Ex vivo study of the adhesion of an epoxy-based sealer to human dentine submitted to irradiation with Er : YAG and Nd : YAG lasers

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Abstract

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Aim To evaluate the adhesion of an epoxy-based sealer to human dentine submitted to irradiation with Er : YAG or Nd : YAG laser at various parameters.

Methodology Ninety maxillary canine teeth were sectioned transversely at the cemento-enamel junction and at the root tip to leave an 8-mm-long cylinder. The tooth specimen was centred in a metallic ring (16 mm diameter and 8 mm height) and embedded in acrylic resin. The root canals were prepared using a low-speed handpiece and a conical diamond bur, which was attached to a paralleling device. This bur was lowered to a depth previously determined by a silicone stop. Specimens were divided into nine groups: group I, dentine was treated with 2 mL of 17% EDTAC for 5 min. Groups II–V were irradiated with Er : YAG laser at the following parameters: group II – 8 Hz and 200 mJ input (120 mJ output); group III – 8 Hz and 400 mJ input (240 mJ output); group IV – 16 Hz and 200 mJ input (120 mJ output); group V – 16 Hz and 400 mJ input (240 mJ output). Groups VI–IX were irradiated with Nd : YAG laser at the following parameters: group VI – 10 Hz and 1 W input (0.4 W output); group VII – 10 Hz and 2 W input (0.8 W output); group VIII – 15 Hz and 1 W input (0.4 W output); group IX – 15 Hz and 2 W input (0.8 W output). The root canals were filled with an epoxy-based root canal sealer and submitted to a push-out test.

Results Statistical analysis showed significant differences ($P < 0.01$) between Er : YAG and Nd : YAG laser treatments at the higher frequencies compared with 17% EDTAC. Greater adhesion values were obtained for groups IV and V (Er : YAG laser) and groups VIII and IX (Nd : YAG laser), which were statistically different from groups II and III (Er : YAG laser) and groups VI and VII (Nd : YAG laser). Treatment with only 17% EDTAC had the lowest adhesion values.

Conclusions An increase in frequency, independent of power settings, of the lasers used in this study increased adhesion of an epoxy-based root canal sealer.

Keywords: adhesion, laser and irradiation, root canal sealer.

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Introduction

To obtain reproducible results and more reliable comparisons of research data, Specification 57 of the American Dental Association (American National Standards Institute 1984) standardized the following tests for evaluation of the physical properties of root canal sealers: working time, flow rate, film thickness, setting time, radiopacity, solubility, disintegration and dimensional stability. This specification does not have any models for adhesion or infiltration tests.

Grossman (1976) and Ørstavik (1983) reported different experimental models designed to evaluate the adhesion of root canal sealers. Grossman (1976) proposed the use of an apparatus built from a T-shaped shaft with two tackles and a string. One of the ends of

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the string is connected to the material to be tested and the other end receives additional load until the material separates from the dentine surface. The necessary mass for the rupture is related to the local gravity acceleration value, thus obtaining the traction force. The traction tension is calculated from the bonded area. Ørstavik (1983) proposed the use of an universal testing machine. Various researchers (Wennberg & Ørstavik 1990, Sousa-Neto *et al.* 2002, Najar *et al.* 2003, Picoli *et al.* 2003) have performed the adhesion test by this method, which allows better precision of the applied forces, i.e. intensity, velocity and direction and thus better reproducibility of the experiment and more reliable data. In addition, the values of tensile strength are expressed in megaPascal (MPa), which allows the comparison of results with other studies.

The adhesion of a root canal sealer may be influenced by many factors, amongst them the presence of smear layer. According to Kennedy *et al.* (1986), smear layer is a negative factor in root canal sealing, because it forms an interface between the sealing material and the root canal walls, thus reducing adhesion.

Different chemical solutions have been recommended for use with instrumentation of root canals to remove debris, smear layer and disinfect the root canal. Amongst the recommended solutions, ethylenediaminetetraacetic acid (EDTA) is the most popular for smear layer removal (Hulsmann *et al.* 2003).

Recently, Er : YAG and Nd : YAG lasers have been studied for applicability in endodontics. Er : YAG lasers remove smear layer from the root canal walls, exposing dentinal tubules (Takeda *et al.* 1998, Sousa-Neto *et al.* 2002, Brugnera-Júnior *et al.* 2003), whilst Nd : YAG lasers remove smear layer and melt and recrystallize dentinal tissue (Israel *et al.* 1997) and reduce apical leakage in teeth obturated with epoxy-based root canal sealers (Park *et al.* 2001).

A great variation of energy and frequency have been used for irradiation with these lasers. Thus, the aim of the present study was to evaluate the adhesion of an epoxy resin-based endodontic sealer to human dentine submitted to irradiation with Er : YAG or Nd : YAG laser at various parameters, using the push-out test (Patierno *et al.* 1996).

Materials and methods

Ninety maxillary canines were sectioned transversally at the cemento-enamel junction and at the root tip to leave an 8-mm cylinder that was then centred inside aluminium rings (16 mm diameter and 8 mm high) and embedded in acrylic resin.

The aluminium rings containing the dentine cylinder were placed in a parallelometer and their coronal and apical surfaces were flattened and made parallel, until a final length of 8 mm was obtained. The root canals of each sample were prepared using a low-speed hand piece and a conical diamond bur (893-047; Brasseler, Savannah, GA, USA), which was attached to the arm of the parallelometer. This arm was lowered to a depth previously determined by a silicone stop, obtaining a standardized sample with the following dimensions: larger diameter = 3.3 mm, smaller diameter = 2.6 mm and length = 8 mm. During preparation, canals were irrigated with distilled water.

Samples were randomly divided into nine groups of 10 teeth each and root canals were submitted to the following treatments: group I, 2 mL of 17% EDTAC for 5 min (control group); group II, irradiation with Er : YAG laser (Opus 20; Opus Dent, Israel) for 1 min, at 8 Hz and 200 mJ input (120 mJ output); group III, irradiation with Er : YAG laser for 1 min, at 16 Hz and 200 mJ (input, 120 mJ output); group IV, irradiation with Er : YAG laser for 1 min, at 8 Hz and 400 mJ input (240 mJ output); group V, irradiation with Er : YAG laser for 1 min, at 16 Hz and 400 mJ input (240 mJ output); group VI, irradiation with Nd : YAG laser (Fotona Medical Lasers, Ljubljana, Slovenia) for 1 min, at 10 Hz and 1 W input (0.4 W output); group VII, irradiation with Nd : YAG laser for 1 min, at 15 Hz and 1 W input (0.4 W output); group VIII, irradiation with Nd : YAG laser for 1 min, at 10 Hz and 2 W input (0.8 W output); group IX, irradiation with Nd : YAG laser for 1 min, at 15 Hz and 2 W input (0.8 W output).

Er : YAG irradiation was performed with a sapphire point of 17 mm length and 1.3 mm diameter. Nd : YAG laser was applied with a quartz 0.25-mm fibreoptic tip. During irradiation with both lasers, canals were irrigated with distilled and deionized water.

The dentine cylinders were then filled with an epoxy resin-based cement (Sealer 26; Dentsply, Petrópolis, RJ, Brazil) and were placed immediately at 37 °C and 95% humidity for a period three times greater than the regular setting time of the sealer (Sealer 26, setting time 18 h; Sousa-Neto *et al.* 2002). Subsequently, samples were dried and fixed securely in a metallic apparatus (Fig. 1) by two screws in the horizontal plane. For the test, a stainless steel support was used to hold the samples (metallic ring + dentine cylinder) in the Instron 4444 universal testing machine (Instron

Corporation, Canton, MA, USA) so 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, Fig. 1). The tip of apparatus used for the load application in the push-out test had diameter of 1.8 mm; the smaller end of the dentine sample (where the tip was placed) was 2.6 mm in diameter, leaving a thin cement layer (0.4 mm) surrounding the tip. 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^{-1}. The tensile load was applied, and the load required to cause failure of the bond was recorded in MPa. These data were submitted to statistical analysis with ANOVA, and the Tukey test ($P < 0.01$).

Results

Statistical analysis (ANOVA and Tukey post-test) revealed significant differences between groups ($P < 0.01$). Table 1 presents the data obtained.

Greater adhesion values were obtained when root canal walls were irradiated at higher frequencies of Er : YAG laser (group IV: 200 mJ input, 120 mJ output and 16 Hz; group V: 400 mJ input, 240 mJ output and 16 Hz) and Nd : YAG laser (group VIII: 1 W input, 0.4 W output and 15 Hz; group IX: 2 W input, 0.8 W output and 15 Hz). These were statistically different from the groups irradiated with Er : YAG (group II: 200 mJ input, 120 mJ output and 8 Hz; group III: 400 mJ input, 240 mJ output and 8 Hz) and Nd : YAG (group VI: 1 W input, 0.4 W output and 10 Hz; group VII: 2 W input, 0.8 W output and 10 Hz), which presented intermediate values. The lowest adhesion values for the epoxy-based sealer were obtained when the root canal walls received only 17% EDTAC for 5 min.

Discussion

Adhesion, which is the capacity of a root canal sealer to adhere to the root canal walls, has been studied since the development of the experimental model proposed by Grossman (1976). This was improved by Ørstavik (1983), who used the universal testing machine to standardize the test, making it reproducible and more reliable.

The present study, using the method proposed by Ørstavik (1983), focused on two variables considered to be of influence on adhesion of root canal sealers, i.e. surface submitted to the test and the treatment of this surface. Previous studies analysing the surface submitted to the test included dentine discs obtained from crowns of third molars (Tagger *et al.* 2002), coronal dentine from the cervical portion of molars (Sousa-Neto *et al.* 2002, Najar *et al.* 2003, Picoli *et al.* 2003) and gutta-percha discs (Lee *et al.* 2002, Saleh *et al.* 2002, 2003). The adhesive capacity of internal radicular dentine is ambiguous and depends on the pattern of the dentine tubule, which differs not only from one tooth to another but can also differ within the same tooth. Thus, there was a need to develop a method that would allow the evaluation of this adhesive capacity using internal radicular dentine as a sample, to allow an understanding of how adhesion occurs on dentine walls in a situation that resembled the *in vivo* situation.

Patierno *et al.* (1996) proposed a method to evaluate resistance to traction of composites in cervical radicular dentine, in which the teeth were sectioned transversely to produce 4-mm-thick cylindrical samples. Root canals were enlarged, adhesive was applied on the dentinal

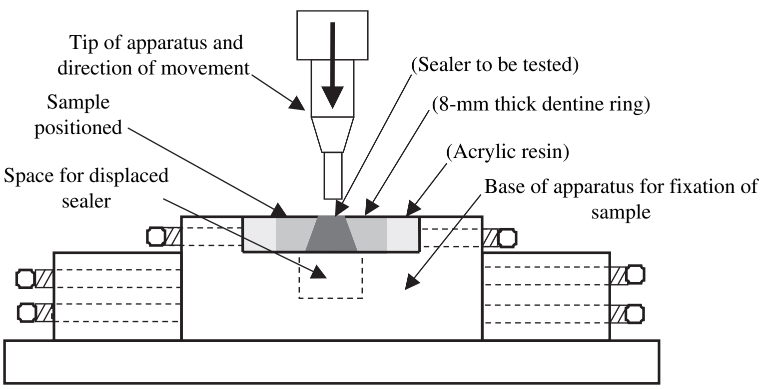

Figure 1 Scheme of the sample positioned on the apparatus for alignment and application of load in the universal testing machine (Instron 4444; Instron Corporation, Canton, MA, USA).

Table 1 Values, in MPa, for displacement of the sealer from the specimen

Group	Mean	SD	Range
Er : YAG (400 mJ, 16 Hz)	12.5416	1.0744	11.2561–13.5589
Er : YAG (200 mJ, 16 Hz)	11.1451	1.0416	10.1345–13.1375
Nd : YAG (2 W, 15 Hz)	10.3013	1.1585	7.9618–12.9966
Nd : YAG (1 W, 15 Hz)	9.5542	1.0746	7.9755–12.1782
Er : YAG (200 mJ, 8 Hz)	9.3383	0.8201	8.3547–10.2250
Nd : YAG (1 W, 10 Hz)	8.5664	1.086	6.2589–10.7294
Er : YAG (400 mJ, 8 Hz)	8.1703	1.1077	7.0806–9.3561
Nd : YAG (2 W, 10 Hz)	7.8706	1.1485	6.0185–9.8854
EDTAC	5.0081	0.6892	4.3696–5.7198

walls and resin was placed inside it for performing the push-out test.

Thus, the experimental model employed in this study was developed from samples obtained from the cervical region of human canine roots. The following advantages of this method, compared with other dentinal surfaces, are: (i) The sealer is placed in contact with the dentinal surface inside the root canal, instead of a flat surface from the crown region, which presents a different pattern of tubules. When the sample is filled with sealer, it adapts to the shape of the canal and penetrates into the dentinal tubules, promoting a mechanical retention similar to clinical conditions. In other experimental models, the sealer is placed on a flat surface. (ii) The results obtained are derived from shear strength forces, and not traction as in previous models. Saleh *et al.* (2002) evaluated the effect of dentine pretreatment on the adhesion of root canals sealers, where the dentine/sealer/gutta-percha interface was tractioned until rupture with the tensile load in the same direction as the dentine tubules. In the present study, the sealer/dentine interface was also evaluated; however, the force was applied perpendicular to the dentine tubules, which is similar to real forces that occur in root canal. It is important to emphasize that because the cement used in this study was a resin-based sealer (Sealer 26; Dentsply) it was possible to apply the push-out test, where none of the samples fractured through cohesive failure within the sealer; only failure of the adhesion at the sealer/dentine interface was recorded. (iii) The laser was applied inside the root canal and not perpendicular to a flat surface. According to Brugnera-Júnior *et al.* (2003), smear layer removal depends on the angle of incidence of the fibreoptic tip and the dentinal surface.

The second variable is the dentinal surface treatment, which aims to remove the smear layer. According to Kennedy *et al.* (1986), smear layer is a negative factor in root canal sealing because it adheres easily to the sealing material and root canal wall interface reducing the adhesion of sealers. Smear layer removal has been carried out with different substances (Hülsmann *et al.* 2003, Saleh *et al.* 2003) or lasers (Sousa-Neto *et al.* 2002, Najar *et al.* 2003, Picoli *et al.* 2003).

Thus, in this study, the adhesion of an epoxy-based sealer to human dentine submitted to irradiation with Er : YAG and Nd : YAG lasers at various parameters was evaluated and compared with 17% EDTAC, which acted as a control group. The greater adhesion values that were obtained when the irradiation frequency was increased, both with the Er : YAG (200 and 400 mJ, 16 Hz) and Nd : YAG lasers (1 and 2 W, 15 Hz) were statistically different from 17% EDTAC.

With the Er : YAG laser, the increase in frequency tends to remove the smear layer as well as create morphological alteration of the dentinal tissue, increasing the area of dentine with irregularities. These alterations were observed using the scanning electron microscope by Takeda *et al.* (1998) and Sousa-Neto *et al.* (2002). These modifications caused by irradiation with the Er : YAG laser may explain the increase in adhesion of the Sealer 26 cement. According to Sousa-Neto *et al.* (2002), the epoxy-based sealer has better penetration in the irregularities, thus increasing mechanical retention and resistance to shear forces, which can be translated as greater adhesion. Furthermore, Sousa-Neto *et al.* (2002) also showed that epoxy-based sealers are able to penetrate into the dentinal tubules exposed by smear layer removal, partially filling them and forming tags similar to that which occurs with dentine adhesives.

Nd : YAG lasers are able to reduce dentine permeability (Miserendino *et al.* 1995), remove smear layer, and melt and recrystallize dentine tissue (Israel *et al.* 1997). Using SEM, Lin *et al.* (2001) observed craters in human dentine irradiated with Nd : YAG laser, whilst Türkmen *et al.* (2000) described the dentinal surface as irregular after irradiation with this laser. Amongst these characteristics, fusion and increased irregularities of the surface can explain the similar adhesion values that were found with Er : YAG laser when the frequency was increased.

The present findings must be complemented by studies to determine a frequency that is safe in terms of heat generation on the external surface of the root, but also efficient in increasing adhesion of the sealer to the root canal walls.

References

American National Standards Institute (1984) Specification no. 57 for endodontic filling materials. *Journal of the American Dental Association* **108**, 88.

Brugnera-Júnior A, Guerisoli DMZ, Marchesan MA, Spanó JCE, Pécora JD (2003) In vitro evaluation of smear layer removal by Er:YAG laser application with five different fiberoptic tip withdrawal techniques. *Proceedings of Laser in Dentistry IX* **4950**, 188–94.

Grossman LI (1976) Physical properties of root canal cements. *Journal of Endodontics* **6**, 66–75.

Hülsmann M, Heckendorff M, Lennon A (2003) Chelating agents in root canal treatment: mode of action and indications for their use. *International Endodontic Journal* **36**, 810–30.

Israel M, Cobb CM, Rossman JA, Spencer P (1997) The effects of CO_2, Nd:YAG and Er:YAG lasers with and without surface coolant on tooth root surfaces. An in vitro study. *Journal of Clinical Periodontology* **24**, 595–602.

Kennedy W, Walker WA, Gouch RW (1986) Smear layer removal effects on apical leakage. *Journal of Endodontics* **12**, 21–7.

Lee KW, Williams MC, Camps JJ, Pashley DH (2002) Adhesion of endodontic sealers to dentin and gutta-percha. *Journal of Endodontics* **28**, 684–8.

Lin CP, Lee BS, Lin FH, Kok SH, Lan WH (2001) Phase, compositional, and morphological changes of human dentin after Nd:YAG laser treatment. *Journal of Endodontics* **27**, 389–93.

Miserendino LJ, Levy GC, Rizoiu IM (1995) Effects of Nd:YAG laser on permeability of root canal wall dentin. *Journal of Endodontics* **21**, 83–7.

Najar AL, Saquy PC, Vansan LP, Sousa Neto MD (2003) Adhesion of a glass–ionomer root canal sealer to human dentine. *Australian Endodontic Journal* **29**, 20–2.

Ørstavik D (1983) Physical properties of root canal sealers: measurement of flow, working time, and compressive strength. *International Endodontic Journal* **16**, 99–107.

Park DS, Yoo HM, Oh TS (2001) Effects of Nd:YAG laser irradiation on the apical leakage of obturated root canals: an electrochemical study. *International Endodontic Journal* **4**, 318–21.

Patierno JM, Rueggeberg FA, Anderson RW, Weller RN, Pashley DH (1996) Push-out strength and SEM evaluation of resin composite bonded to internal cervical dentin. *Endodontics & Dental Traumatology* **12**, 227–36.

Picoli F, Brugnera-Junior A, Saquy PC, Guerisoli DMZ, Pécora JD (2003) Effect of Er:YAG laser and EDTAC on the adhesiveness to dentine of different sealers containing calcium hydroxide. *International Endodontic Journal* **36**, 472–5.

Saleh IM, Ruyter IE, Haapasalo M, Ørstavik D (2002) The effect of dentine pre-treatment on the adhesion of root canals sealers. *International Endodontic Journal* **35**, 859–66.

Saleh IM, Ruyter IE, Nat R, Haapasalo M, Ørstavik D (2003) Adhesion of endodontic sealers: scanning electron microscopy and energy dispersive spectroscopy. *Journal of Endodontics* **29**, 595–601.

Sousa-Neto MD, Marchesan MA, Pécora JD, Brugnera-Júnior A, Silva-Sousa YTC, Saquy PC (2002) Effect of Er:YAG laser on adhesion of root canal sealers. *Journal of Endodontics* **28**, 185–7.

Tagger M, Tagger E, Tjan AH, Bakland LK (2002) Measurement of adhesion of endodontic sealers to dentin. *Journal of Endodontics* **28**, 351–4.

Takeda FH, Harashima T, Kimura Y, Matsumoto K (1998) Efficacy of Er:YAG laser irradiation in removing debris and smear layer on root canal walls. *Journal of Endodontics* **24**, 548–51.

Türkmen C, Gunday M, Karaçorlu M, Basaran B (2000) Effect of CO_2, Nd:YAG, and ArF Excimer laser on dentin morphology and pulp chamber temperature: an in vitro study. *Journal of Endodontics* **26**, 644–8.

Wennberg A, Ørstavik D (1990) Adhesion of root canal sealers to bovine dentine and gutta-percha. *International Endodontic Journal* **23**, 13–9.

