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Bond strength of AH Plus and Epiphany sealers on root dentine irradiated with 980 nm diode laser

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Abstract

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Aim To evaluate the bond strength of AH Plus and Epiphany sealers to human root canal dentine irradiated with a 980 nm diode laser at different power and frequency parameters, using the push-out test.

Methodology Sixty canine roots were sectioned below the cementoenamel junction to provide 4-mm-thick dentine discs that had their root canals prepared with a tapered bur and irrigated with sodium hypochlorite, ethylenediaminetetraacetic acid and distilled water. The specimens were assigned to five groups ($n = 12$): one control (no laser) and four experimental groups that were submitted to 980 nm diode laser irradiation at different power (1.5 and 3.0 W) and frequency (continuous wave and 100 Hz) parameters. Half of specimens in each group had their canals filled with AH Plus sealer and half with Epiphany. The push-out test was performed and data (MPa) were analysed statistically by ANOVA and Tukey's test ($P < 0.05$). The specimens were split longitudinally and examined under SEM to assess the failure modes after sealer displacement.

Results The specimens irradiated with the diode laser and filled with AH Plus had significantly higher bond strength values (8.69 ± 2.44) than those irradiated and filled with Epiphany (3.28 ± 1.58) and the non-irradiated controls (3.86 ± 0.60). The specimens filled with Epiphany did not differ significantly to each other or to the control (1.75 ± 0.69). There was a predominance of adhesive failures at Epiphany–dentine interface (77%) and mixed failures at AH Plus–dentine interface (67%).

Conclusions The 980 nm diode laser irradiation of root canal dentine increased the bond strength of AH Plus sealer, but did not affect the adhesion of Epiphany sealer.

Keywords: 980 nm diode laser, bond strength, push-out test, root canal sealers.

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Introduction

Adhesion of an endodontic sealer is defined as its capacity to adhere to the root canal walls and promote the union of the Gutta-percha cones to each other and to the dentine (Sousa-Neto *et al.* 2005). This property may be influenced by the treatment performed on the root canal walls as well as by the type of sealer used.

An important factor to consider in root canal treatment is the presence of smear layer on dentine surfaces. According to White *et al.* (1984) and Kennedy *et al.* (1986), smear layer is a negative factor in root canal sealing because it forms an interface between the filling material and the root canal walls, hence reducing the adhesion of sealers.

Several chemical substances used as irrigants during biomechanical preparation of the root canal system

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might affect the characteristics of dentine substrate. Amongst the most commonly used irrigants, sodium hypochlorite (NaOCl) has the capacity to dissolve organic tissues, saponificate fats and neutralize toxic products as well as having an antimicrobial and deodorizing action (Spanó *et al.* 2002, Yamashita *et al.* 2003), whilst ethylenediaminetetraacetic acid disodium salt (EDTA) has a calcium ion chelating capacity and promotes dentine demineralization and smear layer removal (Hülsmann *et al.* 2003).

In addition to the routinely used chemical substances, other technologies have been investigated for the treatment of root canal dentine, such as laser irradiation. New lasers with a wide range of characteristics (Nd:YAG, 810 nm diode, Er:YAG and CO_2) are available and their potential applications in the field of Endodontics have been investigated. It has been demonstrated they are effective in smear layer removal, antibacterial activity and sealer adhesion (Takeda *et al.* 1999, Berkiten *et al.* 2000, Gutknecht *et al.* 2004, Stabholz *et al.* 2004, Schoop *et al.* 2006).

A new 980-nm wavelength diode laser has been launched recently to the market (Romanos *et al.* 2004, Wang *et al.* 2005, Schoop *et al.* 2006). This laser transmits energy through thin flexible fibres compatible with the dimensions and curved shapes of root canals. Diode laser units have a low purchasing and maintenance cost as well as greater versatility because of their compact size (Stabholz *et al.* 2004, Nikfarjam *et al.* 2005). Diode laser wavelengths have good penetration potential, with high absorption peaks in melanin and haemoglobin and low interaction with water and hydroxyapatite (Wang *et al.* 2005, Schoop *et al.* 2006). Gutknecht *et al.* (2004) and Lee *et al.* (2006) reported a reduction of the microbial content in hard-to-reach areas, such as dentinal tubules, after diode laser irradiation. Bornstein (2004) have reported that diode lasers (810–980 nm wavelength range) have a depth of penetration *per* pulse 10 000 times greater than that of Er:YAG laser, and may act more deeply within the dentinal tubules. Nevertheless, the findings of these studies are not consistent enough to elucidate their mechanism of action on root canal structures and the possible alterations on endodontic sealer adhesion.

Advances in adhesive technology have reinforced the search for means to minimize apical and coronal marginal leakage by improving sealer adhesion to root canal walls (Tay *et al.* 2005) and thus reducing the susceptibility of root-filled teeth to fracture (Teixeira *et al.* 2004). A new cement supplied with a self-etching primer has been developed recently to fulfil this purpose. Epiphany (Pentron Clinical Technologies, Wallingford, CT, USA) is a dual-cure methacrylate resin-based sealer used with a solid material named Resilon (Resilon Research LLC, Madison, CT, USA), which contains a blend of synthetic thermoplastic polyester polymers. Canal filling using the Epiphany/Resilon system is claimed to create a seal with the dentinal tubules within the root canal system (Shipper *et al.* 2004, Ezzie *et al.* 2006, Versiani *et al.* 2006). In essence, it has the potential to produce a 'monoblock' effect, where the core material (Resilon), sealer (Epiphany) and dentinal tubules become a single solid structure. Shipper *et al.* (2004) have suggested that this monoblock could provide a thorough seal of the root canal system as it would be able to minimize cervical marginal infiltration in case of loss or fracture of the temporary coronal restoration. Laboratory (Shipper *et al.* 2004) and *in vivo* (Shipper *et al.* 2005) studies have demonstrated the resistance of the Epiphany/ Resilon system monoblock to bacterial leakage.

Epoxy resin-based cements perform well as root canal sealers (Schafer & Zandbiglari 2003, Eldeniz *et al.* 2005). For example, AH Plus has been shown to have satisfactory physicochemical properties, low solubility and disintegration (Schafer & Zandbiglari 2003), good adhesion (Eldeniz *et al.* 2005), antimicrobial action (Kayaoglu *et al.* 2005) and good biological properties (Willershausen *et al.* 2000).

The purpose of this laboratory study was to evaluate the adhesion of AH Plus and Epiphany endodontic sealers to human root canal dentine submitted to irradiation with a 980 nm diode laser at different parameters of power (1.5 and 3.0 W) and frequency [continuous wave (CW) and 100 Hz], using the push-out test.

Materials and methods

Sixty roots of maxillary/mandibular canines were sectioned transversally 4 mm below the cementoenamel junction to provide 4-mm-thick dentine discs that were centred inside aluminium rings (16 mm diameter and 4 mm high) and embedded in acrylic resin. The aluminium rings containing the dentine discs were placed in a parallelometer and their coronal and apical surfaces were flattened and polished using wet 100-, 180-, 220- and 300-grit sandpapers (Bosch, São Paulo, SP, Brazil) for 15 s each. The root canal of each specimen was prepared using a tapered diamond bur (PM720G; KG Sorensen Ind. e Com. Ltda, Barueri, São Paulo, SP, Brazil) attached to a low-speed handpiece that was coupled to the arm of the parallelometer. This arm was lowered to a predetermined depth and a space for sealer placement was created with the following dimensions: larger diameter = 2.70 mm; smaller diameter = 2.30 mm and length = 4 mm. During preparation, the canals were irrigated with distilled water and then flooded with 2 mL 1% NaOCl (5 min), 17% EDTA (5 min) and flushed again with distilled water for 1 min.

Next, the specimens were randomly assigned to five groups (n = 12), one control group (no laser irradiation) and four experimental groups that were submitted to laser irradiation with different parameters of power (1.5 and 3.0 W) and frequency (CW and 100 Hz), as follows: 1.5 W/CW; 1.5 W/100 Hz; 3.0 W/CW; 3.0 W/100 Hz. The laser source was a gallium–aluminium arsenide (GaAlAs) semiconductor 980 nm diode laser (SIROlaser 2.2; SIRONA Dental, Bensheim, Germany). The laser beam was delivered to the root canal walls of each specimen using a 200-μm-diameter flexible optical fibre with helicoidal movements along the canal at a delivery rate of approximately 1.0 mm s^{-1} within 8 s. Only a single forward–backward movement was performed maintaining the fibre tip in contact with the root canal dentine wall. Next, 2 mm of the tip of the laser fibre were removed and a new application was performed. The canals remained dry during laser irradiation.

Thereafter, half of the specimens in each group had their canals filled with AH Plus sealer (Dentsply, Rio de Janeiro, RJ, Brazil) and half with Epiphany (Pentron Clinical Technologies LLC), which were handled according to the manufacturers' instructions. The specimens were placed immediately at 37 °C and 95% humidity for a period three times greater than the regular setting time of the sealer to assure a complete polymerization of each sealer before the specimens were submitted to the push-out test (Sousa-Neto *et al.* 2002, Sousa-Neto *et al.* 2005).

Subsequently, the specimens were fixed securely in a metallic apparatus by two screws in the horizontal plane. For the push-out test, a stainless steel support was used to hold the samples (metallic ring + dentine cylinder) in an Instron 4444 universal testing machine (Instron Corporation, Canton, MA, USA) in such a way that the side with the smaller diameter of the root canal faced upwards and was aligned to the shaft that would exert pressure load on the sealer (apical coronally). This method assured the alignment of the specimen in a reproducible manner, and also avoided contact of the shaft with the dentine during testing. The machine was calibrated at a constant speed of 1 mm/min with 1.4-mm-diameter stainless steel cylindrical tip. A constant load was applied until sealer dislodgement was judged to have occurred. The maximum load was then converted to stress by dividing this load by the area of sealer union (Fig. 1). These data were submitted to statistical analysis by ANOVA and Tukey's test ($P < 0.05$).

After the push-out test, a longitudinal groove was prepared on each specimen using a double-faced diamond disc (KG Sorensen) at a low-speed handpiece (Dabi Atlante, Ribeirão Preto, Brazil) with care not to reach the interior of the root canals. The specimens were fractured using a double-bevel chisel and a surgical hammer to expose the internal portion of the canals. The specimens were examined with a scanning electron microscope (Jeol JSM 5410; Jeol Technic Co., Tokyo, Japan) to evaluate the presence/absence of sealer inside the dentinal tubules and to determine the failure modes (adhesive, cohesive and mixed) that occurred for displacement of the sealer from the specimen.

Results

The mean, median, maximum, minimum and standard deviations of the bond strength values (in MPa) for

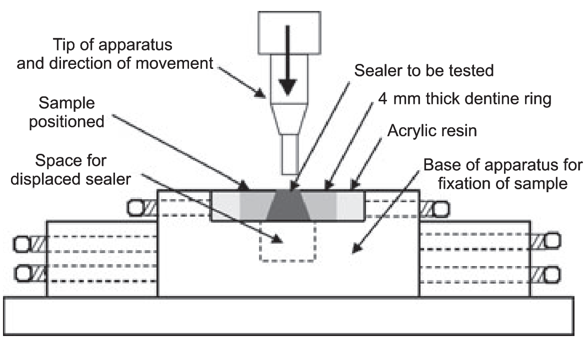

Figure 1 Schematic illustration of the sample positioned on the apparatus for alignment and application of load in the Universal testing machine.

Table 1 Bond strength values in MPa for the displacement of sealer from specimens

	No laser (control)	Diode laser irradiation			
		1.5 W/CW	1.5 W/100 Hz	3.0 W/CW	3.0 W/100 Hz
AH Plus					
Mean	3.86	9.33	10.94	7.01	7.48
Median	3.8	9.47	10.98	6.62	7.94
Maximum	4.79	10.82	14.74	11.39	9.2
Minimum	3.19	7.17	7.99	4.73	4.77
SD	0.6	1.4	2.3	2.44	1.61
Epiphany					
Mean	1.75	4.42	2.76	1.8	4.15
Median	1.7	4.54	3.18	1.91	4.33
Maximum	2.65	5.84	5.26	2.27	5.53
Minimum	1.07	2.7	0.12	1.29	2.56
SD	0.69	1.12	1.83	0.34	1.15

displacement of the sealer from the specimens in the push-out test are given in Table 1.

There was statistically significant difference ($P < 0.05$) amongst the groups regarding the root canal sealers. Tukey's test showed that the specimens irradiated with the diode laser using the different combinations of power and frequency (1.5 W/CW, 1.5 W/100 Hz, 3.0 W/CW and 3.0 W/100 Hz) and filled with AH Plus had significantly higher bond strength values than the specimens irradiated with the same power/frequency settings but filled with Epiphany.

Regardless of the power (1.5 and 3.0 W) or frequency (CW and 100 Hz), the specimens irradiated with the diode laser and filled with AH Plus sealer had significantly higher bond strength values ($P < 0.05$) than the respective control group (no laser irradiation). On the other hand, regardless of the power/frequency settings, the laser-irradiated specimens filled with Epiphany did not differ significantly from the respective control group. There was no statistically significant difference ($P > 0.05$) between the control groups (no laser irradiation).

The failure modes observed at the dentine/sealer interface are given in Table 2. Irrespective of laser application, there was a predominance of mixed failures for AH Plus (67%) and adhesive failures for Epiphany (77%). Figures 2 and 3 show photomicrographs of the dentine/sealer interface after the push-out test in lased-irradiated groups of both sealer.

Discussion

Recent advances in dental research have incorporated laser technology as a potential adjunct in root canal treatment (Moritz *et al.* 2001, Stabholz *et al.* 2004), aimed at reducing the number of micro-organisms within the root canal system and removing smear layer to improve sealer adhesion to the root canal walls (Sousa-Neto *et al.* 2005, Wang *et al.* 2005).

The GaAlAs diode laser used in the present study has a 980 nm wavelength, output power within the 0.5–7.0 W range and can emit energy in both continuous mode (CW) and pulsed (chopped) mode (frequency ranging from 1 to 10 000 Hz). The choice for the power (1.5 and 3.0 W) and frequency (CW and 100 Hz) parameter settings used in this work were based on the results of a recent study (Alfredo *et al.* 2007) that demonstrated that these parameters yielded a temperature raise approximately 10 °C, which does not exceed the limit supported by the periapical tissues (Eriksson & Albrektsson 1983).

The findings of the present study showed that the specimens irradiated with the 980 nm diode laser required a greater load to displace AH Plus sealer from the dentinal walls, compared with the nonlased specimens. This result might be attributed to the alterations caused by the laser on dentine surface, such as fissures and topographic changes (Wang *et al.* 2005). These effects are consistent with those observed by Turkmen *et al.* (2000) and Sousa-Neto *et al.* (2005), who reported that the application of Nd:YAG and Er:YAG lasers, respectively, in root canals increased the surface area because of creation of microirregularities on dentine. In addition, AH Plus sealer penetrates deeper into these microirregularities because of its high flow rate and longer setting time (Saleh *et al.* 2002, 2003). The favourable diffusion of the sealer through the dentinal tubules allied to the cohesion between the sealer molecules (Sousa-Neto *et al.* 2002) increase the resistance to removal and/or displacement from

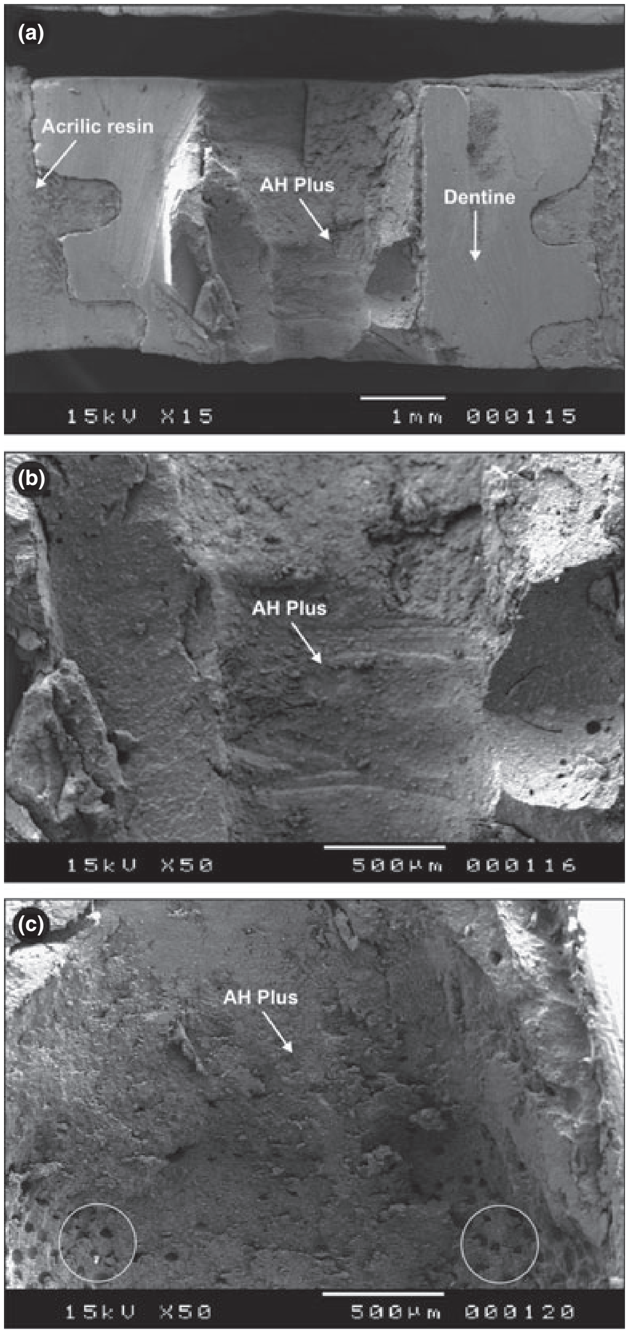

Figure 2 SEM micrographs of the interface of the root canal dentine wall of specimens irradiated with diode laser and filled with AH Plus. (a and b) Sealer adhered to the whole dentinal wall – cohesive failure (1.5 W). (c) Sealer adhered to regions of the dentinal wall (arrow) and the presence of dentinal tubules (circles) – mixed failure (3.0 W).

Table 2 Type and number of failure at the sealer–dentin interface for the different groups

Failure modes	AH Plus sealer: Laser irradiation	AH Plus sealer: No laser (control)	*n* (%)	Epiphany sealer: Laser irradiation	Epiphany sealer: No laser (control)	*n* (%)
Adhesive	0	1	1 (3)	19	4	23 (77)
Cohesive	8	1	9 (30)	0	0	0 (0)
Mixed	16	4	20 (67)	5	2	7 (23)
Total	24	6	30 (100)	24	6	30 (100)

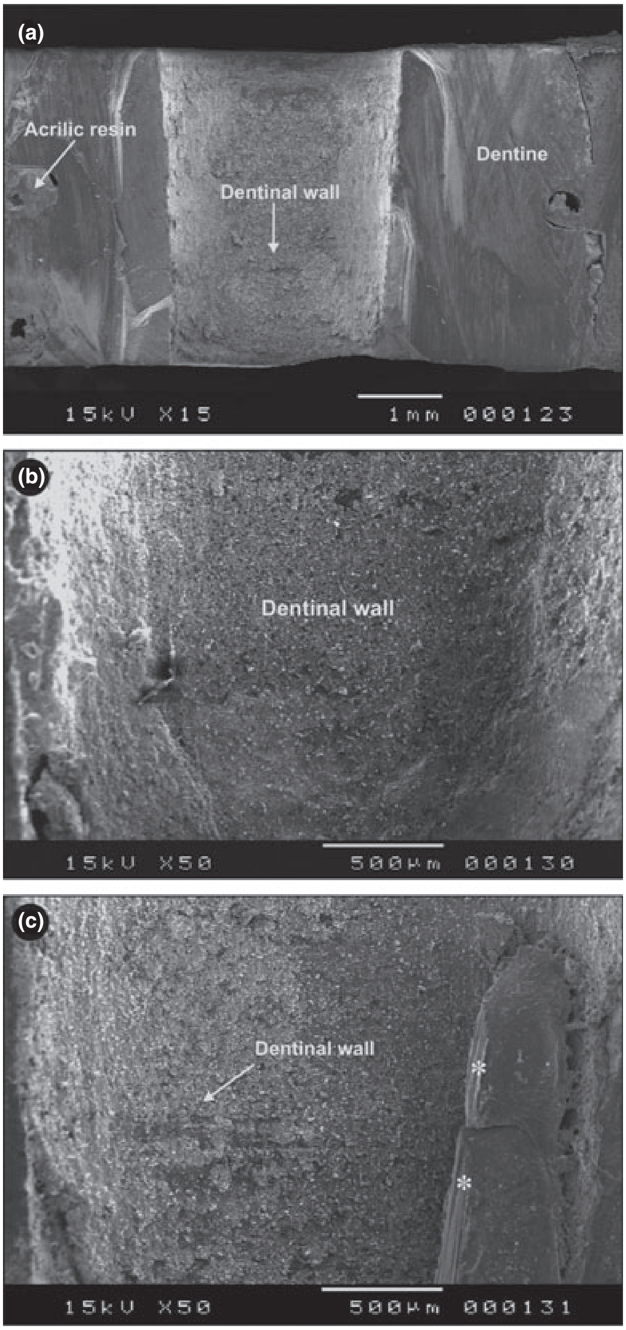

Figure 3 SEM micrographs of the interface of the root canal dentine wall of specimens irradiated with diode laser and filled with Epiphany. (a and b) Root canal dentine wall without sealer – adhesive failure (1.5 W). (c) Sealer adhered to part of the dentinal wall (asterisks) and absent in the remainder (arrow) – mixed failure (3.0 W).

dentine surface. In the present study, this meant higher adhesion.

Epiphany sealer had the lowest bond strength values in both irradiated and nonirradiated groups, which may be explained by the occurrence of physicochemical interferences during polymerization and primer interaction with the root canal walls submitted to different laser treatments. Composite resin polymerization is inhibited by the presence of oxygen and approximately 40–60% of the carbon bonds remain unsaturated (Finger *et al.* 1996, Franco *et al.* 2002). It is likely that it inhibited the polymerization of Epiphany at sealer/dentine interface and inside the dentinal tubules.

Failures at the sealer–dentine interface may also occur because of the polymerization of the methacrylate-based resin sealer immediately after its placement into the root canal (Tay *et al.* 2005). In addition, the coronal photoactivation of the sealer, following the manufacturers' instructions, may reduce its flow and limit its contact with the primer and hence its penetration into the dentinal tubules.

Another aspect that may interfere with the polymerization reaction of the sealer is the lack of photoactivation throughout the specimen extension, which contributes to its incomplete polymerization, leaving residual monomers in the sealer at the deepest regions of the specimen. Regarding the primer, there may be interferences in the chemical reaction with traces of chemical substances during biomechanical preparation, as well as with the substrate, resulting from the treatment of the dentinal walls.

The physicochemical interferences may explain the large number of adhesive failures (77%) at the Epiphany sealer–dentine interface On the other hand, AH Plus sealer had a larger number of mixed failures (67%), which indicates a greater adhesion. The statistical analysis revealed that the specimens irradiated with the diode laser and filled with AH Plus had a significantly higher bond strength. The results obtained for Epiphany sealer in the present study were not higher than those obtained with AH Plus sealer, which is consistent with the findings of Gesi *et al.* (2005).

The different laser parameter settings used in the present study did not interfere with sealer adhesion because the energy increments were probably not sufficient to promote ultrastructural alterations that could differentiate the bond strength of the sealers to dentine.

Another aspect that should be considered in the analysis of the obtained results is that the goal of this study was to evaluate the adhesion AH Plus and Epiphany sealers to laser-irradiated dentine. The heat deposition on lased-irradiated tissue may cause drying and contraction, which could contribute to a fissured surface (Turkmen *et al.* 2000). Besides, in laser-irradiated surfaces, the absence of smear layer and exposure of dentine tubules (Takeda *et al.* 1999) can facilitate the adaptation of the endodontic materials to root canal walls, thus improving the bond strength (Sousa-Neto *et al.* 2005). The Epiphany/Resilon system was not used in this experimental model because its monoblock effect could interfere with the bond strength values (Teixeira *et al.* 2004).

In the present study, 980 nm diode laser irradiation produced alterations on dentine surfaces that increased the bond strength of AH Plus sealer. However, Schoop *et al.* (2006) have reported that this energy is poorly absorbed by the mineralized dental surfaces, thus allowing its propagation throughout the dentine substrate. Gutknecht *et al.* (2004) have suggested that the action of this laser occurs in depth up to 500 μm. Further research is required to investigate whether the potential action in depth of this laser may reduce the microbial content within the dentinal tubules of the root canal.

Conclusion

The application of 980 nm diode laser in the human root canal dentine increased the bond strength of AH Plus sealer, but did not affect the adhesion of Epiphany sealer.

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