



## Influence of curcumin photosensitizer in photodynamic therapy on the mechanical properties and push-out bond strength of glass-fiber posts to intraradicular dentin

Henrico Badaoui Strazzi Sahyon<sup>a</sup>, Paloma Pereira da Silva<sup>b</sup>, Murilo Silva de Oliveira<sup>b</sup>, Luciano Tavares Angelo Cintra<sup>b</sup>, Eloi Dezan-Júnior<sup>b</sup>, João Eduardo Gomes-Filho<sup>b</sup>, Rogério de Castilho Jacinto<sup>b</sup>, Paulo Henrique dos Santos<sup>a</sup>, Gustavo Sivieri-Araujo<sup>b,\*</sup>

<sup>a</sup> Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University – UNESP, Araçatuba, SP, Brazil

<sup>b</sup> Department of Restorative Dentistry, Discipline of Endodontics, Araçatuba School of Dentistry, São Paulo State University – UNESP, Araçatuba, SP, Brazil

### ARTICLE INFO

#### Keywords:

Dentin  
Curcumin  
Elastic modulus  
Hardness  
Photochemotherapy  
Tensile strength

### ABSTRACT

**Background:** The effects of using curcumin as photosensitizer on the mechanical properties of intraradicular dentin and on the bond strength of glass-fiber posts are unknown. Thus, this in vitro study evaluated the influence of using curcumin as photosensitizer during photodynamic therapy on the Martens hardness, elastic modulus, and bond strength of glass-fiber posts luted to intraradicular dentin, in different tooth root thirds.

**Methods:** Eighty bovine teeth were divided into 5 groups according to the concentration of curcumin applied, with or without blue LED light activation: Control-deionized water; Curcumin 500 mg/L; Curcumin 500 mg/L + blue LED; Curcumin 1000 mg/L; and Curcumin 1000 mg/L + blue LED. Mechanical properties were measured in different thirds of the radicular dentin using an ultramicrohardness tester (n = 8). A universal testing machine was used to evaluate the push-out bond strength (n = 8). Mechanical properties were compared across groups with the Kruskal–Wallis test, and across regions with the Friedman test. Bond strength data were subjected to ANOVA, followed by Tukey's test ( $\alpha = 0.05$ ). Scanning electron microscopy was used to analyze the failure mode of the specimens.

**Results:** The use of curcumin photosensitizer, with or without blue LED light, improved mechanical properties compared to those of the control group ( $P < 0.05$ ), and promote no statistically significant difference in bond strength values ( $P > 0.05$ ). Overall, there was no difference among the intraradicular thirds for Martens hardness and push-out bond strength values ( $P > 0.05$ ).

**Conclusions:** Curcumin photosensitizer, with or without photodynamic therapy, changed the mechanical properties of intraradicular dentin; however, the Martens hardness and bond strength values did not differ with the depth of the dentin.

### 1. Introduction

Endodontic treatment requires the reduction or elimination of bacterial infection present in the root canal systems of targeted teeth [1]. Sodium hypochlorite [2], in various concentrations, and calcium hydroxide intracanal medication [3–5] have been widely used for this purpose. In vitro and in vivo culture-dependent studies have compared the antibacterial effects of these irrigating solutions [6,7] on the

endodontic microbiota and have reported conflicting results [8], and have shown negative effects on the bond strength of resin cement, used to adhere glass-fiber posts to root dentin [9]. Intraradicular glass-fiber posts are commonly used in endodontic treatment of teeth with little remaining coronal structure, in order to provide additional support for core material and future indirect restorations [10–12].

New therapeutic treatments have been introduced to potentiate the eradication of endodontic infections; these include photodynamic

\* Corresponding author at: Department of Restorative Dentistry, Discipline of Endodontics, São Paulo State University (UNESP) School of Dentistry, Rua José Bonifácio, 1193, 16015-050 Araçatuba, SP Brazil.

E-mail addresses: [ico\\_strazzi@hotmail.com](mailto:ico_strazzi@hotmail.com) (H.B. Strazzi Sahyon), [lomasilva@hotmail.com](mailto:lomasilva@hotmail.com) (P.P.d. Silva), [muoliveira92@gmail.com](mailto:muoliveira92@gmail.com) (M.S.d. Oliveira), [luciano.cintra@unesp.br](mailto:luciano.cintra@unesp.br) (L.T.A. Cintra), [eloi.dezan@unesp.br](mailto:eloi.dezan@unesp.br) (E. Dezan-Júnior), [joao.eduardo@unesp.br](mailto:joao.eduardo@unesp.br) (J.E. Gomes-Filho), [rogerio.castilho@unesp.com](mailto:rogerio.castilho@unesp.com) (R.d.C. Jacinto), [paulo.santos@unesp.br](mailto:paulo.santos@unesp.br) (P.H. dos Santos), [gustavo.sivieri@unesp.br](mailto:gustavo.sivieri@unesp.br) (G. Sivieri-Araujo).

<https://doi.org/10.1016/j.pdpdt.2019.01.025>

Received 3 October 2018; Received in revised form 18 December 2018; Accepted 18 January 2019

Available online 24 January 2019

1572-1000/ © 2019 Elsevier B.V. All rights reserved.

therapy (PDT) with specific photosensitizers (PSs), such as curcumin, which has shown satisfactory antimicrobial activity, in addition to having antioxidant, anti-tumor, and anti-inflammatory effects [13]. The mechanism of action of PDT involves the absorption of photons from the irradiation source by the PS, resulting in excitation of its electrons. This energy is then transferred to a specific substrate and, in the presence of oxygen, promotes formation of highly reactive and short-lived oxygen species (mainly singlet oxygen), which irreversibly oxidize the cellular components, causing death of microorganisms [9,14,15]. Curcumin PS interacts predominantly with cationic compounds, such as calcium present in the intraradicular dentin, due to the formation of superoxide anions and hydrogen peroxide [16]. However, it remains unknown whether this reactive oxygen may affect the mechanical properties of dentin, binding to molecules present in the intraradicular dentin substrate, which could modify the hydroxyapatite crystals and the adhesive interface between glass-fiber posts and dentin [17].

Hence, the aim of this *in vitro* study was to evaluate the mechanical properties (Martens hardness [MH] and elastic modulus [Eit]) of different thirds of root canal dentin, and the push-out bond strength of glass-fiber posts luted to intraradicular dentin that had been submitted to PDT with curcumin PS. The null hypotheses tested were that the interaction between curcumin PS and dentin substrate would not change the mechanical properties of dentin and the bond strength of glass-fiber posts to endodontically treated intraradicular dentin; and that there would be no difference in the mechanical properties of dentin and the bond strength of the adhesive interface between glass-fiber posts and intraradicular dentin across different thirds of teeth that had undergone PDT with curcumin PS.

## 2. Materials and method

### 2.1. Experimental design

Table 1 describes the materials used in this study. The study was approved by the institutional Ethics Committee (#15-00694). Bovine incisors that had been extracted from cattle approximately 3 years old (28–36 months) were used [18]. All teeth that presented fractures, cracks, and curved roots were excluded; in total, eighty bovine incisors were included in the study. The anatomic crowns of all teeth were removed at 1.0 mm above the cementum–enamel junction using a machine cutter (Isomet 5000; Buehler). To standardize teeth used in this study, only teeth with a mean root canal length of approximately 20 mm and mean root canal diameter of approximately 4 mm were included. The radicular canals were instrumented with K-files #80 (Dentsply Sirona) after the working length had been determined to be 1.0 mm less than this length; the canals were then irrigated with 10 mL of 1% sodium hypochlorite once per 15 s, and dried with sterile paper points and an air jet.

In order to prevent escape of the PS, the apical portion of all the roots were etched with 37% phosphoric acid (FGM) for 15 s, and dried

with sterile papers and an air jet. Dental adhesive (Adper Single Bond 2; 3M ESPE) was applied to the conditioned surface for 15 s and was then activated for 20 s using a light-polymerization unit (Ultraled; Dabi Atlante). The apical forams were sealed with a composite resin (Filtek Z250 XT; 3M ESPE) and the endodontically treated roots were randomly divided into 5 groups ( $n = 16$ ).

Deionized water was inserted into the root canal and no PS or PDT was administered in the control group. In the remaining 4 groups, the root canals were filled with curcumin (500 mg/L [ $C_{500}$ ] or 1000 mg/L [ $C_{1000}$ ]) for 5 min (period of pre-irradiation) and the PS was slowly agitated for 1 min using an ultrasonic Irrisonic E1 tip (Helse Dental Technology) coupled to an ultrasonic unit (Nac Plus, Adiel). Care was taken to avoid contact of the tip with the dentin substrate [19]. In the  $C_{500WL}$  (without LED light activation) and  $C_{1000WL}$  groups, the PS was not activated. In the  $C_{500L}$  (PS activated by LED light) and  $C_{1000L}$ , the PS was activated by 4 min of blue LED light ( $\lambda$  480 nm) irradiation, using a fiber optic of 300  $\mu$ m diameter, inserted into the canal to a level 2 mm apical to the teeth working length [19]. To ensure homogeneous diffusion of light throughout the canal, the flexible optical fiber was moved in the apico-cervical direction, using helicoidal movements [20,21], that were performed 10 times/min [22].

Curcumin PS exhibits absorption peaks at 450–495 nm, with 72 J/cm of final energy [17]. The wavelength of the light source used was thus determined by the absorption property of the PS, and the duration of light action was determined according to the satisfactory antimicrobial activity reported in previous studies [17,21]. Subsequently, 10 mL of deionized water was inserted into the intraradicular canals to remove the curcumin PS and the root canals were then dried with sterile paper points and an air jet. The roots were stored for 7 days at 100% humidity and 37 °C [17,23,24] prior to obturation and further tests.

### 2.2. Analysis of mechanical properties

Eight teeth per group were sectioned under water cooling (Isomet 5000; Buehler), obtaining slices approximately 1.3 mm in thickness. The different thirds of the root canals (cervical middle, and apical) were fixed in acrylic resin (Classico) and manually finished with #320, #600, #800, and #1200 grit silicon carbide papers (Exttec Corp). The specimens were polished with diamond pastes (#6, #3, #1, and #0.25  $\mu$ m; Buehler) for 4 min with each paste. Between each finishing and polishing process, the specimens were washed in an ultrasonic unit (model 2210; Branson Ultrasonic Corp) with deionized water for 2 min [17].

MH and Eit values were measured using an ultramicrohardness tester (DUH-211; Shimadzu). A Vickers diamond tip was used at a load of 3 mN, with a holding time of 5 s. Five indentations were performed in each region of the dentin substrate and the MH and Eit values were automatically measured by the software program installed in the tester [17,25,26].

**Table 1**

Materials, classification, composition, and batch numbers of materials.

Material	Classification	Composition	Batch
Filtek Z350XT (3M ESPE)	Resin Composite	Bis-EMA, Bis-GMA, TEGDMA, UDMA, silica and zirconia nanofillers, and agglomerated zirconia-silica nanoclusters	HB004209993
MTA Fillapex (Angelus)	Endodontic Cement	Salicylate resin, natural resin, diluting resin, bismuth oxide, nanoparticulated silica, MTA and pigments	36870
RelyX Ceramic Primer (3M ESPE)	Silane	3-MPS, ethyl alcohol, water	H0001504424
RelyX U200 (3M ESPE)	Resin Cement	Base: glass fiber, methacrylate phosphoric acid esters, triethylene glycol dimethacrylate, silane treated silica, sodium persulfate. Catalyst: glass fiber, substitute dimethacrylate, silane-treated silica, sodium p-toluenesulfonate, calcium	1518200189

Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; 3-MPS, 3-methacryloxypropyl-trimethoxy silane.

### 2.3. Bonding strength analysis

Forty roots specimens were analyzed in the push-out bond strength test ( $n = 8$ ). After treatment with curcumin PS, with or without PDT, the canals were obturated by the Tagger technique with #70 McSpadden instruments (Dentsply Sirona) and calcium hydroxide cement (MTA Fillapex; Angelus). Temporary cement (White Cimpat; Septodont) was used to seal the coronal access and the obturated root canals were stored at 37 °C and 100% humidity for 7 days.

The gutta percha cones were removed to  $\pm 9$  mm with reference to the working length of the teeth, using a #1 low-speed drill, followed by a #2 drill (Dentsply Sirona). Prior to the adhesive procedure, the glass-fiber post (White Post DCE; FGM) surface was treated by conditioning with 35% phosphoric acid (3M ESPE) for 60 s, followed by washing and drying with an air jet. The surfaces of the posts were silanized with a silane primer (RelyX Ceramic Primer; 3M ESPE) for 60 s, and an air jet was gently applied. The posts were not further manipulated, to prevent surface contamination [25].

The post space was irrigated with physiological saline (0.9%) and the canals were dried with sterile paper points and an air jet before the luting process. Self-adhesive resin cement (RelyX U200; 3M ESPE) was used for luting the posts; this was activated for 40 s, from the occlusal surface, with a polymerization light (Ultraled; Dabi Atlante) of 1125 mW/cm<sup>2</sup> intensity [27]. The specimens were stored for 7 days at 37 °C and 100% humidity.

The specimens were sectioned with a low-speed diamond saw (Isomet 5000; Buehler) under water cooling, to obtain slices of the cervical, middle, and apical thirds. Slice thickness, measured using a digital caliper (Mitutoyo), was approximately 1.3 mm [17,25,28]. A universal testing machine (DL3000, EMIC) was used for the push-out test. A compressive load was applied in a vertical direction with an active tip, with a cross-head speed of 0.5 mm/min, in the center of the glass-fiber post [17,24,29–32].

The failure mode of the root canals was analyzed using a stereomicroscope. Classification of the failure modes was: (1) mixed failure; (2) adhesive failure; (3) cohesive failure in dentin. Representative failure specimens were submitted to coating with gold (Baltec SCD 050; Balzers) and the fracture patterns were evaluated by scanning electron microscopy (SEM-JSM5600LV; JEOL) [17,33,34].

### 2.4. Statistical analysis

MH, Eit, and bond strength data were submitted to normality tests (Shapiro–Wilk). Data of mechanical properties were compared among groups using the nonparametric Kruskal–Wallis test, and among thirds using the Friedman test ( $\alpha = 0.05$ ). Bond strength data were submitted to 2-way ANOVA and the Tukey least significant difference test ( $\alpha = 0.05$ ).

## 3. Results

Higher values of MH and Eit were found for dentin in which the curcumin PS was applied at a higher concentration, without activation by blue LED light (C<sub>1000WL</sub>), than in the control group, in all thirds ( $P < 0.05$ ). There was no statistically significant difference in the MH values of dentin when either concentration of curcumin without blue LED light activation (C<sub>500WL</sub> and C<sub>1000WL</sub>) or a lower concentration of curcumin with blue LED light activation (C<sub>500L</sub>) were applied, in all thirds ( $P > 0.05$ ) (Table 2).

The effect of the application of curcumin PS and PDT was also supported by the Eit values, in which the dentin of the control and C<sub>1000L</sub> groups showed lower Eit values in the cervical, middle, and apical thirds ( $P < 0.05$ ) (Table 2). When comparing the regions, no statistically significant difference in MH values was found in any of the groups analyzed ( $P > 0.05$ ); however, the dentin of the cervical third of the C<sub>500WL</sub> group showed higher Eit values than that of the apical

third ( $P = 0.008$ ).

Regarding the push-out bond strength values (Table 3), the dentin of the C<sub>500WL</sub> group showed higher bond strength values than that of the C<sub>1000WL</sub> specimens in the cervical third ( $P = 0.016$ ). There was no significant difference among the groups in the middle and apical regions ( $P > 0.05$ ). When comparing the intraradicular thirds, there was a significant difference only in the C<sub>500WL</sub> group, where the cervical third showed higher push-out bond strength values than the apical third ( $P = 0.008$ , Table 3). When analyzing the thirds for each group, there was a predominance of mixed-type failure in the C<sub>1000WL</sub>, C<sub>1000L</sub>, and control groups, while for the C<sub>500WL</sub> and C<sub>500L</sub> groups, cohesive type failure was predominant (Fig. 1).

## 4. Discussion

The use of curcumin PS and PDT influenced the MH and Eit mechanical properties and the bond strength of glass-fiber posts to intraradicular dentin (Tables 2 and 3), rejecting the first null hypothesis of the study. Assessment of the root thirds showed differences in the Eit of different regions of intraradicular dentin (Table 2), thus rejecting the second null hypothesis.

The use of curcumin PS at a higher concentration, along with blue LED light activation, promoted lower values of MH and Eit in dentin than in the other curcumin PS groups (Table 2). We speculated that the irradiation promoted significant changes in the dentin substrate, due to blue LED light photoactivation of the curcumin, which promotes formation of hydrogen peroxide [16,35] that can bind to components in dentin, such as the calcium present in the hydroxyapatite crystals [8,17]. The concentration of the compound and the presence of the irradiation source were important factors that influenced the alterations in the dentin, explaining the lower mechanical properties.

Curcumin is an anionic substance that has the ability to bind to cationic molecules, such as the calcium present in hydroxyapatite [17]. The reaction between this PS and calcium, and particularly at a higher concentration of the PS, would result in precipitates that can act as a physical barrier, and reduce the interaction between the resin cement and the surface of the dentin [34]. This calcium-PS relationship could explain the lower bond strength values in the C<sub>1000WL</sub> group than in the C<sub>500WL</sub> group (Table 3).

Furthermore, curcumin is a hydrophobic polyphenol compound [17,36], allowing less water sorption that could decrease the bond strength of the resin interface [17,27]. This could explain, in general, the non-statistically significant difference between the groups that used curcumin, at both concentrations, and the control group (Table 3). In addition, the integrity of the adhesive interface could be enhanced due to the hydrophobic character of the resin cement used for luting the glass-fiber posts in the prosthetic space of root dentin [27]. This adhesive interface integrity can be seen in the images of the C<sub>1000WL</sub> and C<sub>1000L</sub> groups (Fig. 2G–J); these groups showed a higher incidence of mixed failure type (Fig. 1). Similar adhesive interface integrity was also found in a previous study, in which the previous application of curcumin increased the values of the bond strength of glass-fiber posts luted to dentin [17].

Comparing the different thirds of intraradicular dentin, there was a tendency for the dentin of the apical third to exhibit the highest Eit values; the exception was the dentin in the C<sub>500WL</sub> group (Table 2). The higher values for the apical third could be attributed to several factors [25], including cavity configuration [37], presence of apical sclerosis [38], level of access to the apical portion, or restricted cement penetration into the deeper portions of the prosthetic-post space. For the push-out bond strength analysis, there was no statically significant difference between the thirds in any of the evaluated groups, except for the C<sub>500WL</sub> group, which showed lower bond strength values for the apical than the cervical third (Table 3). It could be speculated that the cervical and middle thirds would be more susceptible to PS action, and due to the anionicity of the substance, calcium precipitates were

**Table 2**

Mean ± standard deviation (GPa) values of Martens hardness (MH) and elastic modulus (Eit) of intraradicular dentin as function of photosensitizer agent and photodynamic therapy used, in different root thirds.

Mechanical Property and Region	Control Group	C <sub>500WL</sub> Group	C <sub>500L</sub> Group	C <sub>1000WL</sub> Group	C <sub>1000L</sub> Group
<b>MH</b>					
Cervical	0.44 ± 0.12 Ab	0.93 ± 0.18 Aa	0.85 ± 0.24 Aa	0.86 ± 0.12 Aa	0.51 ± 0.21 Ab
Middle	0.54 ± 0.06 Ac	0.90 ± 0.20 Aa	0.71 ± 0.23 Aabc	0.81 ± 0.09 Aab	0.63 ± 0.29 Abc
Apical	0.55 ± 0.15 Ab	0.71 ± 0.15 Aab	0.94 ± 0.25 Aa	0.94 ± 0.23 Aa	0.63 ± 0.24 Ab
<b>Eit</b>					
Cervical	0.94 ± 0.30 Bc	2.41 ± 0.56 Aa	1.64 ± 0.46 ABab	2.20 ± 0.49 Aa	1.09 ± 0.48 Abc
Middle	1.21 ± 0.24 Ac	1.90 ± 0.67 ABab	1.40 ± 0.35 Bbc	2.04 ± 0.53 Aa	1.32 ± 0.75 Abc
Apical	1.13 ± 0.37 ABc	1.55 ± 0.36 Bbc	1.98 ± 0.51 Aab	2.19 ± 0.57 Aa	1.39 ± 0.62 Abc

C<sub>500WL</sub>, curcumin 500 mg/L without blue LED; C<sub>500L</sub>, curcumin 500 mg/L activated with blue LED; C<sub>1000WL</sub>, curcumin 1000 mg/L without blue LED; C<sub>1000L</sub>, curcumin 1000 mg/L activated with blue LED.

Different superscript uppercase letters in columns and lowercase letters in rows indicate statistically significant differences for each mechanical property analyzed (P < .05).

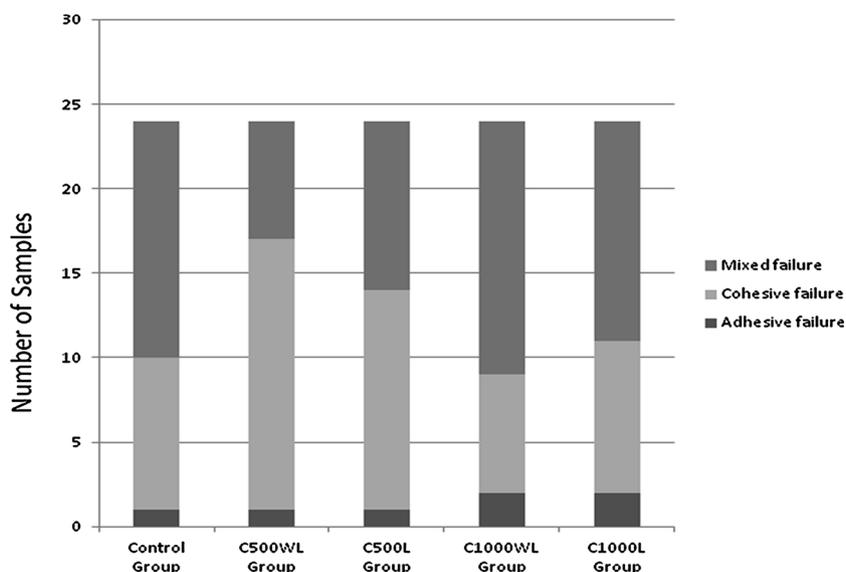
**Table 3**

Mean ± standard deviation (MPa) values of extrusion bond strength (push-out strength test) of intraradicular dentin as function of photosensitizer agent and photodynamic therapy used, in different root thirds.

Bond Strength and Region	Control Group	C <sub>500WL</sub> Group	C <sub>500L</sub> Group	C <sub>1000WL</sub> Group	C <sub>1000L</sub> Group
Cervical	4.51 ± 1.27 Aab	4.81 ± 1.66 Aa	3.44 ± 1.68 Aab	2.51 ± 1.90 Ab	2.77 ± 1.07 Aab
Middle	3.29 ± 1.82 Aa	4.08 ± 1.27 ABa	3.34 ± 1.55 Aa	2.99 ± 1.50 Aa	2.99 ± 1.51 Aa
Apical	3.93 ± 1.98 Aa	3.09 ± 1.71 Ba	3.06 ± 1.51 Aa	2.04 ± 1.35 Aa	3.83 ± 1.51 Aa

C<sub>500WL</sub>, curcumin 500 mg/L without blue LED; C<sub>500L</sub>, curcumin 500 mg/L activated with blue LED; C<sub>1000WL</sub>, curcumin 1000 mg/L without blue LED; C<sub>1000L</sub>, curcumin 1000 mg/L activated with blue LED.

Different superscript uppercase letters in columns and lowercase letters in rows indicate statistically significant differences (P < .05).



**Fig. 1.** Incidence of fracture patterns (thirds specimen numbers) according to failure type. C<sub>500WL</sub>, curcumin 500 mg/L without blue LED; C<sub>500L</sub>, curcumin 500 mg/L activated with blue LED light; C<sub>1000WL</sub>, curcumin 1000 mg/L without blue LED; C<sub>1000L</sub>, curcumin 1000 mg/L activated with blue LED light.

accumulated in the apical third, reducing the interaction between the luting agent and the surface of the root dentin [34].

However, different concentrations of the curcumin PS, with or without PDT, will not always obtain satisfactory results in all analyses. Although no improvement in the bond strength values could be evidenced in this study, the use of curcumin at 500 mg/L, with or without PDT, could be an appropriate alternative to promote prosthetic space antiseptics, since it has satisfactory antimicrobial activity, antioxidant, anti-tumor, and anti-inflammatory effects, as proven in previous studies [39,40].

The difficulty of standardizing the preparation in areas with difficult access and the non-homogeneity of the substrate, due to the peculiarities of each region, can be considered limiting factors of this study.

Further studies should be performed using other photosensitizing substances at different concentrations, as well as over different periods of time and the technique used for the application of the PSs.

In conclusion, the use of 500 mg/L curcumin as a PS, with or without PDT, is a suitable alternative protocol for antiseptics of intraradicular dentin, as it generally led to improved mechanical properties and did not alter the bond strength of the glass-fiber posts. Furthermore, the MH and bond strength of glass-fiber posts were not influenced by the intraradicular depth.

**Conflict of interest**

The authors declare that they have no conflict of interest.

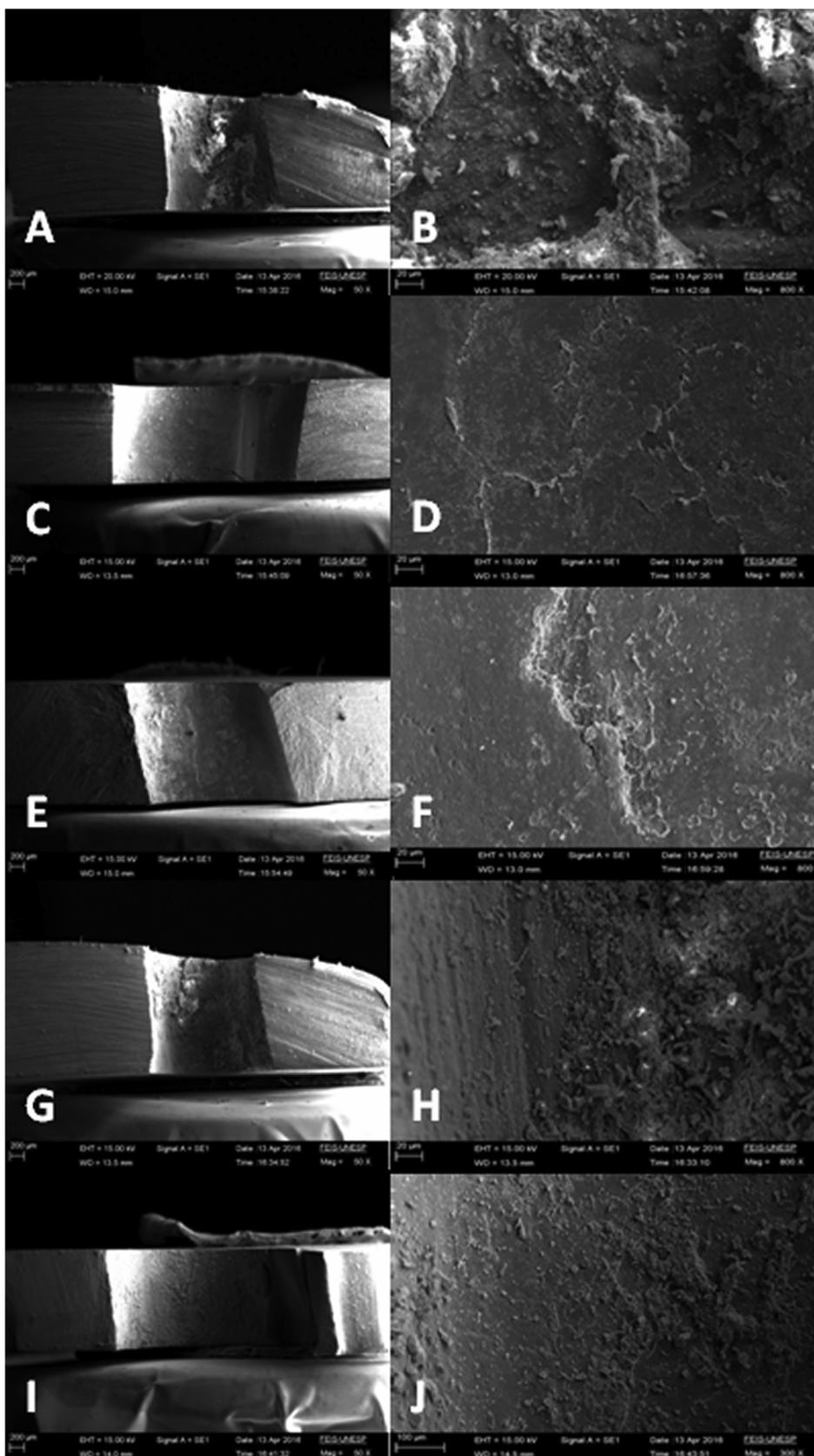


Fig. 2. A,B—Mixed failure of intraradicular dentin irrigated with deionized water solution (control group) (original magnification  $\times 50$  and  $\times 800$ ). C,D—Cohesive failure of intraradicular dentin after treatment with curcumin 500 mg/L ( $C_{500WL}$  group) (original magnification  $\times 50$  and  $\times 800$ ). E,F—Cohesive failure of intraradicular dentin after treatment with curcumin 500 mg/L and blue LED light ( $C_{500L}$  group) (original magnification  $\times 50$  and  $\times 800$ ). G,H—Mixed failure of intraradicular dentin after treatment with curcumin 1000 mg/L ( $C_{1000WL}$  group) (original magnification  $\times 50$  and  $\times 800$ ). I,J—Mixed failure of intraradicular dentin after treatment with curcumin 1000 mg/L and blue LED light ( $C_{1000L}$  group) (original magnification  $\times 50$  and  $\times 800$ ).

**Acknowledgments**

This study was supported by São Paulo Research Foundation (FAPESP) (Grant #2015/06056-3), PROPe—UNESP (Grant #12/2015)

and National Council for Scientific and Technological Development (CNPq) (Grant #447594/2014-1), Brazil. The authors thank Prof. Dr. Juno Gallego of the Department of Mechanical Engineering, São Paulo State University (UNESP), School of Natural Sciences and Engineering,

Ilha Solteira, for contributing to the analyses of mechanical properties.

## References

- [1] H. Zandi, A.K. Kristoffersen, D. Ørstavik, I.N. Rôças, F.J.R. Siqueira, M. Enersen, Microbial analysis of endodontic infections in root-filled teeth with apical periodontitis before and after irrigation using pyrosequencing, *J. Endod.* 44 (2018) 372–378.
- [2] H. Zandi, R.C. Rodrigues, A.K. Kristoffersen, M. Enersen, I. Mdala, D. Ørstavik, et al., Antibacterial effectiveness of 2 root canal irrigants in root-filled teeth with infection: a randomized clinical trial, *J. Endod.* 42 (2016) 1307–1313.
- [3] L. Silva, P. Nelson-Filho, M.R. Leonardo, M.A. Rossi, C.A. Pansani, Effect of calcium hydroxide on bacterial endotoxin in vivo, *J. Endod.* 28 (2002) 94–98.
- [4] J. Vera, J.F. Siqueira Jr., D. Ricucci, S. Loghin, N. Fernández, B. Flores, et al., One-versus two-visit endodontic treatment of teeth with apical periodontitis: a histo-bacteriologic study, *J. Endod.* 38 (2012) 1040–1052.
- [5] F.L. Berbert, G. Sivieri-Araujo, L.T. Ramalho, S.A. Pereira, D.B. Rodrigues, M.S. de Araújo, Quantification of fibrosis and mast cells in the tissue response of endodontic sealer irradiated by low-level laser therapy, *Lasers Med. Sci.* 26 (2011) 741–747.
- [6] M.E. Vianna, B.P. Gomes, V.B. Berber, A.A. Zaia, C.C. Ferraz, F.J. de Souza-Filho, In vitro evaluation of the antimicrobial activity of chlorhexidine and sodium hypochlorite, *Oral Surg. Oral Med. Oral Pathol. Oral Radiol. Endod.* 97 (2004) 79–84.
- [7] E. Ercan, T. Ozekinci, F. Atakul, K. Gül, Antibacterial activity of 2% chlorhexidine glu- € conate and 5.25% sodium hypochlorite in infected root canal: in vivo study, *J. Endod.* 30 (2004) 84–87.
- [8] F.M. Pascon, K.R. Kantovitz, L.E. Soares, A.M. Santo, A.A. Martin, R.M. Puppington-Rontani, Morphological and chemical changes in dentin after using endodontic agents: Fourier transform Raman spectroscopy, energy-dispersive x-ray fluorescence spectrometry, and scanning electron microscopy study, *J. Biomed. Opt.* 17 (2012) 0750081-6.
- [9] A.T.P.R. Ramos, L. Garcia Belizário, A.C. Venção, K.C. Fagundes Jordão-Basso, N.A. de Souza Rastelli, M.F. de Andrade, et al., Effects of photodynamic therapy on the adhesive interface of fiber posts cementation protocols, *J. Endod.* 44 (2018) 173–178.
- [10] A.L. Faria-e-Silva, S. Menezes Mde, F.P. Silva, G.R. Reis, R.P. Moraes, Intra-radicular dentin treatments and retention of fiber posts with self-adhesive resin cements, *Braz. Oral Res.* 27 (2013) 14–19.
- [11] C. Estrela, R. Holland, C.R. Estrela, A.H. Alencar, M.D. Sousa-Neto, J.D. Pécora, Characterization of successful root canal treatment, *Braz. Dent. J.* 25 (2014) 3–11.
- [12] M.T. Durski, M.J. Metz, J.Y. Thompson, A.K. Mascarenhas, G.A. Crim, S. Vieira, et al., Push-out bond strength evaluation of glass fiber posts with different resin cements and application techniques, *Oper. Dent.* 41 (2016) 103–110.
- [13] A. Hosseini, H. Hosseinzadeh, Antidotal or protective effects of curcuma longa (turmeric) and its active ingredient, curcumin, against natural and chemical toxicities: a review, *Biomed. Pharmacother.* 99 (2018) 411–421.
- [14] F.G. Rego-Filho, M.T. de Araujo, K.T. de Oliveira, V.S. Bagnato, Validation of photodynamic action via photobleaching of a new curcumin-based composite with enhanced water solubility, *J. Fluoresc.* 24 (2014) 1407–1413.
- [15] V. Chrepa, G.A. Kotsakis, T.C. Pagonis, K.M. Hargreaves, The effect of photodynamic therapy in root canal disinfection: a systematic review, *J. Endod.* 40 (2014) 891–898.
- [16] T.A. Dahl, W.M. McGowan, M.A. Shand, V.S. Srinivasan, Photokilling of bacteria by the natural dye curcumin, *Arch. Microbiol.* 151 (1989) 183–185.
- [17] H.B. Strazzi Sahyon, P. Pereira da Silva, M. Silva de Oliveira, L.T. Angelo Cintra, J.E. Gomes-Filho, P. Henrique dos Santos, et al., Effect of photodynamic therapy on the mechanical properties and bond strength of glass-fiber posts to endodontically treated intraradicular dentin, *J. Prosthet. Dent.* 120 (2018) e1-317.
- [18] J.G. Neves, M. Danelon, J.P. Pessan, L.R. Figueiredo, E.R. Camargo, A.C.B. Delbem, Surface free energy of enamel treated with sodium hemametaphosphate, calcium and phosphate, *Arch. Oral Biol.* 90 (2018) 108–112.
- [19] I.B. Juric, V. Plecko, D.G. Panduric, I. Anic, The antimicrobial effectiveness of photodynamic therapy used as an addition to the conventional endodontic re-treatment: a clinical study, *Photodiagn. Photodyn. Ther.* 11 (2014) 549–555.
- [20] G. Sivieri-Araujo, Í.O.A. Queiroz, R.D. Fabbro, F. Esteves, L.T.A. Cintra, P.C.T. Duarte, et al., Rat tissue reaction and cytokine production induced by antimicrobial photodynamic therapy, *Photodiagn. Photodyn. Ther.* 18 (2017) 315–318.
- [21] J.E. Gomes-Filho, G. Sivieri-Araujo, C.R. Sipert, L.M. da Silva Santos, Í.O. de Azevedo Queiroz, C. Men Martins, et al., Evaluation of photodynamic therapy on fibroblast viability and cytokine production, *Photodiagn. Photodyn. Ther.* 13 (2016) 97–100.
- [22] G. Sivieri-Araujo, L.M.S. Santos, I.O.A. Queiroz, M.T. Wayama, G.H. Yamanari, C.M. Martins, et al., Photodynamic therapy in Endodontics: use of a supporting strategy to deal with endodontic infection, *Dent. Press Endod.* 3 (2013) 52–58.
- [23] S.A. Shahdad, J.F. McCabe, S. Bull, S. Rusby, R.W. Wassell, Hardness measured with traditional Vickers and Martens hardness method, *Dent. Mater.* 23 (2007) 1079–1085.
- [24] E. Mumcu, U. Erdemir, F.T. Topcu, Comparison of micro push-out bond strengths of two fiber posts luted using simplified adhesive approaches, *Dent. Mater.* 29 (2010) 286–296.
- [25] T.Y. Suzuki, J.E. Gomes-Filho, J. Gallego, S. Pavan, P.H. Dos Santos, A.L. Fraga Briso, Mechanical properties of components of the bonding interface in different regions of radicular dentin surface, *J. Prosthet. Dent.* 113 (2015) 54–61.
- [26] A.P. Guedes, M.D. Moda, T.Y. Suzuki, A.G. Godas, R.H. Sundfeld, A.L. Briso, et al., Effect of fluoride-releasing adhesive systems on the mechanical properties of eroded dentin, *Braz. Dent. J.* 27 (2016) 153–159.
- [27] Y.K. Kim, J.S. Son, K.H. Kim, T.Y. Kwon, Influence of surface energy parameters of dental self-adhesive resin cements on bond strength to dentin, *J. Adhes. Sci. Technol.* 27 (2013) 1778–1789.
- [28] S.B. Berger, S. Pavan, M. Vidal Cde, P.H. Santos, M. Giannini, A.K. Bedran-Russo, Changes in the stiffness of demineralized dentin following application of tooth whitening agents, *Acta Odontol. Scand.* 70 (2012) 56–60.
- [29] J. Brichko, M.F. Burrow, P. Parashos, Design variability of the push-out bond test in endodontic research: a systematic review, *J. Endod.* 44 (2018) 1237–1245.
- [30] E.O. Onay, Y. Korkmaz, A. Kiremitci, Effect of adhesives system type and root region on the push-out strength of glass-fibre posts to radicular dentin, *Int. Endod. J.* 43 (2010) 259–268.
- [31] W.C. Wu, D.M. Wang, Y.C. Lin, C.A. Dai, K.C. Cheng, M.S. Hu, et al., Hydrogen bonds of a novel resin cement contribute to high adhesion strength to human dentin, *Dent. Mater.* 32 (2016) 114–124.
- [32] K. Bitter, L. Polster, H. Askar, M. von Stein-Launsitz, G. Sterzenbach, Effect of final irrigation protocol and etching mode on bond strength of a multimode adhesive in the root canal, *J. Adhes. Dent.* 8 (2017) 245–252.
- [33] T.Y.U. Suzuki, A.G.L. Godas, A.P.A. Guedes, A. Catelan, S. Pavan, A.L. Briso, et al., Microtensile bond strength of resin cements to caries-affected dentin, *J. Prosthet. Dent.* 110 (2013) 47–55.
- [34] V. Di Hipólito, F.P. Rodrigues, F.B. Piveta, C. Azevedo Lda, R.C. Brushi Alonso, N. Silikas, et al., Effectiveness of self-adhesive luting cements in bonding to chlorhexidine-treated dentin, *Dent. Mater.* 28 (2012) 495–501.
- [35] C.F. Chignell, P. Bilski, K.J. Reszka, A.G. Motten, R.H. Sik, T.A. Dahl, Spectral and photochemical properties of curcumin, *Photochem. Photobiol.* 59 (1994) 295–302.
- [36] S. Prasad, S. Gupta, A.K. Tyagi, B.B. Aggarwal, Curcumin, a component of golden spice: from bedside to bench and back, *Biotechnol. Adv.* 32 (2014) 1053–1064.
- [37] F.R. Tay, R.J. Loushine, P. Lambrechts, R.N. Weller, D.H. Pashley, Geometric factors affecting dentin bonding in root canals: a theoretical modeling approach, *J. Endod.* 31 (2005) 584–589.
- [38] F. Paqué, H.U. Luder, B. Sener, M. Zehnder, Tubular sclerosis rather than the smear layer impedes dye penetration into the dentine of endodontically instrumented root canal, *Int. Endod. J.* 39 (2006) 18–25.
- [39] D.A. Cusicanqui Méndez, E. Gutierrez, E. José Dionisio, M. Afonso Rabelo Buzalaf, R. Cardoso Oliveira, M.A. Andrade Moreira Machado, et al., Curcumin-mediated antimicrobial photodynamic therapy reduces the viability and vitality of infected dentin caries microcosms, *Photodiagn. Photodyn. Ther.* 24 (2018) 102–108.
- [40] G.C. Chaves, D.A. Cusicanqui Méndez, E. Gutierrez, E.J. Dionisio, M.A.A. Moreira Machado, T.M. Oliveira, et al., Could being chlorhexidine an adequate positive control to antimicrobial photodynamic therapy in in vitro studies? *Photodiagn. Photodyn. Ther.* (November (3)) (2018), <https://doi.org/10.1016/j.pdpdt.2018.11.004> pii: S1572-1000(18)30270-9.