

Original Research Article

Morphologic assessment of dental surface/glass ionomer cement interface: influence of Er:YAG laser pretreatment

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Abstract

Introduction and objective: The aim of this study was to assess the surface and the substrate/glass ionomer cement (GIC) interface after Er:YAG laser irradiation by means of scanning electron microscopy. **Material and methods:** Thirty human third molars were selected and had their roots removed. Crowns were sectioned to obtain discs that were randomly assigned to three groups according to the surface pretreatment: 40% polyacrylic acid (control); Er:YAG laser irradiation (80mJ/2Hz) or Er:YAG laser followed by 40% polyacrylic acid. Two discs of each group were put aside to the surface analysis and the others were bisected. One half received Ketac-Fil and the other received Fuji II LC. Specimens were prepared for SEM and were analyzed under different magnifications. **Results:** Er:YAG laser group showed no adhesive interface for both enamel and dentin, but strongly damaged the interface build-up for dentin/Fuji II LC. The application of laser irradiation followed by the polyacrylic acid exhibited gaps and irregularities for both substrates. **Conclusion:** Er:YAG laser irradiation combined or not with 40% polyacrylic acid produced a surface unfavorable for GIC interaction, especially for the resin-modified ones.

Introduction

The development of new dental outfits and alternative techniques for teeth restoration are regarded to be relevant to obtain a better bonding between dental structure and restorative materials [2, 7, 17]. Adhesion mechanisms of composite resin systems to dental surface by using acid etching are widely accepted and its efficiency is sufficiently proven [13, 18]. However, surface pretreatment with an acid or other technique prior to glass ionomer cement has not been included in the conventional procedure [4, 9, 22, 23].

Polyacrylic acid is being used before glass ionomer cement restorations to remove only the smear layer without either demineralizing the dentin or removing the plugs [1, 19, 21]. The calcium ions are kept available for chemical reaction with carboxyl ions of hydroxyapatite and allow the cement penetration into the hydrophilic substrate [19]. Indeed, polyacrylic acid improves surface wettability, a prerequisite to effective adhesion [3].

In the last years, alternative techniques to treat enamel and dentin surface have been proposed, aiming to improve the adhesion of restorative systems to dental substrate [6, 12, 14, 15]. Among them, the irradiation of dental surface with the Er:YAG laser at low parameters can be highlighted [2, 5, 20].

During irradiation, the energy is absorbed by water molecules of organic dentinal structures, thus causing heating and water vaporization. The resulting pressure within the irradiated tissue leads to the occurrence of successive microexplosions that causes the ejection of both organic and inorganic particles [5, 7, 11, 20]. This process successfully occurs because of the Er:YAG laser wavelength of $2.94\ \mu\text{m}$ that is coincident to the absorption pick of water and hydroxyapatite [7].

In dental structures, Er:YAG laser produces a micro-retentive pattern with cracks and fissures that helps the adhesion of resinous restorative materials [2, 8]. Some researchers reported the

exposure of dentinal tubules [15, 20] and appearance of "laser-modified layer" after laser treatment [20], but no alteration of calcium, phosphorus or oxygen contents were found [10].

Despite the effectiveness of Er:YAG laser for dental ablation [7], controversial results were verified considering the effects of lasing tooth structure before bonding restorative materials [2, 15, 23]. Moreover, the literature is scarce in studies that report the use of Er:YAG laser in dental surface before the application of glass ionomer cements. Therefore, this study assessed the morphological aspect of dental substrate/glass ionomer cement interface (using both conventional and resin-modified products) and also the dentin and enamel surfaces after Er:YAG laser pretreatment, combined or not with polyacrylic acid, through scanning electron microscopy.

Material and methods

Caries-free third molars, extracted for orthodontics reasons and stored into 0.1% thymol solution at 9°C were washed in running water for 24 h to eliminate thymol residues and were examined at $\times 10$ stereoscopic magnifying glass (Carl Zeiss-Jena), discarding those with structural defects. Thirty molars were selected and their crowns were sectioned perpendicular to the long axis, in parallel sections, obtaining dentin discs of approximately 1 mm thick.

Smear layer was standardized by water glass paper in decreasing grit size (#280, #320, #400 and #600) for 30 seconds each.

Discs were randomly assigned to two groups according to the glass ionomer cement used: conventional GIC (Ketac-fil, 3M/ESPE, Germany) and resin-modified GIC (Fuji II LC, GC Corporation Tokyo, Japan) (table I).

Table I - Details about the tested materials

Material	Surface treatment	Batch/Manufacturer	Composition
Ketac-fil conventional GIC	40% polyacrylic acid	67920	<i>Powder:</i> Calcium, aluminum fluorosilicate glass <i>Liquid:</i> polycarbonic acid, tartaric acid, water, benzoic acid
	Er:YAG laser	3M/ESPE	
	Er:YAG laser + 40% polyacrylic acid	Germany	
Fuji II LC Resin modified GIC	40% polyacrylic acid	0008011	<i>Powder:</i> aluminum silicate glass <i>Liquid:</i> polyacrylic acid, 3-hydrox- ethyl methacrylate, 2,2,4 trimethy hexamethy dicarbonate, triethylene, glycol dimethacrylate
	Er:YAG laser	GC Corporation	
	Er:YAG laser + 40% polyacrylic acid	Tokyo, Japan	

Following, the discs were subdivided into three subgroups according to the surface treatment procedure: 40% polyacrylic acid (Durelon liquid - 3M/ESPE), Er:YAG laser (Kavo Co., Biberach-Germany) and Er:YAG laser + polyacrylic acid.

The surfaces etched by polyacrylic acid were actively treated for 10 seconds. Next, the samples were washed in tap water and excess water was removed with absorbing paper, in order to obtain a moist surface.

For laser conditioning, Kavo Key Laser 2 (Kavo Co., Biberach-Germany) was used, comprising a high power output Er:YAG laser, with 2.94 μm wavelength, power output adjustable between 60 and 250 mJ, 1 to 5 Hz frequency, 250 to 500 μs pulse duration and 12 to 15 mm distance between the lens and target tissue. In this study, the laser beam was used in a defocused [8] non-contact mode at a 17 mm focal distance [11, 14], 80 mJ power output, 2 Hz frequency and under constant refrigeration (3 ml/min).

After the surface treatment, each disc was bisected: one hemi section received the glass ionomer cement (1 mm thick), while the other was prepared for SEM analysis.

Specimens were sectioned in a perpendicular direction to the tooth/restoration interface and sanded with decreasing grit sand paper (#280, #320, #400, #600 and #1200) and thus, they were polished with alumina and diamond solution on silk fabric.

The samples were washed and immersed into a glutaraldehyde solution (2.5%) in sodium cacodilate (0.1M) buffer with a 7.4 pH, for 12 hours at 4°C. After being fixed, the samples were cleaned by ultrasound (Ultrasonic Cleaner T-1449-D, Odontobrás Ind. e Com.) for ten minutes to remove any residues from the bisected surface. Sequentially, it was performed the surface etching with 37% phosphoric acid (3M, Scotchbond Etchant) for 10 seconds. The samples were cleaned in distilled water, dried and finally dehydrated in increasing ethanol series of 20, 50, 75, 95 and 100% for 20, 20, 20, 30 and 60 minutes, respectively, and then immersed in hexamethyldisilazane (HDMS) for 10 minutes. The specimens were metalized with a fine gold overlay, submitted to SEM and photographed at different magnifications, so that the surfaces and the adhesive/enamel and adhesive/dentin interfaces could be qualitatively analyzed.

Results

It was observed that the surface treatment with 40% polyacrylic acid produced micro-porosities in enamel and removal of the *smear layer* in dentin, but no complete removal of *smear plug* from dentinal tubules. The analysis of enamel/material interface revealed formation of an adhesive interface for Ketac-Fil (figure 1A) and an “intermediate layer” for Fuji II LC (figure 1C). The analysis of the dentin/GIC interface revealed formation of small gaps for Ketac-Fil (figure 1B) and good interaction for Fuji II LC (figure 1D).

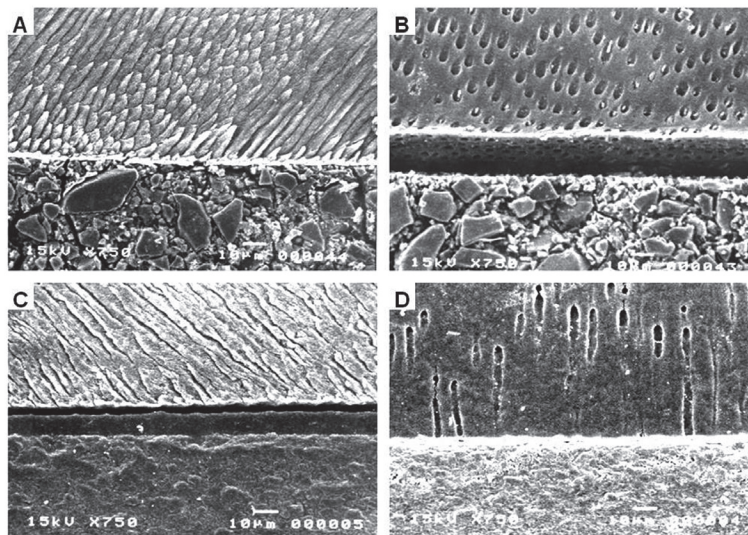


Figure 1 - Control group of 40% polyacrylic acid etching: A) Ketac-Fil/enamel interface (x 750) - Presence of good adhesive interface, B) Ketac-Fil/dentin interface (x 750) - Formation of small gaps, C) Fuji II LC /enamel interface (x 750) - Presence of “intermediate layer” and D) Fuji II LC/dentin interface (x 750) - Note the adhesive interface

Er:YAG laser irradiation on enamel resulted in formation of a chipped irregular surface, without smear layer, some melting areas were also observed (figure 2A). For dentin, the surface became even more irregular, with open dentinal tubules and absence of smear layer (figure 2B). Surface treatment

with Er:YAG laser combined with 40% polyacrylic acid produced for enamel an irregular and scaly surface (figure 2C), and for dentin a superficial aspect similar to the application of Er:YAG laser solely, with open dentinal tubules, absence of smear layer and fissures (figure 2D).

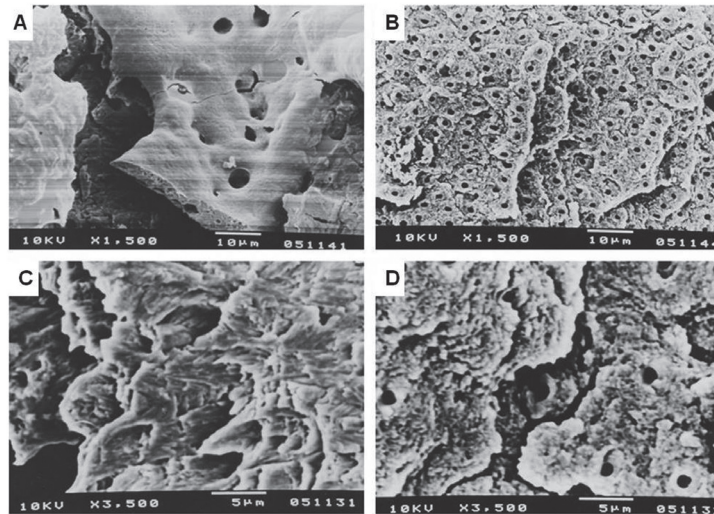


Figure 2 - A) Superficial morphologic aspect of enamel (x 1500) - Note the melting aspect of lased-surface and absence of smear layer, B) Superficial morphologic aspect of dentin (x 1500) - open dentinal tubules, absence of smear layer and irregularities, C) Enamel pretreated with Er:YAG laser + 40% polyacrylic acid (x 3500) - Note the irregular and scaly surface and D) Dentin pretreated with Er:YAG laser + 40% polyacrylic acid (x 3500) - open dentinal tubules, absence of smear layer and fissures

The combination Er:YAG laser with polyacrylic acid kept the adhesive interface similar to those that only received the laser application. In the interfaces analysis, it was verified a good interaction with only small gaps for enamel/Ketac-Fil pretreated with Er:YAG (figure 3A). For dentin/Ketac Fil, laser Er:YAG, combined or not with acid, produced and interface

with gaps without any adhesive bond (figure 3B). The interface for enamel/Fuji II LC, pretreated with Er:YAG laser and acid also had evident gaps in the most of the specimens (figure 3C). In dentin pretreated with laser or laser/acid and restored with Fuji II LC, craters up to 30 µm without adhesive bond were found (figure 3D).

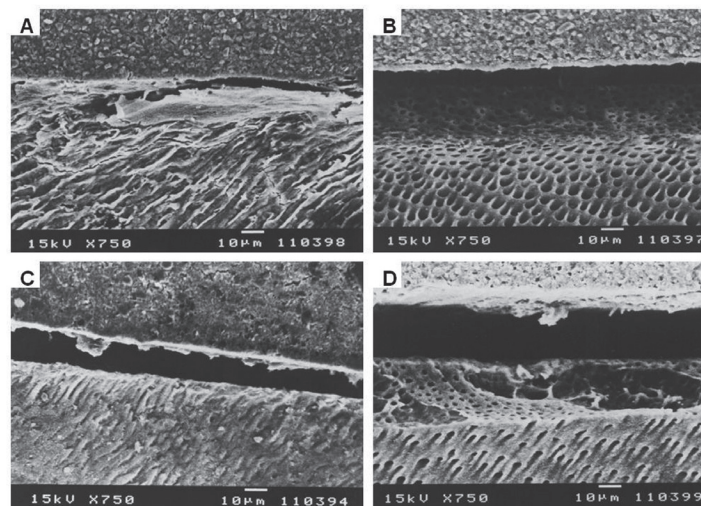


Figure 3 - A) Er:YAG laser irradiation on enamel restored with Ketac-Fil (x 750) - Good interaction with only small gaps, B) Er:YAG laser + polyacrylic acid irradiation on dentin restored with Ketac-Fil (x 750) - Gaps and absence of adhesive interface, C) Enamel/Fuji II LC interface pretreated with Er:YAG laser + polyacrylic acid (x 750) - Note the large gaps, D) Dentin/Fuji II LC interface pretreated with Er:YAG laser (x 750) - Note the craters up to 30 µm without any adhesive bond

Overall, the analysis of the morphological aspects revealed that Er:YAG laser irradiation had stronger influence in the formation of an adhesive interface for GIC restorative material, regardless of the subsequent application of polyacrylic acid.

Discussion

Glass ionomer cement bonds to tooth structure via direct chemical bonding without using any adhesive system [16, 24]. To improve the adhesion of this material, pretreating the dentin surface with a conditioner is recommended [9, 21].

In this study, it was verified that the superficial morphology of enamel and dentin irradiated by Er:YAG laser, whether combined or not with acid conditioning, was different from those achieved by conventional conditioning with polyacrylic acid. Er:YAG laser pretreatment resulted in another type of adhesive interface that impaired the ionomer cements, mainly in dentin substrate.

This outcome can possibly be explained by the stronger action of laser on dentin, rather than on enamel [11, 23]. Such difference of action on the substrates is because of a greater water content from dentinal tubules and a smaller rate of hydroxyapatite in dentin as compared to enamel [13, 18]. On the other hand, dentin is a less favorable substrate than enamel for adhesion due to its tubular structure, significant water content, occurrence of micro-structural alterations and greater rate of organic matrix [18].

It is known that the Er:YAG laser irradiates at the 2.94 μm wavelength, which is very near to water absorption wavelength, therefore a strong absorption of the radiated energy up to water vaporization occurs [2, 7]. A quick shock wave develops, causing expansion of the tissues followed by micro-explosions that eliminate micro-particles able to remove the dental tissue and leave behind a rough surface [2, 23]. So, the laser-irradiated substrate exposed an irregular surface, with micro-cracks, fissures and crater formation, absence of a smear layer and open tubules in dentin [20]. Other authors also found this morphological aspect on Er:YAG laser-irradiated dental [15, 20] and Ceballos *et al.* [5] reported denatured collagen fibers. All these morphological characteristics of the substrate may have impaired the bonding of ionomer cements, specially the resin-modified ones.

Conventional glass ionomer cement (Ketac-Fil) adheres to enamel and dentin by chelation of the polyacid carboxylic groups to hydroxyapatite calcium [19], so it is possible that this chemical reaction is not impaired by the superficial irregularities caused by laser irradiation as it does not modify the chemical composition of dental substrates [24].

Nevertheless, resin-modified glass ionomer cement (Fuji II LC) may have had its adhering mechanism hampered by laser irradiation, because its adhesion process is chemical as in conventional glass ionomer cement; however, it needs an additionally mechanical micro-retention [1] to a well-structured organic matter, so that the resinous components (HEMA) can penetrate the collagen fiber interstices to form the hybrid layer [24].

The combination Er:YAG laser with polyacrylic acid kept the adhesive interface similar to those that only received the laser application. Surface morphology is similar, with open dentinal tubules [23] and probably increasing dental substrate permeability, which could impair the adhesion of glass ionomer cement to dentin. A recent study concluded that surface treatment with phosphoric acid or Er,Cr:YSGG laser increased the bond strength of GIC to composite resin [17].

It should be noticed that that clinical applications of Er:YAG laser is rather distant from those of the laboratory experiments. Depending on the parameter settings used, a specific laser morphological pattern should be expected. Therefore, despite of the divergences as to the morphological appearance of dental surface after Er:YAG laser irradiation, there are important questions of safety, efficacy and substrate interaction with different types of restorative materials that should be analyzed before such technique can be considered as technically feasible.

Conclusion

Based on the microscopic observations and according to the tested parameters, it can be concluded that Er:YAG laser application, whether combined or not to 40% polyacrylic acid, produces an unfavorable surface for the interaction of glass ionomer cements, especially the resin-modified ones.

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