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Literature Review

**“Influence of acid conditioning on adhesion
to dentin prepared with Er,Cr:YSGG laser”**

*“Influência do condicionamento ácido na adesão, em superfícies de
dentina tratadas com laser Er,Cr:YSGG”*

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“Influence of acid conditioning on adhesion to dentin prepared with Er,Cr:YSGG laser” / “Influência do condicionamento ácido na adesão, em superfícies de dentina tratadas com laser Er,Cr:YSGG”

FIELD OF STUDIES – Operative Dentistry

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Índice

Acknowledgements	IV
Abstract	V
Resumo	VI
List of Units	VII
List of Abbreviations	VII
Tables Index	VIII
Figures Index	IX
Keywords	X
Introduction	1
Methodology	4
Results	6
Discussion	9
<i>Dentin Structure</i>	<i>9</i>
<i>Smear Layer</i>	<i>9</i>
<i>Erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser</i>	<i>10</i>
<i>Adhesive Systems</i>	<i>11</i>
• Etch-And-Rinse Adhesive	<i>12</i>
○ <i>Phosphoric acid use</i>	<i>12</i>
• Self-etch Adhesives	<i>14</i>
• Universal Adhesives	<i>14</i>
Conclusions	16
References	18
Anex I	22
<i>Declaração De Autoria Do Trabalho</i>	<i>22</i>
Anex II	24
<i>Parecer – Entrega Do Trabalho Final De Monografia</i>	<i>24</i>

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Nada mais posso dizer do que obrigada por tudo.
Do fundo do coração e sempre,
Obrigada.

Abstract

Introduction: The adoption of a less invasive, more conservative approach to operative dentistry has led to continuous research and development of laser tools as an alternative to rotatory handpieces. Thus, the erbium lasers have gained interest of clinicians. However its use still presents a number of unanswered questions.

Objectives: This review aims to gather and analyze information regarding the need for acid etching when using Er,Cr:YSGG lasers, and if it plays a significant role on resin-dentin adhesion.

Methodology: Electronic research was performed using PubMed Central, Science Direct, Scopus, Scielo and ISI Web of Knowledge as databases. The inclusion criteria were papers published in the last 10 years, that could be found and accessed as Full Text documents, in English, Portuguese or Spanish, conducted in healthy human dentin and therefore not contemplating studies in animals or deciduous teeth. A total of 70 articles was found. A selection by title and abstract was performed to evaluate the articles suitability. After concluding the research, duplicate papers were excluded. In cases where the abstract alone wasn't clear, a full text revision was conducted and other exclusion criteria were applied reaching a total final of 8 articles .

Results and Discussion: From the analysis and comparison of the selected articles the best achieved results of bond strength were (± 41 MPa), from the use of 40% phosphoric acid for 30 seconds as conditioning pre-treatment plus a self-etch adhesive on dentin prepared with the Er,Cr:YSGG laser. The second best result comes at (± 39 MPa) from the use of 40% phosphoric acid and additional 10% sodium hypochlorite gel conditioning. Furthermore, within the articles, a diversity of results was observed, which may be due to the different parameters of the laser devices used in each article, as well as the different combinations of materials used, namely different bonding systems, in association (or not) with pre-treatment using phosphoric acid alone or with sodium hypochlorite, different etching times, and different restorative materials.

Conclusion: Phosphoric acid does play a significant role on resin-dentin adhesion on teeth prepared with Er,Cr:YSGG laser, as the best results of bond strength were achieved with its use (SBS of ± 41 MPa).

Resumo

Introdução: A adoção de uma abordagem menos invasiva em dentisteria operatória tem levado ao desenvolvimento de aparelhos de laser como uma alternativa a instrumentos de alta rotação. Assim, o laser Er,Cr:YSGG tem despertado cada vez mais o interesse dos médicos dentistas. Porém ainda apresenta uma variedade de questões relativamente ao seu uso.

Objetivos: Esta revisão de literatura tem como objetivo, analisar a necessidade do uso de condicionamento ácido em dentina tratada com laser Er,Cr:YSGG, e o seu papel na força de adesão.

Materiais e métodos: Para efetuar a pesquisa para esta revisão, as bases de dados usadas foram PubMed Central, Science Direct, Scopus, Scielo e ISI Web of Knowledge. Foram selecionados estudos publicados nos últimos 10 anos, em Português, Inglês ou Espanhol, com acesso a Full-Text, conduzidos em dentina saudável de dentes permanentes, excluindo assim dentes cariados, tratados com outros lasers, dentes decíduos ou de animais. Um total de 70 artigos foi encontrado. Uma seleção por título e resumo foi realizada para avaliar a sua relevância. Os artigos duplicados foram eliminados, nos casos em que o resumo não era claro, uma leitura completa do artigo foi realizada. A aplicação dos critérios de exclusão levou a um total final de 8 artigos.

Resultados e Discussão: Da análise e comparação dos artigos selecionados, os melhores resultados de força de adesão obtidos foram (± 41 MPa), com a utilização de ácido fosfórico a 40% durante 30 segundos e um adesivo self-etch, em dentina preparada com o laser Er,Cr:YSGG. O segundo melhor resultado foi de (± 39 MPa), ao utilizar ácido fosfórico a 40% e hipoclorito de sódio a 10% como condicionamento. A análise dos artigos demonstrou uma grande variedade de resultados, o que pode ser atribuído ao fato de terem sido utilizados diferentes parâmetros de laser, diferentes combinações de materiais, nomeadamente, diferentes sistemas adesivos com ou sem pré-tratamento, aplicação apenas de ácido fosfórico ou associado a hipoclorito de sódio, diferentes tempos de condicionamento do ácido na dentina e diferentes materiais restauradores.

Conclusão: O ácido fosfórico tem sim um papel importante na adesão entre dentina e resinas, em dentes preparados com o laser Er,Cr:YSGG, como pode ser observado pelo melhor resultado de força de adesão obtido (± 41 MPa).

List of Units

μm	Micrometer (10^{-6} meters)
J/cm^2	Joule per square centimeter
$^{\circ}\text{C}$	Degree Celsius
μs	Microsecond (10^{-6} seconds)
Hz	Hertz (pulses per second)
W	Watt
s	Second
MPa	Megapascal
nm	Nanometer (10^{-9} meters)

List of Abbreviations

Er,Cr:YSGG	Erbium, chromium: yttrium-scandium-gallium-garnet
Er:YAG	Erbium: Yttrium-Aluminum Garnet
Laser	Light amplification by stimulated emission of radiation
TBS	Tensile bond strength
μTBS	Microtensile bond strength
SBS	Shear bond strength
SE	Self-etch

Tables Index

Table I – Inclusion and exclusion criteria of research.....	4
Table II - Comparison of bond strength test results of the indicated articles, on dentin prepared with Er,Cr:YSGG laser (and respective parameters) , with and without prior phosphoric acid conditioning.....	8

Figures Index

Figure 1 – Flowchart of research and selection process.....5

Keywords

Erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser, Dentin, Bond strength, Adhesion, Etching, Phosphoric acid

Introduction

Caries are the main cause of toothache and tooth loss, so its removal is considered the primary goal in restorative dentistry (1).

High-speed rotatory drills have been used as the “gold standard” for removing diseased dental hard tissue and preparing cavities, as they are cost effective and less time consuming when compared to existing new technology, namely lasers. However, studies show that conventional appliances present some limitations, namely: pulpal temperature rise, tissue cracking, unnecessary removal of healthy surrounding tissue during the preparation, risk of cross contamination as well as future tooth sensitivity (2, 3). Not to mention that the vibration and noise caused by the drills/handpieces, together with the need for local anesthesia in most dental procedures lead to discomfort and stress in patients (4).

The adoption of a less invasive, more conservative approach to operative dentistry has led to continuous research and development of laser tools as an alternative to rotatory handpieces (5). The use of lasers for enamel and dentin conditioning and cavity preparation has shown successful results. Therefore, a variety of lasers are currently available, however, the best performance in this area has been seen when using the Erbium laser family. Both Erbium: Yttrium-Aluminum Garnet (Er:YAG) and Erbium Chromium: Yttrium-Scandium-Gallium-Garnet (Er,Cr:YSGG) lasers with respective wavelengths of 2.94 μm and 2.78 μm , produce enamel and dentin surfaces with no smear layer, open dentinal tubules and more prominent peritubular dentin than intertubular dentin (6).

Although both Erbium lasers have been described as appropriate, the Er:YAG laser has had more extensive research conducted and therefore, more papers can be found to help us understand its use (7). This reason conducted my focus to be on the Er,Cr:YSGG laser only, specifically the influence of acid conditioning on resin-dentin bond strength in teeth prepared with this laser, as in addition to not presenting the limitations of high-speed rotatory instruments already mentioned Studies have shown that when used within the correct parameters and with constant water irrigation, these lasers produce a “rough” microretentive morphological pattern, similar to the effect of acid etch techniques and therefore, possibly favorable for bonding procedures (3, 8).

Dentin is a complex mineralized tissue consisting of 70% of its bulk in mineral, 20% organic component and 10% fluid. Collagen type I makes for 90% of the organic matrix, while the remaining 10% consists of noncollagenous proteins like phosphoproteins and proteoglycans (9). Its intricate tree-dimensional structure, with tubules extending from the pulp to the dentino-enamel junction, intratubular and peritubular dentin makes it that only a few structure-property relationships can be performed, therefore making its interaction with other material very limited (10, 11). Dental materials adhesion to dentin has been studied over the years in attempts to understand and improve them. First attempts to condition dentin had poor results due to preferential conditioning of peritubular dentin. Composite resins, being hydrophobic, do not allow their penetration in peritubular walls because of its inability to move the dentinal fluid, resulting in polymerization shrinkage and dentinal tags away from the walls (11). The first generations of dental adhesives came into existence in the 1950s. In early 1980s, second generation adhesives were developed, with improved surface wetting but still low shear bond strength. Third generation, still in the 1980s used acids to remove the smear layer on prepared teeth. Fourth generation (early 1990s) were based on the etch-and-rinse technique involving conditioning, priming and bonding. Fifth generation bonding agents combined the primer and bond in a single bottle, but required prior enamel and dentin conditioning. The sixth generation ones (2000s), marked the elimination of acid etching and therefore reducing post-operative sensitivity. The seventh generation, introduced in 2002/2003 as “all-in-one” adhesives have the advantage of an easier application procedure (12, 13). Currently, a mix of these adhesive systems are used, being the most popular: etch-and-rinse adhesives, which remove the smear layer and superficial hydroxyapatite through separate etching; self-etch adhesives which makes the smear layer permeable without removing it and self-adhesives which have inherent bonding capacity (14, 15). Phosphoric acid etching on dentin, removes the smear layer and smear plugs, demineralizes the inter-tubular dentin leaving a tridimensional microporous mesh of collagen fibrils exposed and open dentin tubules, theoretically optimal for resin tags penetration (16). As previously mentioned, dentin surfaces prepared with Er,Cr:YSGG lasers show similar surfaces to acid etched ones, which has encouraged clinicians to use them as an alternative to chemical etching (17). With this being said, there is no consensus

on this front. Some studies defend that the application of phosphoric acid to erbium laser-irradiated dentin can have advantages, such as decrease marginal leakage and enhanced bond strength, while others show significantly lower bond strength values for Erbium laser-treated surfaces than for acid-etched dentin (18). This review aims to gather and analyze information regarding the need for acid etching when using Er,Cr:YSGG lasers, and if it plays a significant role on resin-dentin adhesion.

Methodology

Electronic research was performed through Porto University's virtual private network (VPN), using PubMed Central, Science Direct, Scopus, Scielo and ISI Web of Knowledge as databases. The search keywords were *Erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser, Dentin, Bond strength, Adhesion, Etching and Phosphoric acid*.

The inclusion criteria [Table I] were papers published in the last 10 years, that could be found and accessed as Full Text documents, in English, Portuguese or Spanish, conducted in healthy human dentin and therefore not contemplating studies in animals or deciduous teeth. A total of 70 articles was found. A selection by title and abstract was performed to evaluate the articles suitability. After concluding the research, duplicate papers were excluded. In cases where the abstract alone wasn't clear, a full text revision was conducted and other exclusion criteria were applied [Table I] reaching a total final of 8 articles [Figure 1].

Inclusion Criteria	Exclusion Criteria
Human healthy teeth	Animal teeth
Full Text	Deciduous teeth
English, Portuguese, Spanish	Carious teeth
2010 - 2020	Duplicate articles
Preparations in dentin with the Er,Cr:YSGG laser	Papers without Full-Text access
Papers with <i>in vitro</i> adhesion tests on dentin	Tests on dentin treated with another laser beyond the Er,Cr:YSGG laser
<i>In vitro</i> studies with and without acid conditioning	Papers that do not offer adhesion tests

Table I – Inclusion and exclusion criteria of research.

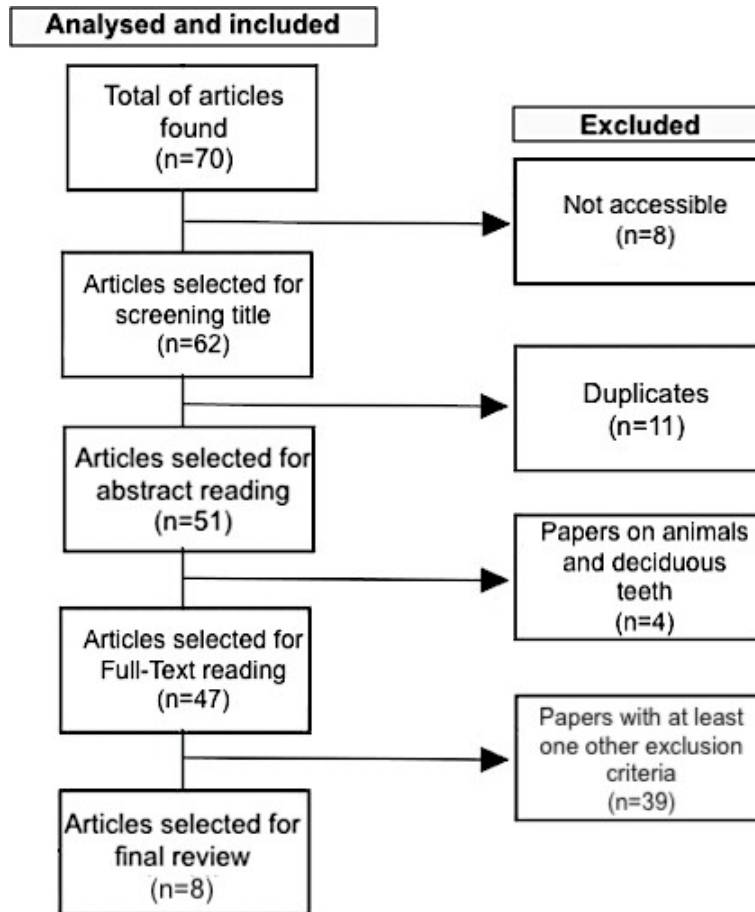


Figure 1 – Flowchart of research and selection process.

Results

Studies have been conducted in the last few years to try to understand the effect of different adhesive systems and pre-treatments have on dentin-resin bond strength, when Er,Cr:YSGG laser is used for cavity preparations. The following is a brief summary of the studies found during this research, and their results.

Obeidi *et al.* (19) in 2010 tested different etching times (15 s and 30 s) with a 37% phosphoric acid (Patterson Brand LOT # 060278). Rinsed for 30 and 60 seconds respectively. The used adhesive was (Adper Single bond plus, 3 M-ESPE, MN, USA LOT # 7LE). An Er,Cr:YSGG laser at 2,780 nm, 20 Hz and 140 μ s pulse was used with parameters of 3 W, 60% air, 70% water. Best SBS results were: (13.62 \pm 7.28 MPa) for conditioning for 15 s.

Ferreira *et al.* (18) in 2010, also tested different acid etching times (15 s, 30 s and 60 s) but with a 35% phosphoric acid (3M ESPE, São Paulo, SP, Brazil) followed by rinsing for respectively 15 s, 30 s, 60 s. The adhesive (Single Bond, 3M ESPE, batch number OEH) was applied, followed by the composite resin (Z250, 3M ESPE, batch number 1KK). An Er,Cr:YSGG laser at 2.78 μ m, 20 Hz and 140 μ s pulse was used with parameters of 4 W, 75% water, 65% air. Best SBS results were: (12.38 \pm 2.47 MPa) for conditioning for 15 s.

Carvalho *et al.* (20) in 2011 tested two adhesives (Clearfil SE Bond and Single Bond Plus) with and without a combination of 37% phosphoric acid and 10% sodium hypochlorite solution (both for 60 s plus rinsing). An Er,Cr:YSGG laser at 2.78 μ m, 20 Hz was used with parameters of 3.5 W, 65% air, 55% water. Best SBS results were: (21.9 \pm 4.7 MPa) for conditioning with laser only.

Beer *et al.* (21) in 2012, tested the adhesives Excite (Ivoclar Vivadent, Schaan, Liechtenstein) – a one-bottle universal dentin adhesive, Scotchbond Multipurpose (3 M ESPE, St. Paul, MN) – a two-bottle system (primer/bonding) and Syntac Classic (Ivoclar Vivadent, Schaan, Liechtenstein) – a three-bottle system (primer/bonding/adhesive). With and without acid etching with 37% phosphoric acid (3 M, St. Paul, MN) for 15 s plus rinsing for 30 s. Being restored

with the resin (Z100 MP Restorative A3; 3 M ESPE, St. Paul, MN). The Er,Cr:YSGG laser settings were 2 W, 55% water, 65% air. Best SBS results were: (mean 14.07 MPa) for conditioning without etching.

Ramos *et al.* (8) in 2014 tested an etch-and-rinse adhesive system (Single Bond, 3M ESPE; St. Paul, MN, USA) with 37% phosphoric acid etching and a self-etching adhesive system (Clearfil SE Bond; Kuraray Medical, Inc., Osaka, Japan). An Er,Cr:YSGG laser at 2.78 μm , 30 Hz and 140 μs was used with parameters of 1.5 W, 65% air, 70% water. The resin composite used was shade A3, Filtek Z250, 3M ESPE, Dental Products; St. Paul, MN, USA). Best μTBS results were: (38.7 ± 16.5 MPa) for Clearfil SE Bond, when compared to the etch-and-rinse group at (33.4 ± 6.1 MPa)

Takada *et al.* (22) in 2015, tested a two-step self-etching system (Clearfil SE Bond, Kuraray Noritake Dental, Osaka, Japan) and a one-step self-etching system (Clearfil Tri-S Bond, Kuraray Noritake Dental). A 40% phosphoric acid solution (K-etchant Gel, Kuraray Noritake Dental) and a 10% sodium hypochlorite gel (AD Gel, Kuraray Noritake Dental) were also used as pre-treatment agents in some samples. The resins used were: a flowable composite resin (Clearfil Majesty LV, Kuraray Noritake Dental) and a universal composite resin (Clearfil Majesty, Kuraray Noritake Dental). The Er,Cr:YSGG laser parameters used were 2.0 W (75% of water spray and 60% air spray), 140 μs , 20 Hz. Best μTBS results were: (about 41 MPa) for using laser plus phosphoric acid etching at 40% for 30 s, and applying the adhesive Clearfil SE bond.

Jhingan *et al.* (12) in 2015 compared Clearfil SE Prime and Bond (Kuraray Noritake; Tokyo, Japan) with and without prior acid etching (K-etchant gel, 40% phosphoric acid; Kuraray Noritake) and Clearfil S3 Bond (Kuraray Noritake) with prior acid etching. The Er,Cr:YSGG laser was used at 6 W, 15 Hz, 80% air, 50% water. Best SBS results were: (33.46 ± 9.19 MPa) for laser with no acid etching and Clearfil SE Prime and Bond.

Vohra *et al.* (3) in 2018 tested the etch-and-rinse adhesive Harvard Bond TE Mono (Harvard Dental International, GmbH, Hoppegarten, Germany) 30 s, and

SE bond (Harvard Bond SE Mono) 30 s, using Multi Core Flow (MC) (Ivoclar Vivadent Schaan Liechtenstein) resin composite for restoration. The Er,Cr:YSGG at 4.5 W, 50 Hz. Best SBS results: (23.66 ± 2.56 MPa) for laser plus etch-and-rinse, compared to SE (11.87 ± 1.21 MPa).

*DS – Does not specify

Author	Conditioning	Etch time (s)	SBS/TBS (MPa)	[No conditioning] SBS/TBS (MPa)	Power (W)	Pulse duration (µs)	Frequency (Hz)	Water (%)	Air (%)
Obeidi <i>et al.</i> ⁽¹⁹⁾	37% phosphoric acid	30	13.15	-	3	140	20	70	60
		15	13.62						
Ferreira <i>et al.</i> ⁽¹⁸⁾	35% phosphoric acid	60	8.80	-	4	140	20	75	65
		30	10.69						
		15	12.38						
Carvalho <i>et al.</i> ⁽²⁰⁾	37% phosphoric acid + 10% sodium hypochlorite	60 each	21.3	21.9	3.5	DS	20	55	65
Beer <i>et al.</i> ⁽²¹⁾	37% phosphoric acid	15	6.28	14.04	2	DS	20	55	65
Ramos <i>et al.</i> ⁽⁰⁸⁾	37% phosphoric acid	15	33.4	38.7	2	140	20	75	60
Takada <i>et al.</i> ⁽²²⁾	40% phosphoric acid	30	±41	±22	2	140	20	75	60
		30	±39						
		90							
Jhingan <i>et al.</i> ⁽¹²⁾	40% phosphoric acid	30	24.69	33.46	6	DS	15	50	80
Vohra <i>et al.</i> ⁽⁰³⁾	Phosphoric acid	DS*	23.66	11.87	4.5	DS	50	DS	DS

Table II - Comparison of bond strength test results of the indicated articles, on dentin prepared with Er,Cr:YSGG laser (and respective parameters) , with and without prior phosphoric acid conditioning

As can be seen from the comparative table above [table II], the best achieved results of bond strength were seen on Takada *et al.*(22) - (±41 MPa), from the use of 40% phosphoric acid for 30 seconds as conditioning pre-treatment, when prepared with the Er,Cr:YSGG laser of 2.0 W (75% of water spray and 60% air spray), 140 µs, 20 Hz, and with the self-etch adhesive (Clearfil SE Bond, Kuraray Noritake Dental, Osaka, Japan). The second best result comes at (±39 MPa), also from Takada *et al.*(22) with the use of 40% phosphoric acid and additional 10% sodium hypochlorite gel conditioning and the same laser parameters.

The best results from Er,Cr:YSGG laser conditioning alone, with no phosphoric acid pre-treatment were of (38.7 MPa), by using the self-etching adhesive system (Clearfil SE Bond; Kuraray Medical, Inc., Osaka, Japan), with laser settings of 1.5 W ,70% of water spray and 65% air spray, 140 µs of pulse duration and 30 Hz of frequency.

Discussion

Dentin Structure

Dentin's microstructure and properties are the principal determinants of nearly all procedures in operative dentistry. Dentin is described to be a complex hydrated biologic structure (15). It is arranged in an intricate three-dimensional frame made of tubules that extend from the pulp to the dentino-enamel junction, intratubular and peritubular dentin (9). The peritubular dentin, is a hypermineralized dentin sheath that lines the tubules, whereas the intertubular dentin consists of a less mineralized area between dentinal tubules. Intertubular dentin contains collagen fibrils with characteristic banding and is available for adhesion (14).

In terms of composition, it consists of 70% mineral components, 20% organic and 10% fluid. Fibrillar type I collagen makes 90% of the organic matrix, while the remaining 10% consist of noncollagenous proteins such as phosphoproteins and proteoglycans (9). Its complex structure makes it so that available structure-material interactions are limited. Moreover, dentin gets modified over time by aging, physiological and disease/carious processes to create different forms of itself, which makes adhesive procedures more complex when compared to enamel (16, 23).

Smear Layer

Cavity preparation using rotatory drills create a smear layer which is loosely attached to the tooth surface. This smear layer is composed of organic and inorganic components from enamel, dentin, or cementum, which in clinical conditions is usually contaminated by saliva, blood, and bacteria (16). Some of these components are hydroxyapatite and altered collagen, with an external surface formed by gel-like denatured collagen. This altered collagen may acquire a gelatinized consistency, due to the friction and heat created by the preparation procedure. The morphology of the smear layer is ultimately determined by the type of instrument used during the preparation and by the dentin site where it is formed (24). The smear layer covers the dentin surface and blocks the dentinal tubules by forming dentinal plugs and should be removed before chemical bonding is attempted (24, 25). To allow resin infiltration and adhesion to dentin, it has to be

conditioned with acid to remove the smear layer, open the dentinal tubules and decalcify the underlying dentin (26).

Erbium, chromium: yttrium-scandium-gallium-garnet (Er,Cr:YSGG) laser

The introduction of laser technology in dentistry offers a new alternative to conventional treatments using high speed rotatory instruments. Laser treatments involve minimal loss of dental tissue, no vibration or heat, reducing the need for local anesthesia and increasing the patients comfort (8).

The Er,Cr:YSGG laser has been considered one of the most efficient and safe hard tissue laser systems for dental hard tissue procedures, including cavity preparation and laser conditioning. Using a pulse-beam system, fiber delivery and a sapphire tip bathed in a mixture of air and water vapor, it has shown success in soft tissue surgery as well as cutting enamel, dentin and bone (2). At a wavelength of 2.78 μm , it presents superior cutting ability of that of the Er:YAG laser and its water/air ratio can be manipulated according to the cutting conditions (22). The ablation mechanism promoted by Er,Cr:YSGG laser consists of micro-explosions that eject hard-tissue particles from the irradiated dentinal surfaces. The laser's energy is absorbed by the dental tissues, vaporizing the water and hydrated components. The dentin ablation threshold is 2.69-3.66 J/cm^2 .

At energies below their ablation threshold, they have also been used for reducing hypersensitivity and reducing demineralization and susceptibility to secondary caries (27). Several factors influence the effect of this laser irradiation on the tooth surface, as the fluence applied on the surface, focal distance, beam spot size, repetition rate, structural properties of the target tissue, amount of water during irradiation, and pulse duration (28). Moretto *et al.* (29), tested four different power settings of the Er,Cr:YSGG laser at 2.78 μm , 20 Hz (with 65% air and 55% water) laser: 2 W/20 Hz; 2.5 W/20 Hz; 3 W/20 Hz and 4 W/20 Hz with a two-step etch-and-rinse adhesive. The best μTBS results were: (17.56 ± 2.07) for the (2 W/20 Hz) setting plus etch-and-rinse adhesive, compared to (17.54 ± 3.01) , (14.97 ± 2.86) and (16.16 ± 4.96) for the other settings respectively. These results go along with the idea that the laser device settings affect the influence it has on the tooth surface, in this case, bond strength of an adhesive to dentin.

The results of laser preparations are open dentinal tubules, a rough surface, due to the removal of intertubular dentin, and no smear layer (11, 20). However, the potential impact of the laser on the collagen network is still not fully understood. Its thermomechanical effects may cause a superficial denaturation of collagen fibrils on dentin. Collagen denaturation results in the formation of an acid-resistant surface with granular structures that is either carbonized or covered by dentin melting products (30). Being that bonding to dentin surface relies directly on the infiltration of hydrophilic monomers to the exposed dentin collagen webs, its success depends on the exposition and integrity of the lather (31, 32), therefore, this matter requires further studying.

Adhesive Systems

Adhesive systems are used to improve the marginal seal of a composite resin restoration at the interface between enamel and dentin. Such bonding systems require the application of acid conditioners (etch-and-rinse or self-etch adhesives) to promote the superficial demineralization of enamel and dentin (33). Currently, it is believed that the effectiveness of dentin adhesives depends on the infiltration of monomers with high hydrophilic affinity into the network of collagen fibers in the etched dentin structure. This entanglement of monomers with collagen fibrils and scarce residual hydroxyapatite crystals forms a hybrid tissue known as resin-dentin interdiffusion zone or hybrid layer (34). A concern regarding the efficacy of the hybrid layer as a bonding mechanism for adhesive resin to dentin is that the resin monomers could not reach the bottom of the demineralized zone. Strong acids such as phosphoric acid create demineralization deeper than the diffusion capacity of the resin monomers thus leaving collagen fibers in the deep tissue layers unprotected (21). The bond strength at dentin-resin interface depends on the hybrid layer thickness, resin tag length, adhesive lateral branch formation, surface roughness, interface integrity, and void formation (35). Surface characteristics of tooth structure resulting from different preparations and protocols may affect bonding effectiveness, but it also may depend on the type of adhesive system used.

- Etch-And-Rinse Adhesive

Enamel and dentin are treated with phosphoric acid to remove the smear layer and demineralize the superficial dentin layer. Phosphoric acid concentration varies from 30% to 50% (pH = 0.1 – 0.4). The most used one, being at 37%, as it produces more consistent conditioning patterns, without negative effects on the pulp. For these total etching adhesive systems, hybrid layer formation relies on the demineralization of superficial dentin by inorganic acids, which exposes collagen fibrils that are then infiltrated by hydrophilic monomers. (24, 36). They can be applied in a three-step system, where the acid etching and rinsing is followed by the application of a primer and lastly a hydrophobic resin. For the two step simplified version, the primer and resin are combined in a single bottle (16).

- *Phosphoric acid use*

Phosphoric acid presents a few disadvantage, namely being technique sensitive and possible irreversible demineralization for overuse (37). Along with the fact that it makes for a discrepancy between dentin demineralization and resin penetration, as it leaves “spaces” deeper than the resin can reach (34).

Both Obeidi *et al.* (19) and Ferreira *et al.* (18) tested different etching times on laser treated dentin, having the first one used 37% phosphoric acid and the latter, 35% phosphoric acid. Both had best results with smaller etching times (15 s), concluding that an increased etching time is not able to modify the strength of the bond of the adhesive to irradiated dentin.

Laser manufacturers commonly argue that at low-energy settings, enamel and dentin can be conditioned with the laser instead of conventional phosphoric acid etching, as pretreatment for adhesive procedures (38). Jaber *et al.* (39) tested the effect of Er,Cr:YSGG laser irradiation in both enamel and dentin using one hundred and seventy five teeth divided into enamel and dentinal groups. The dentinal groups included two subgroups, a bur-cut and laser-etched one and a bur-cut and acid-etched. They were bonded with (Single Bond, 3M ESPE Dental Products, USA) and restored with Z100 composite (3 M, Dental Product, USA).

The Er,Cr:YSGG laser that was used was the Waterlase from Biolase technologies (San Clemente, CA, USA), which operates at a wavelength of 2.78 μm and has a pulse duration of 140 μs with a repetition rate of 20 Hz. The power settings were 1 W (55% water and 65% air spray). Within the dentinal groups, this study demonstrated that the bond strength is significantly weaker when tooth surfaces are prepared with the Er,Cr:YSGG laser compared to a diamond bur, concluding that re-etching with phosphoric acid would be recommended if an Er,Cr:YSGG laser is used for tooth preparation or surface treatment.

Also, the association of phosphoric acid conditioning with sodium hypochlorite has been discussed. Carvalho *et al.* (20) and Takada *et al.* (22) used 10% sodium hypochlorite after etching with phosphoric acid in hope to achieve a better bonding outcome. Carvalho *et al.* (20) states that the reason behind it, is that it would remove the denatured organic matrix, which is fused with hydroxyapatite. In order to established bonding with only the mineral content of dentin, complete deproteinization of acid-etched dentin has been indicated in adhesive procedures. Collagen depletion may increase bond stability by removing the weakest component of the bonded interface that can be hydrolyzed by bacterial or endogenous enzymatic factors. While in their study, results didn't have an expressive difference, in Takada *et al.* (22) this association of pre-treatments made for the second best bonding strength within all the compared papers.

Beer *et al.* (21), Ramos *et al.* (8), Jhingan *et al.* (12) and Vohra *et al.* (03) also compared the bond strength of preparations with and without phosphoric acid conditioning, having had different results. While the first three authors, had best result without acid etched dentin, Vohra *et al.* (03) did not.

The explanation might be that laser treated dentin already does not have smear layer, and being that phosphoric acid's main function is to remove said layer and partially demineralize the dentin to allow adhesive penetration into the collagen fibrils, the etching time of 15 seconds of the used acids (recommended by the manufacturer) might be longer than necessary to demineralize the already smear layer-free dentin. Therefore the over-etching might have interfered with the hybrid layer formation and consequently the successful infiltration of the monomers across the depth of demineralized surface (8), causing weaker bonds.

- Self-etch Adhesives

These adhesives don't require a separate etching step because they contain acidic monomers that act as both an etchant and primer, preparing the tooth surface for bonding (40). Contrary to the etch-and-rinse systems, in this case the smear layer isn't completely removed, which may prevent post-operative sensitivity (16). The acids are weak so instead of removing it, they modify the smear layer. The modified products are integrated into the adhesive compound, therefore inhibiting the formation of tags. The absence of a smear layer after Er,Cr:YSGG laser preparation avoids this zone of altered material. Conditioning agents such as weak acids remove the smear layer but do not demineralize the inorganic matrix of the tooth (21). This system also relies on the fact that demineralization and infiltration take place simultaneously and to the same path, which theoretically should mean a complete penetration of the adhesive, leaving no sub-infiltrated demineralized areas (16). Therefore, these systems would theoretically be the most appropriate to use in laser-prepared dentin, and should obtain the best adhesion results (11), as their weak acidic agents wouldn't compromise the dentin surface with over-etching and consequently formation of weaker bonds of dentin-resin. From the articles compared on [Table II], the best bond strength resulted from the combination of phosphoric acid with the self-etch adhesive (Clearfil SE Bond, Kuraray Noritake Dental, Osaka, Japan) on dentin treated with Er,Cr:YSGG laser in the study conducted by Takada *et al.* (22)

- Universal Adhesives

A new generation of adhesives, designed under the "all in one" concept of the already existing one step self-etch adhesives, but also incorporating the versatility of being adaptable to the clinical situation (36). This means that the professional can decide which adhesive strategy to use: etch-and-rinse or self-etch. Existing literature shows that the best performance of this system is seen when combined with prior acid etching (on enamel, as the same wasn't achieved with dentin) (16). Adebayo *et al.* (5) tested one two-step self-etching primer adhesive (Clearfil SE Bond – CSE, Kuraray Medical, Okayama, Japan), two "all-

in-one” adhesive systems (Xeno IV – XE, Denstply Caulk, Milford, DE, USA; and Tokuyama Bond Force – TK, Tokuyama Dental Corp, Tokyo, Japan), and the Filtek Silorane Adhesive System (FS, 3M ESPE, St Paul, MN, USA)., prepared with the Er,Cr:YSGG laser at 2,780 nm, 20 Hz and 140 μ s pulse was used with parameters of 3,5 W, 65% air, 60% water was used. The best and worst SBS results were (10.4 ± 2.85 MPa) - (Xeno IV – XE, Denstply Caulk, Milford, DE, USA); and (Tokuyama Bond Force – TK, Tokuyama Dental Corp, Tokyo, Japan) at (6.78 ± 2.12 MPa) respectively. More studies should be conducted to best analyze these adhesive’s performance when applied after laser irradiation.

Conclusions

This dissertation shows a variety of results when regarding the influence phosphoric acid has on adhesion to dentin prepared with the Er,Cr:YSGG laser. This diversity of results may be due to different factors, including the different parameters of the laser devices used in each article, as well as the different combinations of materials used, namely different bonding systems, in association (or not) with pre-treatment (phosphoric acid and sodium hypochlorite), different etching times, and different restorative materials. With that being said, self-etch adhesive systems should theoretically be the most appropriate to use in laser-prepared dentin, and should obtain the best adhesion results due to their weak acidic components. As such, the best results of bond strength to dentin from the compared articles were achieved by Takada *et al.* (22) with a combination of these adhesive systems with prior application of phosphoric acid as follows: SBS of (± 41 MPa), from the use of 40% phosphoric acid for 30 seconds as conditioning pre-treatment, when prepared with the Er,Cr:YSGG laser of 2.0 W (75% of water spray and 60% air spray), 140 μ s, 20 Hz, and with the self-etch adhesive (Clearfil SE Bond, Kuraray Noritake Dental, Osaka, Japan). The best results from Er,Cr:YSGG laser conditioning alone, with no phosphoric acid pre-treatment were of (38.7 MPa), achieved using the self-etching adhesive system (Clearfil SE Bond; Kuraray Medical, Inc., Osaka, Japan), with laser settings of 1.5 W, 70% of water spray and 65% air spray, 140 μ s of pulse duration and 30 Hz of frequency. From these results, we can say that phosphoric acid application does play a role in adhesion to dentin treated with Er,Cr:YSGG lasers, as the best results achieved were with its use.

As laser technology is still considered new, high-speed rotatory drills are still seen as the “gold standard” for treatment in operative dentistry. With that being said, not many articles can be found that focus solely on laser use and interaction with different materials, making it hard to reach any definite conclusions. The industry is still primed to produce and develop dental materials to better work on teeth prepared with rotatory handpieces, making it so that clinicians have to adapt their practice and do trials and errors when using them with lasers. Therefore, more products focused on the specific characteristics of laser use should be developed

for better, safer results, as there are no doubts these laser devices will continue to grow in popularity for their wide range of possible applications and benefits.

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Anex I

Declaração De Autoria Do Trabalho

DECLARAÇÃO DE AUTORIA DO TRABALHO

Declaro que o presente trabalho, intitulado: *“Influence of acid conditioning on adhesion to dentin prepared with Er,Cr:YSGG laser” / “Influência do condicionamento ácido na adesão, em superfícies de dentina tratadas com laser Er,Cr:YSGG”*, no âmbito da Monografia de Investigação/Relatório de Atividade Clínica, integrada no Mestrado Integrado em Medicina Dentária da Faculdade de Medicina Dentária da Universidade do Porto, é da minha autoria e todas as fontes foram devidamente referenciadas.

Porto, 22 de Maio de 2020

A estudante,

Suely Lopes Varela

(Suely Lopes Varela)

Anex II

Parecer – Entrega Do Trabalho Final De Monografia



PARECER

Entrega do trabalho final de Monografia

Informo que o trabalho de Monografia desenvolvido pela estudante Suely Lopes Varela, com o título *“Influence of acid conditioning on adhesion to dentin prepared with Er,Cr:YSGG laser” / “Influência do condicionamento ácido na adesão, em superfícies de dentina tratadas com laser Er,Cr:YSGG”* está de acordo com as normas e regras estipuladas pela Faculdade de Medicina Dentária da Universidade do Porto, foi por mim conferido e encontra-se em condições de ser apresentado e defendido em provas públicas.

Porto, 22 de Maio de 2020

A orientadora,

Ana Catarina Nogueira da Silva

(Prof. Dra. Ana Catarina Nogueira da Silva)

