of radicular pulp; **d**) obturation with gutta- purcha with lining of Adseal; **e**) post-space formation; **f**) cementation of fiber post after disinfection of post space with photosensitizer.

of radicular region using rotating diamond cutting saw machine Micracut 125 Low Speed Precision Cutter (Metkon, Bursa, Turkey). The sectioning of the tooth was conducted under thorough water cooling as shown in pictorial form in the Figure 1 (a, b). Subsequently, the sixty-six samples were embedded in an upright position in epoxy resin using Teflon molds. The cervical third and partially middle third of the radicular region were left exposed for the measurement of tensile strength.

Endodontic Therapy

The debridement of radicular tissues from the canals was commenced by shaping with #10 K-file up to 1 mm less than that of the working length (Figure 1 c). The canals were expanded till K-file #25. It was trailed by shaping of canals with X-smart held with ProTaper Next rotary NiTi files (Dentsply Sirona, Gloucestershire, UK) in conjunction with thorough 1% Sodium Hypochlorite (NaOCl) irrigation. The canals were later desiccated by ProTaper Universal paper points (Dentsply Sirona, Gloucestershire, UK). The desiccated canals were then impeded by Pro Taper Next Obturators gutta-percha points (Dentsply Sirona, Gloucestershire, UK) and ADSEAL (Meta Biomed, Chungcheongbuk-do, South Korea) (Figure 1 d). The specimens were stored for seven days at a calibrated temperature of 37°C under 100% humidity to mimic oral environment.

Peso-reamers (Mani, Utsunomiya, Japan) were used to develop a space of approximately 7 mm in gutta-percha for placement of the FP. The samples were randomly divided into three groups (n=22) for three photosensitizer, disinfecting the FP space in radicular canals trailed by treatment with two different luting regimes respectively (Figure 1 e).

Group A: Chitosan

In Group A's 22 specimens, the radicular canals were filled with 3 mg/mL Chitosan photosensitizer (Sigma Aldrich, Madrid, Spain). The chitosan was admixed with 1% acetic acid (Sigma Aldrich, Madrid, Spain) in order to prepare Chitosan at a concentration of 3 mg/mL. The Chitosan was injected in the post space at 100 µL with sterile micro-pipets. The topical Chitosan was photo-initiated by diode laser, at a wavelength of 660 nm for 60 seconds at energy of 100 mW. The Chitosan was mined back from the post space trailed by disinfection with normal saline and air dried.

Group B: 13-15 mg/mL Toluidine Blue (TB)

The TB (Sigma Aldrich, St. Louis, MO, USA) was prepared in deionized distilled water at a concentration of 13-15 mg/mL. The canals for the 22 specimens included in Group B were treated with TB solution. After injecting TB solution for 120 seconds in the canals, the photosensitizer was activated by a diode laser with a total dwell time of 120 seconds at energy of 120 mW. The diode laser of 635 nm wavelength was used for photo-irradiation by a special rod-shaped laser guide and was kept 1 mm short of the root apex. In order to ensure uniform photo-irradiation, the laser guide was moved in a spiral pattern apico-coronally. It was followed by disinfection of the radicular canals with normal saline and air dry.

Group C: Chlorine p6 (Clp6)

The Clp6 was prepared as per the guidelines reported by Datta et al[25]. The canals were filled with 100 µL Clp6 by the micro-pipettes. Later after air dry Clp6 was photo-activated by a flexible laser diode fiber tip of 600 µm for 120 seconds, at a wavelength of 670 nm (Denfotex Light System Ltd, Inverkeithing, UK). The total power delivered at the tip during the dwell time was 65 mW. It was straggled by normal saline irrigation and through drying.

Placement of Prefabricated Composite FP

The photo-irradiated specimens of all three groups were subjected to irrigation with 5 mL deionized distilled water trailed by drying with ProTaper Universal paper points (Dentsply Sirona, Gloucestershire, UK). The decontaminated RelyX™ Fiber Post 3D (3M Science Applied to Life, Minnesota, MN, USA) were placed in the radicular space after application of two different luting cements, eleven samples from each group respectively (Figure 1 f).

The post space luting in the eleven samples for the group 1, 2 and 3 respectively was done by self-adhesive resin modified cement (SARC) (3M™ RelyX™ Unicem 2 Automix Self-Adhesive Resin Cement, 3M Science Applied to Life, Minnesota, MN, USA). The cement was used with endo-tip for direct application in the post space reducing the chances of voids. The FP in the remaining eleven samples in each group was cemented by conventional Glass Ionomer Luting Cement (AquaCem, Dentsply Sirona, Konstanz, Germany). Subsequently, the specimens cemented with FP were incubated at 37°C for 48 hrs under a controlled environment of 100% humidity. Before undergoing tensile testing, the samples were subjected to thermo-cycling in an Automated Thermal Cycler (Applied Biosystems, Mississauga, Canada) at a temperature ranging from 5-60°C at 10K cycles for time duration of 30 seconds.

Exploration of the Tensile Bond Strength (BS) Testing

The resin enforced composite FP of the specimens was subjected to a cell capacity of 50 kilograms (Kg) HBM-S40 (Instron, High Wycombe, UK) tensile strength at a minimal speed of 0.5 mm/min. The sample was fixed with metal clamps at two ends i) coronal region of the FP; and ii) epoxy resin block. The samples were marked with specific codes based on photosensitizer and luting agents. The load required for the displacement of the FP from the canals was considered as point for measurement of tensile strength. The force applied to measure the BS was measured in International System of Units "Newton" (N).

Evaluating Adhesion Failure/Fracture of FP

The failure/fracture of FPs were analyzed under 50x magnification of a light microscope (Nikon, Japan). The failure/fracture of FPs were categorized in three different categories: i) category I: adhesion failure (AF) displaying failure at the interface of radicular dentin and luting dental cement; ii) category II: cohesion fracture/failure (CF) demonstrating failure between dental cement and FPs; and iii) category III: combination of AF and CF.

Statistical Analysis

The force of tensile BS for various groups was stati stically analyzed using SPSS software Version 18 (Chicago, IL, USA) with significance level set at $p \leq 0.05$. For inferential statistic Levene's

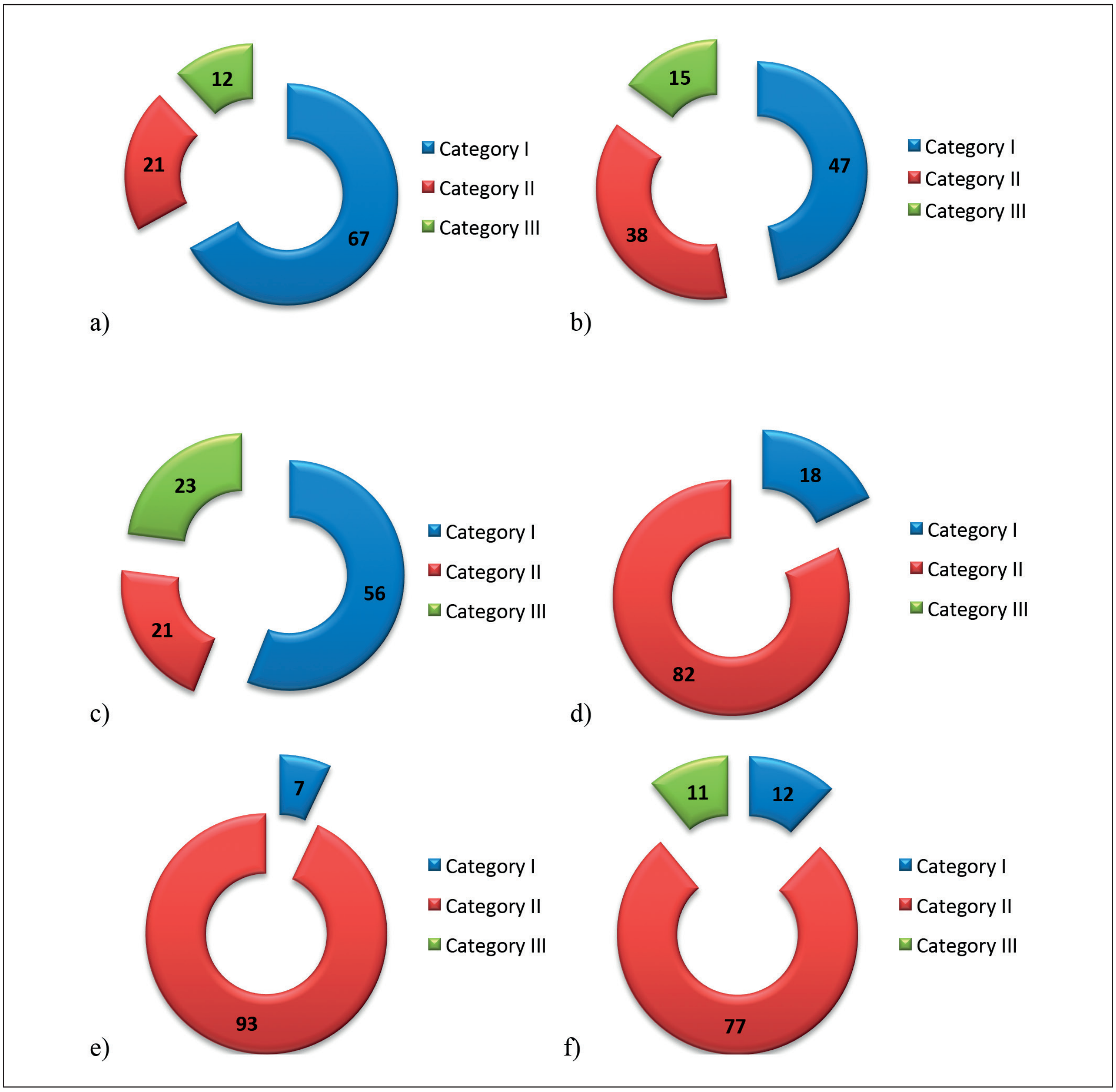

Figure 1. Stages for sample preparation; **a**) mandibular pre-molar; **b**) sectioning at cemento-enamel junction; **c**) removal of radicular pulp; **d**) obturation with gutta- purcha with lining of Adseal; **e**) post-space formation; **f**) cementation of fiber post after disinfection of post space with photosensitizer.

test was used to evaluate the variances for various groups of photosensitizers. Later, One-way ANOVA and Tukey's post-hoc test were used to statistically compare the tensile BS between the groups of specimens treated by various photosensitizers.

Results

The FPs space disinfected with TB photosensitizer and luted with SARC revealed highest tensile BS (291.47 N ± 5.36) as shown in Table I, Figure 2. On contrary, FPs cemented by SARC and radicular post space photo irritated by Clp6 showed lowest tensile BS of nearly 276.62 N ± 4.31. The canal space sanitized by TB and FBs cemented *via* conventional GIC luting cement demonstrated highest tensile BS of 238.81 N ± 5.73. Contradictorily post space disinfected by Clp6 displayed minimal BS on cementation of FPs with AquaCem (GIC luting cement) (233.53 N ± 3.82).

The intra-group comparison for the teeth disinfected by respective photosensitizers displayed statistically significant difference ($p \leq 0.05$) between

Table I. Materials used in study, their composition and comparison of means ± SD tensile BS values among study groups.

Type of Luting cement	Brand Name	Composition	Photosensitizers: 3 mg/mL Chitosan (Group 1)	Photosensitizers: 13-15 mg/mL Toluidine Blue (TB) (Group 2)	Photosensitizers: Chlorine p6 (Clp6) (Group 3)
Conventional Glass ionomer Cement	AquaCem (GIC luting cement) (AquaCem, Dentsply Sirona, Konstanz, Germany)	***Bottle 1 (powder)*** Sodium calcium-alumino-fluorosilicate, barium sulfate ***Dropper Bottle II*** Polyacrylic acid, pigments, tartaric acid, water	236.36 N ± 4.63^{Aa}	238.81 N ± 5.73^{Ab}	233.53 N ± 3.82^{Aa}
Self-adhesive resin cement	3M™ RelyX™ Unicem 2 Automix Self-Adhesive Resin Cement (3M Science Applied to Life, Minnesota, MN, USA)	***Base paste*** Methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives ***Catalyst paste*** Methacrylate monomers, alkaline (basic) fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	284.23 N ± 6.02^{Ab}	291.47 N ± 5.36^{Bb}	276.62 N ± 4.31^{Cb}

Statistical analysis using ANOVA and Tukey multiple comparisons test.
$^{A–C}$Different superscript capital letters display a statistically significant difference in tensile BS between the groups on FPs cementation with same cement ($p < 0.05$).
$^{a–b}$Different superscript lower case letter means statistically significant difference in tensile BS within the group on FPs cementation with different luting cement ($p < 0.05$).

the tensile BS sustained by the two luting cements as shown in Table I. There was no inter-group difference observed for chitosan, TB and Clp6 luted with conventional GIC ($p \geq 0.05$). But a significant difference between tensile BS was observed for teeth disinfected by three different photosensitizer chitosan, TB and Clp6 respectively bonded with self-adhesive luting cement ($p \leq 0.05$).

The failure mode analyzed for FPs between different experimental groups is displayed in Figure 2 on luting with conventional GIC (A) and Automix self-adhesive resin modified GIC (B). The type of fracture defined as category I (adhesive failure) was most prevalent trailed by category II (cohesive failure) for FPs luted with the conventionally used GIC. On contrary, the limited number of combinational (adhesive & cohesive failure) was observed.

Discussion

The contemporaneous study evaluated and compared the tensile BS of conventional and self-adhesive luting GIC on FP with prior disinfection of radicular post space by the diode laser activated novel photosensitizer chitosan, TB and Clp6. It was hypothesized that the BS for both conventional and resin modified self-adhesive GIC might display similar strength irrespective the type of photosensitizer used for disinfection. Whereas, the FPs luted with self-adhesive GIC demonstrated to have enhanced BS in comparison to posts placed after luting with conventional cement.

The improved tensile BS for retention of FPs is based on the chemo-mechanical properties of the luting cement[26]. This study stressed to estimate the tensile BS of luting cements used during conservancy of a tooth with lost coronal region, endodontically treated with a prefabricated resin filled composite FPs of a standard diameter and taper. In current study conventional and self-adhesive GIC were used for the cementation of the FPs. The FPs cemented by the self-adhesive luting GIC after photodynamic therapy displayed highest tensile BS. It could be attributed to the enhanced chemo-mechanical properties of cement as it has higher content of inorganic resin fillers, monomer and rheology modifiers that enhances the processing of filler particles. Additionally the presence of phosphoric acid methacrylate as an adhesion monomer optimizes the inter-locking of the material with radicular dentine and it has higher tolerance against the moisture. The 3M™ RelyX™ Unicem 2 Automix Self-Adhesive Resin Cement has consumer friendly dispensing. It has been used as a treatment of choice as it leads to no sensitivity. On contrary, the conventional GIC has lower chemo-mechanical retention (tensile BS) and more moisture sensitive (moisture sorption without varnish application)[27]. Our results are in agreement with the data reported by Qamar et al[2] and Le bell et al[28] displaying the tensile BS is greater for the resin reinforced dental cement in comparison to conventional cements.

In present study, the FPs bonded by resin modified self-cure luting cements after disinfection with photo-activated (diode laser) photosensitizers. The photosensitizer TBs and chitosan displayed higher tensile BS compared to conventional GIC luting material. The TB on activation with diode laser leads to the formation of radicals against oxygen dependent species causing degradation of target microbial species[29]. The factors in particular contributing to enhance BS of the FPs are linked to the morphological alterations in tubules of photosensitized radicular dentin, formation of retention tags and hyper-calcified peri-tubular dentinal matrix[13,30]. Foremost, during the setting process of resin modified cement, the single monomer molecules form a 3D-polymer network creating a strong bond between dental tissue and FPs. Concurrently acidic neutralization effect of the material enhances the long-term stability of resin modified cement.

The fracture modes analyzed were generally apparent for FPs cemented by conventional GIC luting material. The category I (adhesion failure) type of bond fracture was most evident, displaying adhesion failure between radicular dentin and cementing material. The adhesion failure is attributed to cements moisture sorption capability, easy solubility, absence of resin fillers and inappropriate mechanical retention potential[28,31]. On contrary, resin modified displayed common category II failures between the resin modified cement and FPs. The dynamics for cohesive failure are dependent on resistance in adherence of the material, photosensitizer, cement interaction, arrangement of radicular dentin collagen matrix, inadequate integration of the cement and parameters involved in photo-activation and effectiveness of dental cement[32-34].

There are various factors to be taken under consideration as limitation of the study as reduction in BS for the adhesion of FPs could include configuration of the dentinal tubules, dentinal fluid, apatite crystals precipitates, and presence of smear layer that could influence the results. Further analysis using atomic-force microscope and scanning electron microscope can be used to compare tensile BS of luting cements in relation to various photodynamic therapies used for disinfection of the radicular dentin. Further *in vitro* studies prior to clinical trials are recommended to evaluate the effect of photosensitizers on the radicular dentin which can modulate the BS of luting cements.

Conclusions

FPs luted with self-adhesive resin cement after radicular dentin disinfection with TB demonstrated highest tensile BS in comparison to Clp6 and Chitosan. In general FPs luted with self-adhesive resin modified cement after disinfection displayed improved tensile BS in comparison to FBs luted with conventional GIC cement.

Conflict of Interest

The authors declare that they have no conflict of interest.

Authors' Contribution

Conceptualization, Z.Q.; methodology, Z.Q. and T.Z.; software, Z.Q. and N.S.A.; validation, Z.Q., N.S.A. and A.K.; formal analysis, R.N.R.; investigation, R.N.R., M.S, A.H.R.; resources, M.S.; data curation, R.N.R.; writing—original draft preparation, Z.Q., and A.H-R.; writing—review and editing, T.Z.; visualization, Z.Q.; supervision, N.S.A., T.Z. and A.H.R.; project administration, Z.Q.; funding acquisition, Z.Q. All authors have read and agreed to the published version of the manuscript.

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