Evaluation of a self-adhesive composite in dentin surfaces

Preparation with Er,Cr:YSGG laser

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Introduction

Flowable resin composites appeared in the late 1990s, and they have properties like low modulus of elasticity and high wettability, which are very desirable for the clinical work.[1,2] The development of dentistry has eventually led to minimal invasive dentistry, with smaller preparations, giving the flowable resin composite an important role due to its flow characteristics and easy application.[3]

Self-adhesive flowable resin composites combine a bonding system and a flowable composite. This kind of material is an adequate tool to use in more difficult patients and children, because it allows a restorative procedure with less steps and less time in the dentist chair. The self-adhesive flowable resin composite used in this study was the Vertise™ Flow (VF) from Kerr, was released in the market in 1992, and has the OptiBond™ bonding mechanism to dentine.[4] This material has the characteristics described in the Table 1.

The GPDM (phosphate functional group) forms a chemical bond with the calcium ions on the tooth, and the prepolymerized filler present in the VF minimises the shrinkage and enhance properties of clinical handling. The VF does a micromechanical adhesion with the formation of a hybrid layer, which consists in resin impregnation with collagen fibres and dentin smear layer.[4]

Dentin is a mineralised subtract of the tooth which has an intricate three-dimensional frame, with tubules extending from the pulp to the dentino-enamel junction, intratubular and peritubular dentin. It has 70 % (by weight) of mineral, 20 % of organic component and 10 % of fluid. The composition of the organic matrix is 90 % of fibrillar type I collagen and 10 % of noncollagenous proteins like phosphoproteins and proteoglycans. Because of this complex structure, only a few structure-property relationships can be performed.[5,6]

Actually, the formation of a hybrid layer, with the monomers impregnation into the dentin partially demineralised, and its subsequent polymerisation seems to be the most successful method.[7,8] Erbium lasers cavity preparation results in the absence of smear layer, opened dentinal tubules and micro irregularities on the dentin in result of the removal of the intertubular dentin, outcoming in a dentin surface more suitable to adhesive procedures. The effect of laser on the collagen network is still not completely

Table 1: Self-adhesive flowable resin composite used in this study.

Self-adhesive flowable resin composite

ID	Material	Manufacturer	Compositions
VF	Vertise™ Flow	Kerr	GPDM, HEMA, prepolymerized filler, 1-µm barium glass filler, nanosized colloidal silica, nanosized Ytterbium fluoride

clear, but it is known that laser irradiation can develop microstructural alterations and micro rupture of collagen fibres.[9] The reported bond strengths of composite resin to dentine substrate prepared by erbium lasers have often been confusing and contradictory.[10]

The Er,Cr:YSGG laser has a wavelength of 2.79 µm. The ablation threshold in human dentin is 2.69 to 3.66 J/cm^2.[11] The laser frequency is the number of laser pulses per second.[12] Theoretically, an increase of the laser frequency would result in a smoother surface, which could reduce the gaps between the composite and the dental surface.

The main objective of this study was to evaluate the bond strength of self-adhesive flowable composite Vertise™ Flow in dentin surfaces prepared with Er,Cr:YSGG laser with two different settings.

The null hypotheses to test were: (a) the micro-shear bond strength of the restorations with Vertise™ Flow self-adhesive flowable composite is the same, in dentin surfaces prepared with bur and Er,Cr:YSGG laser; (b) micro-shear bond strength of the restorations with Vertise™ Flow self-adhesive flowable composite is the same, in dentin surfaces prepared with Er,Cr:YSGG laser with the settings 4.5 W, 50 Hz, 50 µs, 70 % air, 90 % water and with the settings 4.5 W, 75 Hz, 50 µs, 70 % air, 90 % water.

Materials and methods

The sample consisted of 15 non carious molars extracted by periodontal or orthodontic reasons. After extraction, the teeth were cleaned and stored in 0.5 % chloramine T at a temperature of about 4 °C to carry out the disinfection and preventing bacterial growth for no longer than three months until used in the experiment. Afterwards, the teeth were numbered by a person assigned, and distributed to the three groups (n=5) by another person assigned.

To obtain the samples for the electron microscopy, two from each group, the coronal occlusal third of the teeth and the roots were removed by bisecting the tooth transversely with a low speed diamond saw and copious supply of water. It resulted in approximately 2 mm thick dentin discs.

To obtain the samples to the Tensile Bond Strength (TBS) test, three teeth from each group were longitudinally sectioned into two parts where the proximal enamel was removed by a disk to expose the dentin.

Groups

The laser samples were submitted to an Er,Cr:YSGG laser (Waterlase iPlus—Biolase Technology Inc., Irvine, CA, USA), with two different settings , with the MZ8 tip, using the beam with an angle of 45° to the surface, with smooth movements in the horizontal and vertical, during twelve seconds in the dentin discs, and eight seconds in the teeth's proximal surface, where the area was smaller than the discs. The dentin samples from the control group were not submitted to any laser treatment:

Group 1: Control Group (n=10) Material: Vertise™ Flow (Kerr, Orange, CA, USA) (without laser surface treatment).

Group 2: Settings 4.5 W, 50 Hz, 50 µs, 70 % air, 90 % water (n=10) (Fig. 7) Material: Vertise™ Flow (Kerr, Orange, CA, USA).

Group 3: Settings 4.5 W, 75 Hz, 50 µs, 70 % air, 90 % water (n=10) (Fig. 8) Material: Vertise™ Flow (Kerr, Orange, CA, USA).

The Vertise™ Flow self-adhesive flowable composite was applied in the dentin surface from each sample, about 3 mm delimited by a tape of polyester (Mylar) and a silicone mold with 3 mm of diameter and 4 mm of high to the samples to the SBS test, directly to dentin in the discs used in the SEM, and lighted cured for 20 seconds with a LED curing light Bluephase C8 (Ivoclar Vivadent) 800 mW/cm^2.

Subsequently, the samples were placed in an incubator at 37 °C with 100 % humidity for 24 hours. Next, they were thermocycled 500 times in baths of 5 °C and 55 °C for 20 seconds at each temperature, to simulate the *in vivo* conditions in the oral cavity. At the end of thermocycling, the samples returned to the incubator for more than 24 hours.

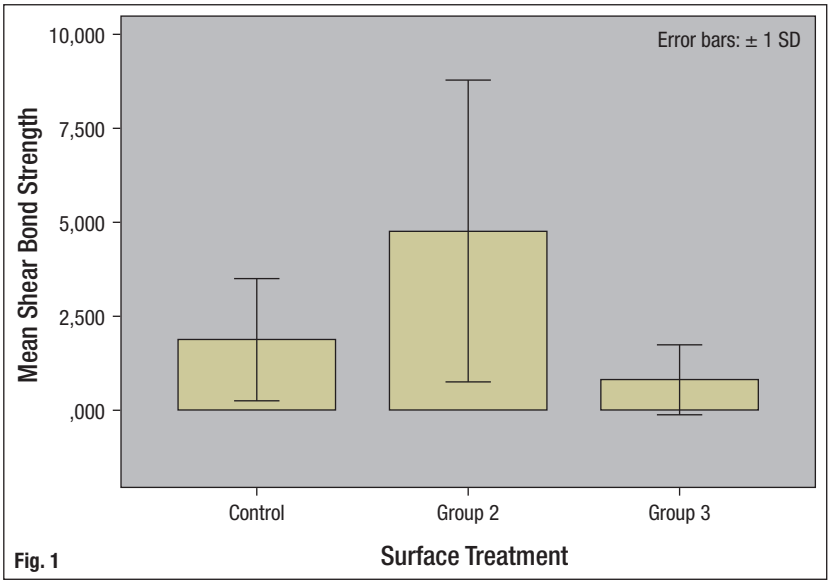


Fig. 1: Mean and standard deviation of the Shear Bond Strength (MPa) in the three groups.

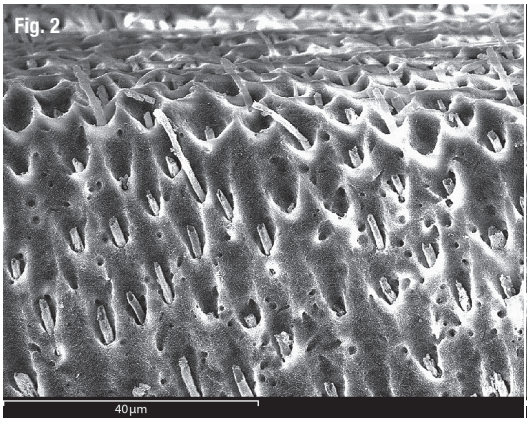


Fig. 2: SEM of the dentin surface without laser preparation (Control), showing the micromorphological aspects of the bonding region produced by Vertise Flow™. Note the regular surface and the wide dentinal tubules with resin tags (x1,500).

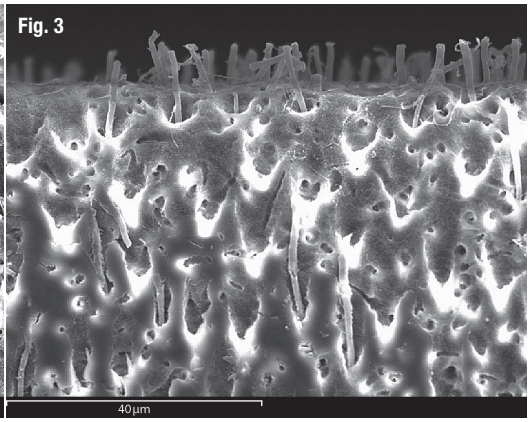


Fig. 3: SEM of the dentin surface without laser preparation (Control), showing the micromorphological aspects of the bonding region produced by Vertise Flow™. Note the sectioned resin tags in the dentin surface (x1,500).

Preparation of samples for scanning electron microscopy

The samples discs were split in half (MD) with a diamond blade mounted on the handpiece resulting in restored hemi-discs. The hemi-discs were prepared to be observed on the SEM. The observations were made using the electron microscope scanning—FE SEM (JEOL JSM 6301F) at 10–15 kV and were focussed in the area resin-dentin interdiffusion. The electron microscopy photomicrographs were taken in a variation of 300 x and 5,000 x magnifications.

Shear Bond Strength (SBS) test

The Shear Bond Strength was performed using the Instron device connected to a computer, (system developed at the University of California, San Francisco). Shear Bond Strength was tested at 1 mm/min.

Statistical analysis

The Shear Bond Strength values were analysed using the Shapiro-Wilk test to control the normality assumptions, because the number of the sample was lower than thirty. The homogeneity assumptions were controlled by the Levene test. Although the data were normally distributed, the Levene test rejected the homogeneity of the variances. Therefore, the Welch test was applied and the Games-Howell post-hoc test was used to compare pairwise, with a confidence interval of 95%.

Results

Shear Bond Strength test (SBS test)

All statistical analyses were performed using SPSS version 21.0 (SPSS Inc., Chicago, IL, USA). Group 2 was the one with the highest Shear Bond Strength mean (4.76 ± 3.99 MPa), although the standard deviation is the highest, and the Group 3 had the lowest Shear Bond Strength mean (0.81 ± 0.93 MPa), as we can see in the graph (Fig. 1). This means of the Shear Bond Strength of the Control Group (1.87 ± 1.61 MPa), of Group 2 (4.76 ± 3.99 MPa) and of the Group 3 (0.81 ± 0.93 MPa), were not statistically different between each other ($p > 0.05$).

Scanning electron microscopy

Control Group

In these dentin samples we saw a regular surface, with resin tags in the surface of the dentin going through the open dentinal tubules. The resin tags appeared to be sectioned, showing an adhesive failure (Figs. 2 and 3).

Group 2

In the analysis of the samples of Group 2, a rougher dentin surface was noted, with several resin tags. It was possible to see a hybrid layer for-

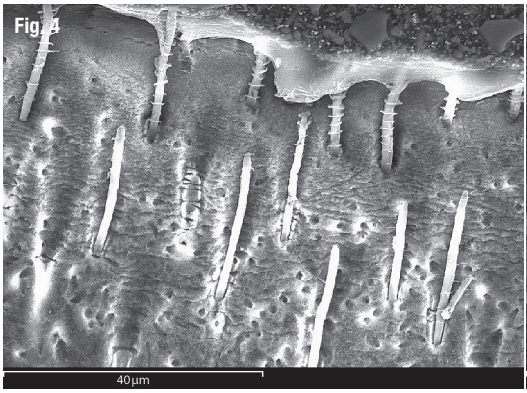


Fig. 4: SEM of the dentin surface pre-treated with Er,Cr:YSGG laser from Group 2, showing long resin tags with lateral branches (x1,500).

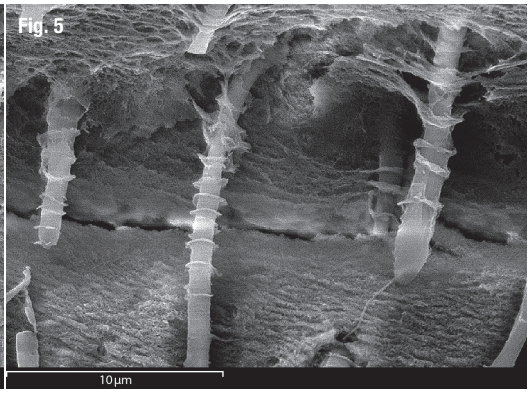
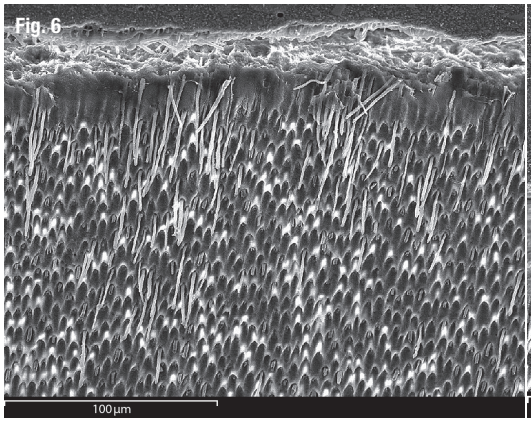


Fig. 5: SEM of the dentin surface pre-treated with Er,Cr:YSGG laser from Group 2, showing long resin tags with lateral branches. The formation of a gap along the interface was observed (x5,000).



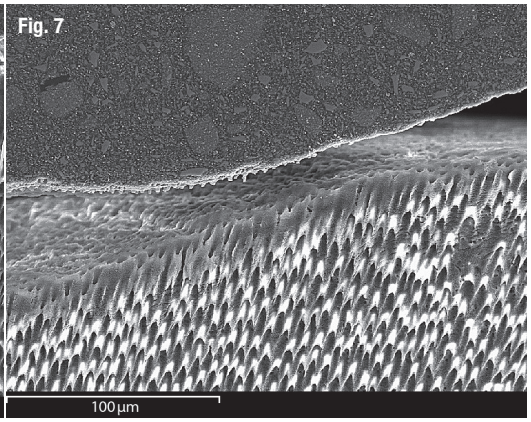


Fig. 6: SEM of the dentin surface pre-treated with Er,Cr:YSGG laser from Group 3, showing the micromorphological aspects of the bonding region produced by Vertise™ Flow. The formation of a gap along the interface was observed (x500).

Fig. 7: SEM of the dentin surface pre-treated with Er,Cr:YSGG laser from Group 3, showing the formation of a gap along the interface (x500).

mation in all the samples and the presence of resin tags with lateral branches. A gap along the interface between the VF and the dentin surface was seen in two of the samples (Figs. 4 and 5).

Group 3

In the dentin surface of the samples of Group 3, it was possible to observe what seems to be melted dentin, with a few open dentinal tubules, and a gap along the interface (Fig. 6). In a sample, it was not possible to observe resin tags in the dentinal tubules, there was almost no hybrid layer and a big gap along the interface between the VF and the dentin surface was observed (Fig. 7). The samples of this group seemed to have a smoother dentin surface than those in Group 2, but rougher than the samples of the Control Group (Figs. 6 and 7).

Discussion

There are already several studies about adhesion in dental surfaces prepared with laser, but mostly with Er:YAG and enamel, and fewer about self-adhesive flowable in dentin, especially with Er,Cr:YSGG laser. Several factors can influence the adhesion to dentin, such as the dentin substrate itself, the treatment and the dentin conditioning.[13,14] The adhesion to dentin was always a greater challenge because of the water and collagen content.[15] The dentin hybridisation is the accepted mechanism to explain the resin-dentin bond, which consists in demineralised dentin with infiltrated monomers and its polymerization.[8]

Several articles showed that dentin surfaces prepared with Er,Cr:YSGG laser appear with open dentinal tubules looking cuff-like, irregular and rough, and are without smear layer.[9] These features of lased dentin theoretically should have better conditions for adhesion.[15] The comparison presented by Beer et al., between dentin surface prepared with Er,Cr:YSGG laser and self-etch system and a dentin surface also prepared with Er,Cr:YSGG laser and etched with 37% phosphoric acid, showed better SBS with the first system.[16] The application of acid in the dental surface prepared with laser dissolves the intertubular dentin, altering the surface produced by laser, and leading to unknown dentin-demineralised depths, which could interfere with the monomers diffusion.[17–19]

Therefore, theoretically we do not need acid etching prior the adhesive to accomplish an adequate adhesion in lased surfaces. Despite the advantages of the dentin surfaces prepared with Er,Cr:YSGG in adhesion,[15] several studies have demonstrated a lower SBS when compared with surfaces treated with conventional methods.[9,15,20,21] However, this subject is still treated controversially.[22,23]

Group 2 presented higher mean values of bond strength (4.76 ± 3.99 MPa), followed by the Control Group (1.87 ± 1.61 MPa). A lower adhesion result was observed in Group 3 (0.81 ± 0.93 MPa). The high standard deviation presenting in Group 2 shows that probably there was more variability in the sample than expected. The age of the dentin samples could be a factor with an impact on the Shear Bond Strength,[24] that was not controlled in this study, and also the sample probably should be higher. None of the SBS differences between the three groups were statistically significant ($p > 0.05$), so neither of the null hypothesises were rejected.

The settings used in Group 2 resulted in a surface without smear layer, which was rougher than the Control Group. The absence of smear layer and open dentinal tubules promotes a better surface for adhesion, leading to a better infiltration of the resin tags, which were observed in the SEM micrographs of this study.[25] A study performed by Yazici et al., comparing the Shear Bond Strength of the VF in human dentin lased with Er:YAG and unlased surfaces, also showed better results in the lased ones.[3]

Moreover, the manufacturer of VF claims that the acidic phosphate group etches the dental sur-

face, creating a chemical bond with the calcium, probably enhancing the adhesion. According to Visuri et al., the main presence of peritubular dentin in the dentin surface treated with laser can explain why the SBS was better than that of the Control Group.[26] This surface results from a higher content of water in the intertubular dentin, leading to more ablation of this substract.[9] The mean of the SBS of the VF in Group 2 (4.76 ± 3.99 MPa) was lower compared to other studies performed by Altunsoy et al., and Yazici et al. in lased dentin with Er:YAG.[1,3]

One of the main reasons that could explain this difference is the use Er:YAG laser in these studies, and the different settings applied.[1,3] In all samples of Group 3, and in one of Group 2, a gap in the interface of the dentin surface and the VF was seen, probably due to thermocycling. According to El-Marhomy et al., the thermocycling influences the marginal gap of composite restorations,[27] because the hot water can accelerate the hydrolysis of the interface components and induce stress between the composite and the dental surface.[28] Despite these findings, the effect of thermocycling is still controversial because some studies showed no influence of thermocycling on gap formation.[29]

One of the objectives of this study was to compare the influence of the laser frequency on the SBS of the VF. The results showed that even if you get a smoother surface with the increasing of the laser frequency, as we can see in the SEM micrographs, the SBS is lower (0.81 ± 0.93 MPa) compared to Group 2 (4.76 ± 3.99 MPa) and Control Group (1.87 ± 1.61 MPa). These results concur with a study performed by Samad-Zadeh et al., in which the authors concluded that the SBS was higher in the laser-textured dentin substrate, with greater spacing patterns.[30]

Comparing the results of this study with other studies is always a challenge because different lasers and parameters influence the laser-tissue interaction, leading to different outcomes. All dental products on the market were produced to work in dental surfaces prepared by conventional methods like the bur. This study and all the studies referred to in this article tested those materials available in the market. The results should be used to develop new products, with a laser-treated dental surface in mind.

Conclusion

Although the bond strength of the Vertise™ Flow was influenced by the type of dentin surface and the laser parameters, the results of each group were not statistically different between each other ($p > 0.05$), showing no significant difference concerning dentin-surface treatment. The increase of only the laser frequency resulted in lower SBS. Additional studies should be carried out in order to reach a better adhesion of self-etch flowable composites in dentin surfaces prepared with Er,Cr:YSGG laser, possibly trying new laser settings._

Editorial note: A list of references is available from the publisher.

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Kurz & bündig

Selbstklebende, fließfähige Harz-Composites verbinden ein Bondingsystem mit einem fließfähigen Composite. Diese Art Material ist ideal für den Einsatz bei schwierigen Patienten sowie Kindern, da es eine Restauration in wenigen Schritten mit einer kürzeren Behandlungsdauer begünstigt. In der vorliegenden Studie wurde das selbstklebende, fließfähige Harz-Composite Vertise™ Flow (VF) der Firma Kerr verwendet.

Das Hauptziel dieser Studie war es, die Bondingstärke des selbstklebenden, fließfähigen Composites Vertise™ Flow an Dentinoberflächen zu ermitteln, welche mithilfe eines Er,Cr:YSGG-Lasers in zwei verschiedenen Voreinstellungen präpariert wurden. Obwohl die Bondingstärke des verwendeten Composites durch die Dentinoberfläche und die Laserparameter beeinflusst wurde, unterschieden sich die Ergebnisse der verschiedenen Versuchsgruppen statistisch nicht voneinander ($p > 0.05$), sodass kein signifikanter Unterschied hinsichtlich der Behandlung der Dentinoberfläche festgestellt werden konnte. Ein Erhöhen der Laserfrequenz bewirkte eine niedrigere SBS (Shear Bonding Strength). Weitere Studien sollten durchgeführt werden, um eine verbesserte Adhäsion der selbstätzenden, fließfähigen Composites an Dentinoberflächen zu erreichen, welche durch Er,Cr:YSGG-Laser präpariert wurden. Dabei könnten weitere Laser-Einstellungen getestet werden.

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