



**CESPU**  
INSTITUTO UNIVERSITÁRIO  
DE CIÊNCIAS DA SAÚDE

Relatório de Estágio do Mestrado Integrado em Medicina Dentária

Surface modification of ultra-thin zirconia veneer for cementation: a scoping review of surface treatment and bond strength

Silvia Stuber Fogli

Setembro – 2019



**CESPU**  
INSTITUTO UNIVERSITÁRIO  
DE CIÊNCIAS DA SAÚDE

**Surface modification of ultra-thin zirconia veneer for cementation: a scoping review of surface treatment and bond strength**

Relatório de Estágio do Mestrado Integrado em Medicina Dentária apresentado para cumprimento dos requisitos necessários à obtenção do grau de Mestre em Medicina Dentária realizado sob a orientação científica do Professor Mestre Júlio César Matias de Souza.

## Declaração de relatório de estágio

Eu, Silvia Stuber Fogli, estudante do Curso de Mestrado Integrado em Medicina Dentária do Instituto Universitário de Ciências da Saúde, da Cooperativa de Ensino Superior Politécnico e Universitário, declaro ter atuado com absoluta integridade na elaboração deste Relatório de Estágio intitulado: "Surface modification of ultra-thin zirconia veneer for cementation: a scoping review of surface treatment and bond strength"

Confirmo que em todo o trabalho conducente à sua elaboração não recorri a qualquer forma de falsificação de resultados ou à prática de plágio (ato pelo qual um indivíduo, mesmo por omissão, assume a autoria do trabalho intelectual pertencente a outrem, na sua totalidade ou em partes dele). Mais declaro que todas as frases que retirei de trabalhos anteriores pertencentes a outros autores foram referenciados ou redigidos com novas palavras, tendo neste caso colocado a citação da fonte bibliográfica.

Relatório apresentado no Instituto Universitário de Ciências da Saúde.

Orientador: Professor Mestre Júlio César Matias de Souza

Gandra, \_\_\_\_ de setembro de 2019

-----

Silvia Stuber Fogli

**Aceitação do orientador:**

Eu, Júlio César Matias de Souza, com a categoria profissional de Professor do Instituto Universitário de Ciências da Saúde, tendo assumido o papel de Orientador do Relatório Final de Estágio intitulado: "Surface modification of ultra-thin zirconia veneer for cementation: a scoping review of surface treatment and bond strength" da aluna de Mestrado Integrado em Medicina Dentária, Silvia Stuber Fogli, declaro que sou de parecer favorável para que este relatório final possa ser presente ao júri para admissão a provas conducentes para obtenção do grau de Mestre.

Gandra, 22 de setembro de 2019

O orientador

-----

Júlio César Matias de Souza

## **Agradecimentos**

Ao meu orientador, Professor Doutor Júlio César Matias de Souza, pelo apoio, compreensão e disponibilidade constante para acompanhar a realização deste relatório final de estágio.

A toda a minha família, por me acompanharem, mesmo a distância de um oceano, neste e em todos os períodos da minha vida.

Ao meu companheiro, Antonio A. Lobo por sempre me apoiar nessa nova jornada de vida.

As minhas colegas de jornada, Mayara Boin, Nayra Bittencourt, Raissa Félix, Christian Coelho e Leandra Lopes.

Aos funcionários da CESP, meu muito obrigado.



## Resumo

O objetivo deste estudo foi realizar uma revisão integrativa sobre a modificação da superfície interna de facetas ultra-finas em zircônia para aumentar a resistência de união aos cimentos resinosos. Foi realizada uma busca eletrônica no banco de dados de literatura médica e científica on-line (PUBMED), com a seguinte combinação de termos de pesquisa: zircônia ultrafina, superfície, rugosidade, resistência da união e adesão. A busca identificou 643 estudos, dos quais 42 foram considerados relevantes para este estudo. A maioria dos estudos relatou o jateamento abrasivo como o principal método de modificação para superfícies à base de zircônia, embora possam ocorrer falhas por formação de fissuras devido as diferenças na pressão e no tipo de partículas abrasivas. A rugosidade das superfícies modificadas por jateamento variou de 0,25  $\mu\text{m}$  a 1,3  $\mu\text{m}$ . O aumento da rugosidade resultou em altos valores médios de resistência da união da zircônia aos cimentos resinosos. Métodos alternativos, como irradiação a laser e aplicação de camada de revestimento à base de vitrocerâmica foram descritos como métodos potenciais para modificação da superfície à base de zircônia. Os valores médios mais altos de resistência de união foram registrados em 41 MPa para superfícies modificadas por infiltração adesiva seletiva quando comparados aos valores médios em 37 MPa para superfícies com jateamento abrasivo. Assim, a combinação de métodos para modificação de superfície, infiltração seletiva adesiva após jateamento abrasivo, aumentou a resistência de união de superfícies à base de zircônia com cimentos resinosos, uma vez que os aspectos morfológicos e a rugosidade da superfície promoveram o aumento da resistência a união entre os dois materiais.

Palavras-chave: Modificação de superfície, facetas ultra finas de zircônia, resistência de união cimentos resinosos.

## Abstract

The aim of this study was to perform a scoping review on the surface modification of ultra-thin zirconia veneer to enhance the bond strength to resin-matrix cements. An electronic search on PUBMED database was performed using the following combination of search items: ultra-thin zirconia, surface, roughness, bond strength, and adhesion. The search identified 643 studies, of which 42 were considered relevant to this study. Most studies reported grit-blasting as the main ordinary modification method for zirconia-based surfaces although crack-like failures can occur due to bias in pressure and abrasive particle type. The roughness of the surfaces modified by grit-blasting ranged from 0.25 $\mu\text{m}$  up to 1.3  $\mu\text{m}$ . The increase in roughness resulted in high mean values of bond strength of zirconia surfaces to resin-matrix cements. Thus, alternative methods such as laser irradiation and etched glass-based coatings were described as potential methods for zirconia-based surface modification. The highest mean values of bond strength were recorded at 41 MPa for surfaces modified by selective infiltration etching when compared to mean values at 37 MPa for ordinary grit-blasted surfaces. Thus, the combination of surface modification methods, selective infiltration etching after grit blasting enhanced the bond strength of zirconia-based surfaces to resin-matrix cements, once the morphological aspects and roughness of the surface promoted the interlocking of resin-matrix cements.

Keywords: Surface modification, zirconia ultra-thin veneer, resin-matrix cements bond strength.



## Lista de abreviaturas

- 1- APA – air borne particle abrasion
- 2- AFM - atomic force microscopy
- 3- CAD/CAM - design assistido por computador / manufatura assistida por computador
- 4- CESPU - Centro de estudos superiores politécnico universitário
- 5- DGS - Direção Geral de Saúde
- 6- EDS - energy dispersive spectroscopy
- 7- SEM - scanning eletron microscopy
- 8- *Ra* - Arytmetic roughness
- 9- SIE - selective infiltration etch
- 10- SBS - shear bond strenght
- 11- XRD - X-ray diffraction

## Índice

Chapter I - Surface modification of ultra-thin zirconia veneer for cementation: a scoping review of surface treatment and bond strength.....	1
Introduction .....	1
2. Methods.....	2
3. Results .....	3
4. Discussion.....	5
4.1. Ordinary surface modification .....	5
4.2 Laser irradiation of zirconia surfaces .....	7
4.3 Silica and glass-ceramic coatings.....	8
5. Conclusions .....	10
References.....	11
Anexo .....	16
Capítulo II - Relatório das atividades práticas das disciplinas de estágio supervisionado.....	25
2.1 Estágio em clínica geral dentária .....	25
2.2 Estágio em clínica hospitalar .....	26
2.3 Estágio em saúde oral e comunitária.....	27

## **Chapter I - Surface modification of ultra-thin zirconia veneer for cementation: a scoping review of surface treatment and bond strength**

### **1. INTRODUCTION**

The rising concern on the health and aesthetics dentistry has encouraged the development of minimally invasive strategies considering clinical procedures and ceramic manufacturing approaches. Glass-ceramics are proper materials for manufacturing veneers, taking into account optical properties that mimic the teeth features. However, fractures can occur depending on the processing procedures, material mechanical properties, and patient conditions (1-8,18,19,27,28,37). Yttria tetragonal stabilized zirconia polycrystal (YTZP) has become a noteworthy choice to overcome the mechanical issues, although YTZP has limitations on the optical properties (9,17,34,38). Additionally, the modification of zirconia surfaces for veneering with glass-ceramics or cementation with resin-based cements is still a challenge concerning manufacturing, prosthetic design and zirconia properties. Most of the veneers' failures are related to the low bond strength of zirconia to glass-ceramics (chipping) or to resin-based cements.

In the last years, ultrathin YTZP veneers (0.1-0.3 mm) has been used depending on the clinical cases, zirconia type, and dental practitioner skills (4,5,35,39,42). A major drawback with the first generation of conventional YTZP is related to optical properties. Then, glass-ceramics are often applied onto the ultra-thin veneers to mimic the optical properties of teeth enamel. However, the thickness of the zirconia-based veneer varies from 0.2 up to 0.7 mm and the modification of the inner surfaces (surface treatment) for cementation can alter the selection of color and translucence (1-5,8,9,17,34,38) YTZP is a crystalline material which cannot be modified by ordinary acidic conditioning (e.g. phosphoric or hydrofluoric acid etching) and therefore the airborne particle abrasion (grit-blasting) is still widely used by technicians to modify the zirconia surfaces (5,8,14,19,25,30). Other physical approaches to increase zirconia

roughness have been reported in literature such as: grit-blasting with different silica particles, laser ablation, and micro-machining procedures (6-17,20-23,26,29,36,40). Therefore, silanization procedure is used to functionalize the zirconia surface for adhesion of the resin-based cement. However, there is no consensus on the roughness threshold of zirconia surfaces for cementation without compromise the mechanical and optical properties of the aesthetic veneers.

Thus, the selection of the resin-based cement and its thickness also can affect the optical properties of the aesthetic veneer (1-3,9,17,34,38). The modification of the inner surface of the zirconia surfaces results in irregularities like peak and valleys at macro- and micro-scale. Thus, the resin-matrix cement flow throughout the irregularities to fill the valleys leading to a variation in the cement line. Previous studies have reported a stress concentration at the resin-matrix cement during the thermal and occlusal loading variations in the oral cavity (1,2,4,5). Regarding the resin-matrix cement has a lower strength and elastic modulus than that recorded for zirconia, mechanical failures occur at the interface (5,6,9,22,24,31-34). Also, the high coefficient of thermal expansion of the resin-matrix cement determine also a high shrinkage during the decrease in temperature of oral cavity (6,17,33,41).

The aim of this study was to perform a scoping review on the inner surface modification approaches of ultra-thin zirconia restoration to enhance the bond strength to resin-matrix cements. It was hypothesized that the combination of physicochemical procedures to modify the inner zirconia surfaces can enhance the bond strength of zirconia to resin-matrix cements and therefore the long-term performance of the ultra-thin veneer restoration.

## 2. METHODS

A literature search was conducted on PUBMED database using the following search terms: "ultrathin zirconia" AND "adhesion" OR "bond strength" OR "roughness" OR "surface modification" OR "surface treatment" AND "resin cement" OR "cementation" OR "adhesive". A

manual search of the reference lists in the selected articles was also performed. The inclusion criteria encompassed articles published in the English language over the period from January 2003 up to February 2019, on the surface modification of ultra-thin zirconia veneers for resin-based cementation. Two of the authors (JCMS, SSF) independently evaluated the titles and abstracts of potentially relevant articles. The total of articles was retrieved for each combination of key terms and therefore the duplicates were removed using Mendeley citation manager. A preliminary evaluation of the abstracts was carried out to establish whether the articles met the inclusion criteria. Selected articles were individually read and analyzed concerning the purpose of this study. The following variables were harvested for this review: author names, journal, publication year, zirconia surface treatment & analysis, resin-matrix cement, bond strength, and failure mode (Table 1).

### **3. RESULTS**

A total of 643 papers were retrieved from the search on the electronic database. A number of 86 duplicates was removed. After reading the title and abstract, 204 articles were evaluated as potential studies and therefore 76 were excluded because they did not provide pertinent findings on the purpose of the present study. Out of the 118 excluded records were not eligible because of evaluating surface modifications of implants and crowns surfaces. Finally, 42 were selected for full reading in this review study. The selection method of studies is illustrated in Figure 1.

All involved studies were related to surface modifications of ultra-thin zirconia veneers, illustrated in Figure 2, and increase of bond strength of resin-matrix cements to ultrathin zirconia surfaces. The surface modifications are illustrated in Figure 3.

A total of 38% articles focused on the zirconia surface modification by airborne particle abrasion while 30% reported the laser irradiation effect on the zirconia surface. A total of 32% recorded the bond strength of zirconia to resin-based cements after zirconia surface modification by different approaches. The selected studies revealed the morphological and

chemical aspects of the modified zirconia surfaces by different techniques such as: scanning electron microscopy (SEM), atomic force microscopy (AFM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD). Roughness of the zirconia was measured at different parameters (e.g.  $R_a$  and  $R_t$ ) by optical profilometry or AFM. The surface analysis was illustrated in Figure 2. Scientific findings were related to the increase of bond strength of ultrathin zirconia surfaces to resin-matrix cements as follows:

- Air-borne particle abrasion (grit-blasting) of the inner surface of zirconia promoted an increase in roughness from 0.05 up to 1.0  $\mu\text{m}$  (5,6). The main abrasive alumina particles size was the following: 25, 50, and 110  $\mu\text{m}$  (5-16). As a result, the high-level roughness increased the bond strength of zirconia to resin-matrix cements (5,8,9,12,14);
- Pre-sintered zirconia showed higher mean values of roughness after grit-blasting with alumina particles since the hardness of pre-sintered is lower than that recorded for sintered zirconia (5,8,9,14). Also, the sintering of zirconia after grit-blasting promoted a decrease in cracks induced by the airborne particle abrasion which could occur by zirconia shrinkage during phase transformation (8);
- Defects like cracks originated from surface treatment negatively affected the strength of the thin zirconia-based veneers (10,11,16). The removal of material during laser ablation promoted a decrease in zirconia thickness at certain micro-regions that could be stress concentration spots for fracture propagation;
- $\text{CO}_2$  laser at 4W and Er,Cr:YSGG laser at 3W output power can be regarded as surface treatment methods for roughening the zirconia surface to increase the shear bond strength of zirconia to resin-matrix cements (7,10,11,16);
- Femtosecond laser irradiation on zirconia associated with a tribochemical silica coating resulted in standard roughness leading to an improvement of the adhesion between the zirconia and resin-matrix cements <sup>5</sup>

- Selective Infiltration Etching (SIE) significantly enhanced the average roughness of zirconia surfaces (6,13-17). Roughness values were higher ( $R_a$  at 378 nm) comparing to those recorded for grit-blasted surfaces ( $R_a$  at 157 nm) <sup>6</sup>. Roughness was affected by the infiltration material, etching time, and acid concentration <sup>7</sup>.
- Regarding the resin-matrix cements, the presence of MDP also improved the bond strength of zirconia to resin-matrix cements (8,9,12,13,15). The mean values of shear bond strength of zirconia to resin-matrix cements containing MDP ranged from 35 MPa up to 41 MPa while the values for zirconia to resin cements without MDP were at 8.7 MPa (13).

#### 4. DISCUSSION

The findings retrieved from the selected studies support the rejection of the null hypothesis. They showed significant increase in shear bond strength of resin-matrix cements to ultra-thin zirconia veneer surfaces. Results from the selected studies shown in Table 1 are detailly analyzed as follow:

##### 4.1. Ordinary surface modification

Ordinary methods are used to modify prosthetic surfaces to enhance the bond strength to other materials (2,5,8,12,14). Air borne particle abrasion is one of the ordinary methods to increase the zirconia-based roughness prior to cementation (5,8)

The zirconia surface modification is a hard task, since zirconia is a densely sintered material and consequently exhibits high hardness. That requires higher air pressure and/or abrasive  $Al_2O_3$  particles capable of promoting a desirable roughness (5,8). However, the association of high pressure and abrasive can result in micro-cracks from the surface towards into the zirconia bulk compromising its mechanical properties (2,12). Then, the grit-blasting of zirconia prior to

sintering can avoid the formation of micro-cracks once the material has a low hardness against the harder particles (5,8). Such procedure modification may allow the use of smaller particles to provide a surface whose roughness and topography are proper for cementation (5). Thus, air abrasion provided an increase in the monoclinic phase that occurs after grit-blasting although the sintering process can induce the total incorporation of monoclinic phase into tetragonal and/or cubic phases (8).

A previous study (5), evaluated the influence of surface modification of dyed and non-dyed zirconia on the bond between resin-matrix cement and zirconia ceramics and therefore findings revealed that non-dyed zirconia surface had significantly higher  $R_a$  roughness than those for dyed ceramic surface before grit-blasting. That indicates a high risk of crack propagation through non-dyed zirconia (5,6,8,14). Other studies (5,8) evaluated the effect of grit-blasting after sintering with different particles types. In fact, the extent of morphological changes on zirconia and the resultant phase transformation depends on the particles size and grit-blasting pressure. The highest mean values of shear bond strength were recorded at around 33 MPa for zirconia surfaces grit-blasted with 110  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  or  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  at 2.8 bar for 10 or 15 seconds (15).

The etching of zirconia surface with hydrofluoric acid (HF) has been studied considering the HF corrosive behavior although the zirconia has a high crystalline phase content (2,12). Previous studies showed a positive effect of surface treatment using a combination of grit-blasting and HF etching for 2 h at different concentration (20,30,40% HF) on the shear bond strength (SBS) of resin-matrix cement to zirconia. The highest SBS mean values were recorded for zirconia etched with 40% HF. Etching with HF resulted in a lower rate of monoclinic phase transformation when compared to solely grit-blasted zirconia. However, another study compared different surface treatment using grit-blasting with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  followed by 9% HF etching and NaOH solution. However, no significant difference was noticed between grit-blasting and etching treatment and therefore no significant effect of the etching on the roughness. SEM micrographs exhibited similar morphological aspects of the surfaces for experimental groups; however, grit-blasted surfaces showed more surface irregularities than



those on the other group. SBS highest mean values were recorded at around 9 MPa for groups modified by 0.01-MPa NaOH solution after grit-blasting procedure while grit-blasted surfaces without NaOH conditioning revealed a SBS at 3.3 MPa (12). Different procedures of grit-blasting has been studied to assess the roughness, zirconia structure, and bond strength to resin-matrix cements (9,15). The increase in roughness promotes a higher area for flowing of the resin-matrix cement and mechanical interlocking (6).

A previous study evaluated the combination of different grit-blasting procedures and three silane types. The grit-blasted methods performed on zirconia used 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , 30 $\mu\text{m}$  silica/alumina particles, 30 $\mu\text{m}$  silica/alumina particles with silica-encapsulated silane. Surfaces were grit-blasted using a grit-blasting handheld apparatus at 2.3 bar air pressure for 5 s (9). The silica used as abrasive particle can increase the mechanical interlocking between the resin-matrix cement and the zirconia surface since the silane was applied prior to resin-matrix cement. Also, acidic ethanol solutions of silane with phosphate coupling agents have been introduced due to the low adhesion between silanes and zirconia. Then, hydrolyzed methacryloxypropyl trimethoxysilane (MPTMS) is usually mixed with 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). Primers selected in the previous study were the following: S-Bond (SB, prehydrolyzed) and Clearfil Ceramic Primer Plus (CP, prehydrolyzed with 10-MDP). The main advantages of this combined procedure are the chemical bonding of coupling agents with zirconia and the remnant silica particles on the surface. Grit-blasting of zirconia with increased silica/alumina ratio powder and conditioning with a MPTMS/10-MDP silane was reported as the highest reliability procedure to enhance the bond strength of resin-matrix cements to zirconia. These findings highlighted the important role of 10-MDP on the bonding of rough ceramic substrate (9).

#### 4.2 Laser irradiation of zirconia surfaces

Laser irradiation is a recent method to increase the roughness of inner zirconia surface roughness for bonding to resin-matrix cements. The use of Nd:YAG laser on feldspar-based porcelain prior to adhesive cementation has resulted in improved bond strength values

comparable with surfaces etched in hydrofluoric acid solution. Also, there are some studies reporting that the Nd:YAG laser-treated alumina showed a micromechanical retention pattern which is more favorable for resin-matrix bonding (11). Another study evaluated the effect of various chairside treatments, such as Nd:YAG laser, grit-blasting with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , and glaze application followed by 9.5 % HF acid gel on the SBS of zirconia to resin-matrix cements (10). Highest SBS value was 8.17 MPa Nd:YAG short pulse with  $R_a$  roughness of zirconia at 6.39  $\mu\text{m}$  while the lowest mean values of SBS were at 4.26 MPa and  $R_a$  roughness at 0.16  $\mu\text{m}$  for grit-blasted surfaces.

Femtosecond laser is a novel laser-treatment currently being explored once the irradiation produces optical pulses lasting femtoseconds (1 fs =  $10^{-15}$ ) (7). Femtosecond laser (7) could represent a new alternative to conventional surface treatment. A previous study used 60 squares-like samples, of sintered Y-TZP as follow: group 1: control- no treatment surface; Group 2: Airborne Particle Abrasion (APA); Group 3: Tribochemical Silica Coating (TSC); Group 4: Femtosecond Laser irradiation. The results were that the femtosecond Laser irradiation and the TSC promoted a standard roughness on zirconia surface leading to an improvement in bond strength values to resin-matrix cements (7).

A previous study (4) compared the effect of different laser  $\text{CO}_2$ (4W) and Er,Cr: YSGG (3W and 4W) and grit-blasting with 50  $\mu\text{m}$   $\text{Al}_2\text{O}_3$ . Air borne abrasion (grit-blasting) showed the highest shear bond strength (37.3 MPa) when compared to surfaces irradiated with Carbon Dioxide ( $\text{CO}_2$ ) laser (29 MPa) and Er,Cr:YSGG laser irradiation (21.5 MPa) (10). In agreement with another study (16), the highest SBS value was at 12 MPa for grit-blasted surfaces with 110  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  ( $R_a$  roughness at 0.38  $\mu\text{m}$ ) when compared to surfaces irradiated with  $\lambda$  2940nm Er:YAG (SBS at 5.6 MPa and  $R_a$  roughness at 0.66  $\mu\text{m}$ ) (10,16).

### 4.3 Silica and glass-ceramic coatings

Selective Infiltration Etching (SIE) is an alternative promising method used to increase bond strength of zirconia to resin-matrix cements. SIE relies on a heat-induced maturation process that acts by stressing zirconia grain boundaries by two short thermal cycles to promote a highly retentive, reactive and nanoporous surface. A previous study compared grit-blasting and SIE to modify zirconia surfaces (13). Thus, SIE provided a selective area of zirconia grain to be in contact with MDP from resin-matrix cements leading to enhancement of chemical reactivity of the surface (13). In combination, a thin layer of a low melting glass with various additives and different coefficient of thermal expansion is applied to the surface. On the semi-liquid state, the molten glass infiltrates selectively between the surface grains and exerts surface tension and capillary forces, allowing sliding and splitting of the grains. This rearrangement movements result in a three-dimensional network of integrate porosity. The treatment acts only on the surface grains that contact the infiltration glass and therefore the operator can control the treated area. Following the thermal treatment protocol, traces of the infiltration agent are dissolved in a 9% hydrofluoric acid solution and the final surface can be infiltrated and conditioned by MPTMS and MDP silane and primer prior to the resin-matrix cement (11). However, it has long been stated that mixing hydrolyzed silanes with dental monomers possessing OH groups, deactivates the silanol (Si-OH) groups via condensation (9). SIE revealed the highest SBS at around 41.5 MPa value (13) that revealed an combined and hybrid approach to enhance the bond strength of zirconia to resin-matrix methods.

## 5. CONCLUSIONS

Considering the relevant findings reported in the last fifteen years, several methods for zirconia surface modifications have emerged and numerous studies claimed to increase roughness and bond strength of zirconia to resin-matrix cements. Within the limitations of in vitro studies, the following outcome of the retrieved studies can be drawn:

- The surface treatment with alumina grit-blasting as an ordinary method had revealed clinically acceptable shear bond strength values. However, failures due to the presence of micro-cracks can occur due to the air borne abrasion pressure and particle type;
- The grit-blasting prior to final sintering resulted in a decrease in micro-cracks in the zirconia;
- The combination of surface conditioning with sodium hydroxide or hydrofluoric acid after grit-blasting also enhance the bond strength of zirconia to resin-matrix cements;
- The difference between treated surfaces by different laser irradiation (e.g. CO<sub>2</sub> and Er:YAG) had no significant differences in shear bond strength of zirconia to resin-matrix cements. However, the roughness increased in function of the irradiation time;
- Selective infiltration etching provided high mean values of shear bond strength of zirconia to resin-matrix cements considering the reactive and retentive area in contact with resin-matrix contents;
- The coating of zirconia with glass-ceramic can be useful for etching with hydrofluoric acid leading to an increase in roughness and mechanical interlocking of the resin-matrix cement;
- Further studies should be performed on the different parameters of laser irradiation such as power, irradiation time, and type of laser. In addition, the combination of ordinary techniques with advance laser treatment, glass-ceramic, and acidic etching can enhance the bond strength of zirconia to resin-matrix cements.

## REFERENCES

1. LE M, LARSSON C, PAPIA E. Bond strength between MDP-based cement and translucent zirconia. *Dent Mater J* [Internet]. 2019; Available from: [https://www.jstage.jst.go.jp/article/dmj/advpub/0/advpub\\_2018-194/\\_article](https://www.jstage.jst.go.jp/article/dmj/advpub/0/advpub_2018-194/_article)
2. Chen X-D, Hong G, Xing W-Z, Wang Y-N. The influence of resin cements on the final color of ceramic veneers. *J Prosthodont Res*. 2015;59(3):172–7.
3. Della Bona A, Borba M, Benetti P, Pecho OE, Alessandretti R, Mosele JC, et al. Adhesion to Dental Ceramics. *Curr Oral Heal Reports*. 2014;1(4):232–8.
4. Akhavan Zanjani V, Ahmadi H, Nateghifard A, Ghasemi A, Torabzadeh H, Abdoh Tabrizi M, et al. Effect of different laser surface treatment on microshear bond strength between zirconia ceramic and resin cement. *J Investig Clin Dent*. 2015;6(4):294–300.
5. Vicente Prieto M, Gomes ALC, Montero Martín J, Alvarado Lorenzo A, Seoane Mato V, Albaladejo Martínez A. The Effect of Femtosecond Laser Treatment on the Effectiveness of Resin-Zirconia Adhesive: An In Vitro Study. *J Lasers Med Sci* [Internet]. 2017;7(4):214–9. Available from: <http://dx.doi.org/0.15171/jlms.2016.38>
6. Akay C, Tanış MÇ, Mumcu E, Kılıçarslan MA, Şen M. Influence of nano alumina coating on the flexural bond strength between zirconia and resin cement. *J Adv Prosthodont*. 2018;10(1):43.
7. Oh G-J, Yoon J-H, Vu VT, Ji M-K, Kim J-H, Kim J-W, et al. Surface Characteristics of Bioactive Glass-Infiltrated Zirconia with Different Hydrofluoric Acid Etching Conditions. *J Nanosci Nanotechnol*. 2017;17(4):2645–8.
8. Abi-Rached F, Martins S, Almeida-Júnior A, Adabo G, Góes MS, Fonseca R. Air Abrasion

Before and/or After Zirconia Sintering: Surface Characterization, Flexural Strength, and Resin Cement Bond Strength. *Oper Dent.* 2014;40(2):E66–75.

9. Skienhe H, Habchi R, Ounsi H, Ferrari M, Salameh Z. Evaluation of the Effect of Different Types of Abrasive Surface Treatment before and after Zirconia Sintering on Its Structural Composition and Bond Strength with Resin Cement. *Biomed Res Int.* 2018;2018:1–12.
10. Lin Y, Song X, Chen Y, Zhu Q, Zhang W. Effect of Er:YAG Laser Irradiation on Bonding Property of Zirconia Ceramics to Resin Cement. *Photomed Laser Surg.* 2013;31(12):619–25.
11. Lv P, Yang X, Jiang T. Influence of hot-etching surface treatment on zirconia/resin shear bond strength. *Materials (Basel).* 2015;8(12):8087–96.
12. Flores-Ferreyra BI, Cougall-Vilchis RJS, Velazquez-Enriquez U, Garcia-Contreras R, Aguillon-Sol L, Olea-Mejia OF. Effect of airborne-particle abrasion and, acid and alkaline treatments on shear bond strength of dental zirconia. *Dent Mater J.* 2019;38(2):182–8.
13. Akay C, Tanış MÇ, Mumcu E, Kılıçarslan MA, Şen M. Influence of nano alumina coating on the flexural bond strength between zirconia and resin cement. *J Adv Prosthodont.* 2018;10(1):43.
14. Xie ZG, Meng XF, Xu LN, Yoshida K, Luo XP, Gu N. Effect of air abrasion and dye on the surface element ratio and resin bond of zirconia ceramic. *Biomed Mater.* 2011;6(6).
15. Akay C, Tanış MÇ, Mumcu E, Kılıçarslan MA, Şen M. Influence of nano alumina coating on the flexural bond strength between zirconia and resin cement. *J Adv Prosthodont.* 2018;10(1):43.
16. Lin Y, Song X, Chen Y, Zhu Q, Zhang W. Effect of Er:YAG Laser Irradiation on Bonding Property of Zirconia Ceramics to Resin Cement. *Photomed Laser Surg.* 2013;31(12):619–25.

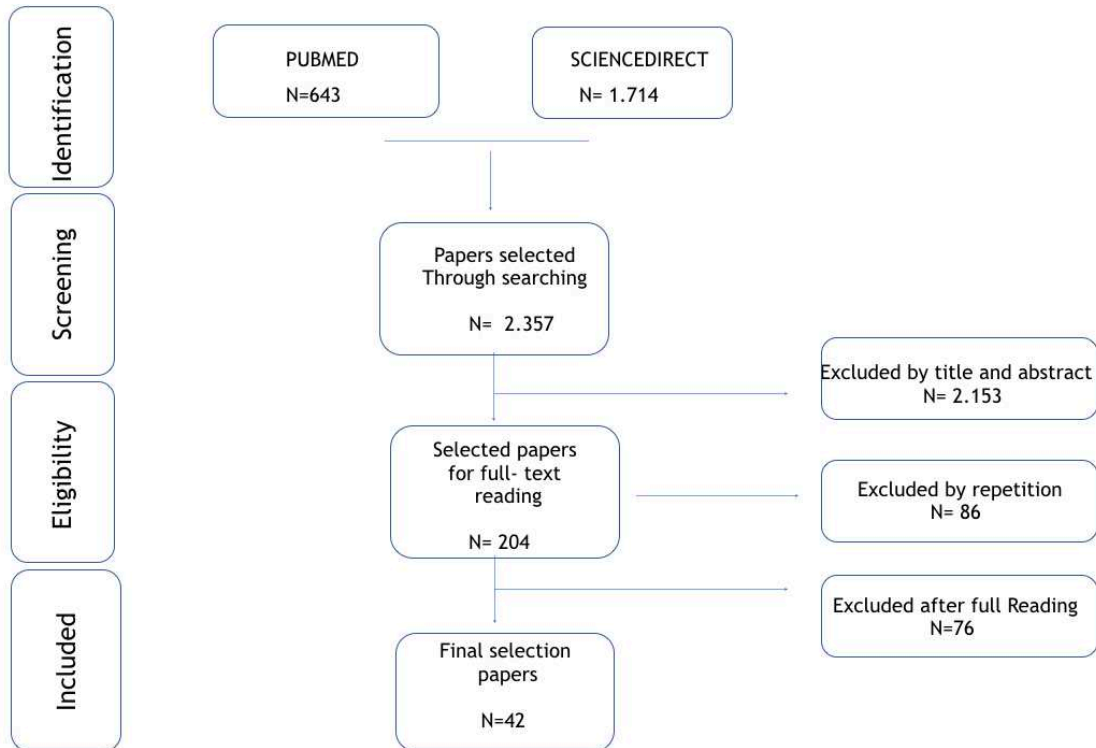
17. Oh G-J, Yoon J-H, Vu VT, Ji M-K, Kim J-H, Kim J-W, et al. Surface Characteristics of Bioactive Glass-Infiltrated Zirconia with Different Hydrofluoric Acid Etching Conditions. *J Nanosci Nanotechnol*. 2017;17(4):2645–8.
18. Blatz MB, Alvarez M, Sawyer K, Brindis M. How to Bond Zirconia: The APC Concept. *Compend Contin Educ Dent*. 2016;37(9):611–8.
19. Vanderlei AD, Queiroz JRC, Bottino MA, Valandro LF. Improved adhesion of Y-TZP ceramics: A novel approach for surface modification. *Gen Dent*. 2014;62(1):e22–7.
20. Sarmento HR, Campos F, Sousa RS, Machado JPB, Souza ROA, Bottino MA, et al. Influence of air-particle deposition protocols on the surface topography and adhesion of resin cement to zirconia. *Acta Odontol Scand*. 2014;72(5):346–53.
21. Lee MH, Son JS, Kim KH, Kwon TY. Improved resin-zirconia bonding by room temperature hydrofluoric acid etching. *Materials (Basel)*. 2015;8(3):850–66.
22. Phark JH, Duarte S, Hernandez A, Blatz MB, Sadan A. In vitro shear bond strength of dual-curing resin cements to two different high-strength ceramic materials with different surface texture. *Acta Odontol Scand*. 2009;67(6):346–54.
- 23.. de Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic. *Dent Mater*. 2009;25(2):172–9.
24. Lung CYK, Kukk E, Matinlinna JP. Shear bond strength between resin and zirconia with two different silane blends. *Acta Odontol Scand*. 2012;70(5):405–13.
25. Sawada T, Spintzyk S, Schille C, Zöldföldi J, Paterakis A, Schweizer E, et al. Influence of pre-sintered zirconia surface conditioning on shear bond strength to resin cement. *Materials (Basel)*. 2016;9(7).

26. Jiang T, Chen C, Lvc P. Selective infiltrated etching to surface treat zirconia using a modified glass agent. *J Adhes Dent.* 2014;16(6):553–7.
27. Viana PC, Portugal J, Kovacs Z, Lopes I, Correia A. Resin-bonded fixed dental prosthesis with a modified treatment surface in a zirconia framework: a case report. *Int J Esthet Dent.* 2016;11(3):378–92.
28. Elsaka SE. Influence of surface treatment on the bond strength of resin cements to monolithic zirconia. *J Adhes Dent.* 2016;18(5):387–95.
29. Esteves Oliveira M, Wehner M, Silva M, de Paula Eduardo C, Dohrn A, Meyer-Lückel H, et al. Surface Characterization and Short-term Adhesion to Zirconia after Ultra-short Pulsed Laser Irradiation. *J Adhes Dent.* 2017;18(6):1–10.
30. Nakazawa K, Nakamura K, Harada A, Shirato M, Inagaki R, Örtengren U, et al. Surface properties of dental zirconia ceramics affected by ultrasonic scaling and low-temperature degradation. *PLoS One.* 2018;13(9).
31. Ioannidis A, Mühlemann S, Özcan M, Hüsler J, Hämmerle CHF, Benic GI. Ultra-thin occlusal veneers bonded to enamel and made of ceramic or hybrid materials exhibit load-bearing capacities not different from conventional restorations. *J Mech Behav Biomed Mater.* 2019;90:433–40.
32. De Sousa RS, Campos F, Sarmiento HR, Alves MLL, Dal Piva AMDO, Gondim LD, et al. Surface roughness and bond strength between Y-TZP and self-adhesive resin cement after air particle abrasion protocols. *Gen Dent.* 2016;64(5):50–5.
33. Blatz MB, Vonderheide M, Conejo J. The Effect of Resin Bonding on Long-Term Success of High-Strength Ceramics. *J Dent Res.* 2018;97(2):132–9.

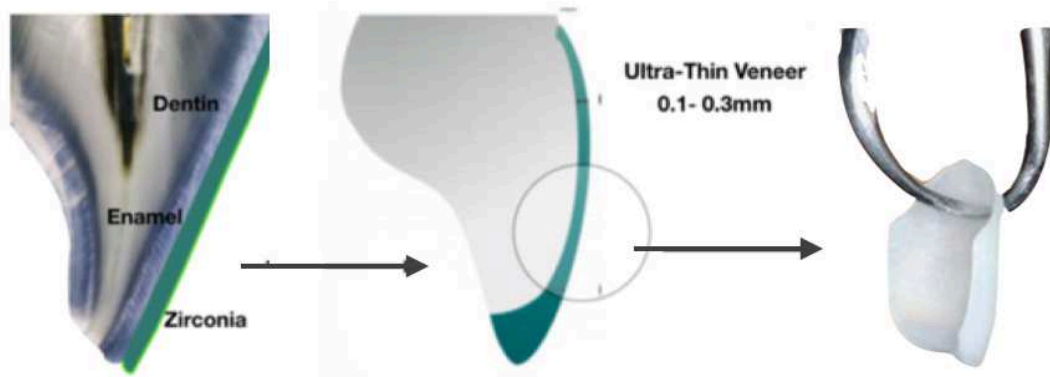


34. Souza R, Barbosa F, Araújo G, Miyashita E, Bottino MA, Melo R, et al. Ultrathin monolithic zirconia veneers: Reality or future? report of a clinical case and one-year follow-up. *Oper Dent*. 2018;43(1):3–11.
35. LE M, LARSSON C, PAPIA E. Bond strength between MDP-based cement and translucent zirconia. *Dent Mater J [Internet]*. 2019;
36. Wang C, Niu LN, Wang YJ, Jiao K, Liu Y, Zhou W, et al. Bonding of resin cement to zirconia with high pressure primer coating. *PLoS One*. 2014;9(7):3–4.
37. Egilmez F, Ergun G, Cekic-Nagas I, Vallittu PK, Ozcan M, Lassila LVJ. Effect of surface modification on the bond strength between zirconia and resin cement. *J Prosthodont*. 2013;22(7):529–36.
- 38.. Tabatabaian F, Bakhshaei D, Namdari M. Effect of Resin Cement Brand on the Color of Zirconia-Based Restorations. *J Prosthodont*. 2018;
39. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: Where are we now? *Dent Mater*. 2011;27(1):71–82.
40. Ghasemi A, Torabzadeh H, Kermanshah H, Ghavam M, Nateghifard A, Zolfagharnasab K, et al. Effect of Er,Cr: YSGG laser treatment on microshear bond strength of zirconia to resin cement before and after sintering. *J Adhes Dent*. 2014;16(4):377–82.
41. Paranhos MPG, Burnett LH, Magne P. Effect Of Nd:YAG laser and CO2 laser treatment on the resin bond strength to zirconia ceramic. *Quintessence Int [Internet]*. 2011;42(1):79–89.
42. Kim MJ, Kim YK, Kim KH, Kwon TY. Shear bond strengths of various luting cements to zirconia ceramic: Surface chemical aspects. *J Dent*. 2011;39(11):795–803.

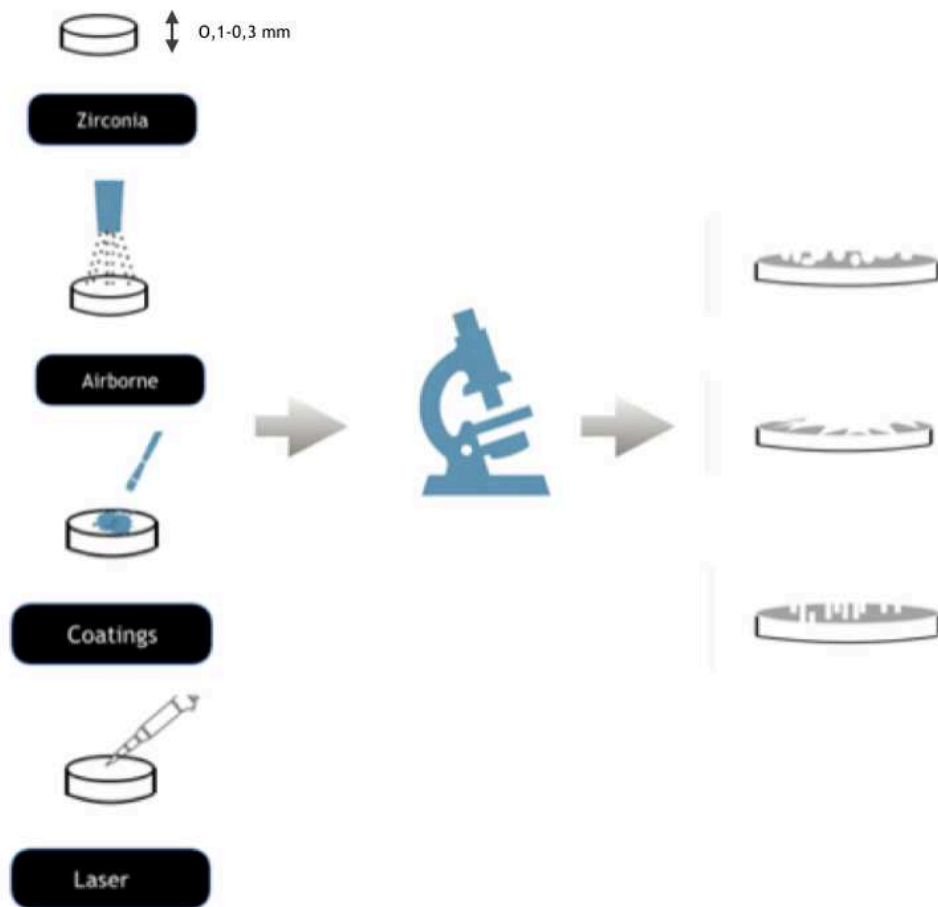
## Annex



*Figure 1. Search Strategy used in this study*



*Figure 2. Schematic of ultra-thin zirconia veneer*



*Figure 3. Surface treatments and analysis*

**Table 1. Summary of relevant Studies on zirconia surface treatment.**

Author (Year)	Surface Treatment	Shearbond Strength (MPa)	Roughness ( $\mu\text{m}$ )	Failure Mode	Surface Analysis	Resin-matrix cement
Skienhe et al. (2018)	Grit-blasting ( $\text{Al}_2\text{O}_3$ ) A) $\text{Al}_2\text{O}_3$ control B) $50\mu\text{m}$ $\text{Al}_2\text{O}_3$ B1) After Sintering B2) Before Sintering C) $25\mu\text{m}$ $\text{Al}_2\text{O}_3$ C1) After Sintering C2) Before Sintering	A) 11.58 Mpa  B1) 17.59 B2) 15.86  C1) 10.21 C2) 12.07	A) $0.05\mu\text{m}$  B1) 0.0 B2) 0.1  C1) 0.7 C2) 0.7	Adhesive	SEM XRD	Rely X ARC 3M/ESPE  Base: Glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator Catalyst: Methacrylated, phosphoric esters, dimethacrylates, acetate, stabilizers, self-cure initiators, light-cure initiators
Pilo et al. (2017)	A) $\text{Al}_2\text{O}_3$ grit-blasting B) Silane B1) $30\mu\text{m}$ silica/alumina B2) $30\mu\text{m}$ silica/alumina B3) $30\mu\text{m}$ silica/alumina, silica encapsulated $\gamma$ -MPTMS( $\gamma$ -methacryloxypropyl trimethoxy silane) $50\mu\text{m}$ alumina particles	A) 9.14MPa  B1) 14.0 B2) 14.0 B3) 12.0	A) $4.2\mu\text{m}$  B1) 3.16 B2) 3.72 B3) 3.37	Adhesive	Optical Profiler Mirau Lens 40x	Clearfill SA Ceramic Primer(CP), 10MDP Kuraray Noritake Bis GMA, UDMA,TEGDMA, Barium glass

Gye et al. (2017)	A) B.Glass +HF10% A1) Glass and 10% HF for 10min A2) Glass and 10%HF for 1h B)Glass +HF20%  B1) Glass +HF20% for 10min B2) B.Glass +HF20% for 1h		A1) 0.3μm A2) 1.1  B1) 0.5 B2) 2.0		SEM	
Pin et al. (2015)	Hot Etching Surface Grit-blasting (Al <sub>2</sub> O <sub>3</sub> )  A)Control B) 50μm Al <sub>2</sub> O <sub>3</sub> for 10min C)Hotetching for 1h	A)9.61MPa B)5.8 C)29.2	A)1.31μm B)6.64 C)6.41	Adhesive Mixed	SEM AFM XRD	Panavia F Kuraray Noritake Bis GMA, UDMA,TEGDMA, Barium glass, MDP
Abi et al. (2015)	Grit-blasting (50μm Al <sub>2</sub> O <sub>3</sub> )  A) Before sintering B) After sintering C) Before after sintering	A)2.6MPa B)5.4 C)7.0	A)1.3μm B)0.7 C)1.0	Adhesive	Profilometer SEM	Relyx ARC
Uzumez et al. (2013)	Grit-blasting GLAZE LASER Nd:YAG  A)Glaze B) 50μm Al <sub>2</sub> O <sub>3</sub> C)Control D)Nd:YAG /180μs E) Nd:YAG/320μs	A)4.99MPa B)4.26 C)3.73 D)6.99 E)8.17	A)0.67μm B)0.16 C)0.19 D)3.41 E)6.39	Adhesive	X-Ray SEM	Clearfill Primer

Xie et al.  
(2011)

Grit-blasting Al<sub>2</sub>O<sub>3</sub>  
Dyed Zirc.

Adhesive

3D LaserScannig  
Microscope

Panavia F

A) Polished  
B) Dyed +polished  
C) 50μm Al<sub>2</sub>O<sub>3</sub>  
D) Dyed + Al<sub>2</sub>O<sub>3</sub>

A) 0.59MPa  
B) 0.50  
C) 14.63  
D) 14.58

A) 0.28 μm  
B) 0.25  
C) 0.49  
D) 0.53

Canan et al.  
(2018)

Grit-blasting +Silica coated  
Aluminum oxide

SEM

Panavia F

A) Control  
B) Grit-blasting (110μm Al<sub>2</sub>O<sub>3</sub>)  
C) ROC(110μm Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub>)  
D) Tribochemical silica coat  
E) Alumin Nitrate 1.2μm coating

A) 12.0MPa  
B) 35.6  
C) 47.8  
D) 30.8  
E) 50.4

B) 157nm  
C) 378  
D) 216  
E) 195

Zanjani et al.  
(2014)

Grit-blasting Al<sub>2</sub>O<sub>3</sub>+ Laser CO<sub>2</sub>  
Er,Cr:YSGG

Mixed

SEM

Panavia F

A) 50μm Al<sub>2</sub>O<sub>3</sub>  
B) CO<sub>2</sub> Laser(4w)  
C) Er,Cr :YSGG(3W)  
D) Er,Cr:YSGG(4W)

A) 37.30MPa  
B) 29.08  
C) 27.52  
D) 21.52

Ferreya et al. (2018)	Grit-blasting + HF				Mixed	AFM SEM	Clearfill Primer
	Grit-blasting +NaOH						
	A) Grit-blasting 50µm Al <sub>2</sub> O <sub>3</sub>	A)8.4MPa	A)0.30µm				
	B) Grit-blasting +HF 9%	B)3.3	B)0.28				
	C) Grit-blasting +NaOH (0.01-M)	C)9.0	C)0.35				Panavia F2.0
Tanis et al. (2018)	Grit-blasting +Selective Infiltration etching(SIE)					AFM	Variolink
	A) Grit-blasting (50µm Al <sub>2</sub> O <sub>3</sub> )	A1)8.57 MPa					
	A1) Grit-blasting + Variolink	A2)35.17MPa					
	A2) Grit-blasting + Panavia	B1)28.47					
	B)SIE	B2)41.50					
	B1)SIE+ Variolink						
B2)SIE+Panavia						Panavia F2.0	



Lin et al. (2013)	Grit-blasting / Er:YAGG			Adhesive Mixed	SEM	Clearfill Cement
	A) Grit-blasting 50 $\mu$ m Al <sub>2</sub> O <sub>3</sub>	A)12.03MPa	A)0.38 $\mu$ m			
	B)Er:YAGG					
	B1)100mJ pw1 for 5min	B1) 4.26	B1) 0.90			
	B2) 100mJ pw1 for 10min	B2) 3.66	B2) 0.30			
	B3) 100mJ pw1 for 15min	B3) 3.83	B3) 0.30			
	C) Er:YAGG					
	C1) 200mJ pw1 for 5 min	C1) 4.60	C1) 0.55			
	C2) 200mJ pw1 for 10min '	C2) 5.37	C2) 0.56			
	C3) 200mJ pw1 for 15min	C3) 4.82	C3) 0.59			
	D) Er:YAGG	D1) 4.88				
	D1) 300mJ pw1 for 5 min	D2) 5.74	D1) 0.59			
	D2) 300mJ pw1 for 10min	D3) 5.61	D2) 0.64			
	D3) 300mJ pw1 for 15min		D3) 0.66			
Lee et al. (2017)	Grit-blasting + HF				CLSM	Variolink
	A) Grit-blasting (110 $\mu$ m Al <sub>2</sub> O <sub>3</sub> )	A)1.45MPa				
	B) Grit-blasting +HF				SEM	
	B1) Grit-blasting +HF 20% for 1h	B1)0.048	B1) 26.03nm			
	B2) Grit-blasting +HF 20% for 2h	B2)0.062	B2) 33.67		XRD	
	B3) Grit-blasting +HF 30% for 1h	B3)1.95	B3) 40.6			Panavia F2.0
	B4) Grit-blasting +HF 30% for 2h	B4)2.56	B4) 51.03			

Pietro et al.  
(2016)

Femtosecond Laser  
Grit-blasting  
Tribochemical coating(TSC)  
A)Control  
B) 25 $\mu$ m Al<sub>2</sub>O<sub>3</sub>  
C) TSC  
D)Femtosecond  
800nm,4J,40fs/1kh2

A)4.4MPa  
B)8.1  
C) 9.5  
D) 10.8

Adhesive

SEM

Clearfill Cement

## Capítulo II - Relatório das atividades práticas das disciplinas de estágio supervisionado

### 2.1 Estágio em clínica geral dentária

O estágio em clínica geral dentária decorreu na Clínica Universitária Filinto Baptista - Gandra – Paredes, num período de cinco horas semanais, às sextas-feiras, das 19h às 23h, com início no dia 14 de setembro de 2018 e término no dia 11 de junho de 2019.

Supervisionados pelo professor doutor João Batista o estágio nos permitiu aplicar os conhecimentos teóricos em um contexto clínico satisfatório, que se mostrou uma mais valia no aprimoramento da prática médico-dentária. Os atos clínicos realizados nesse estágio estão discriminados na seguinte tabela:

Ato Clínico	Operador	Assistente	Total
Triagem	04	03	07
Dentisteria	10	03	13
Endodontia	03	01	04
Exodontia	08	06	14
Destartarização	08	04	12
Outros*	09	04	13
Total	42	21	63

\*Procedimentos pós-operatório (remoção de espículas ósseas, de sutura), medicação, radiografia, referência.

## 2.2 Estágio em clínica hospitalar

O estágio em clínica hospitalar foi realizado no Hospital de Guimarães, no período de 14 de setembro 2018 a 14 de junho de 2019, com uma carga semanal de 3 horas compreendidas entre 09:00h - 12:00h das sextas-feiras, sob a supervisão da professor doutor Fernando Figueira. Esse estágio nos permitiu contactar com uma diversidade de pacientes: polimedicados, portadores de doenças sistémicas, pacientes com problemas psicológicos, em situação de vulnerabilidade social e com baixa literacia em saúde oral, oferecendo ferramentas importantes para uma atuação clínica autónoma e responsável, assente nas decisões mais adequadas frente às diversas situações clínicas com que podemos nos deparar.

Os atos clínicos realizados nesse estágio estão discriminados na seguinte tabela:

Ato Clínico	Operador	Assistente	Total
Triagem	01	05	06
Dentisteria	16	13	29
Endodontia	02	02	04
Exodontia	22	23	45
Destartarização	10	08	18
Outros	08	04	12
Total	64	57	121

\*Procedimentos pós-operatório (remoção de espículas ósseas, de sutura), medicação, radiografia, referênciação.

### 2.3 Estágio em saúde oral e comunitária

O estágio em saúde oral e comunitária decorreu num período de 3,5 horas semanais, compreendidas entre as 9h e 12:00h de quinta-feira, com início no dia 13 de setembro de 2018 e término no dia 14 de junho de 2019 e sob a supervisão do professor doutor Paulo Rompante.

O plano de atividade de cada binómio da turma foi enviado via plataforma digital. Foram realizadas diversas atividades, com a finalidade de promover a saúde oral da população de Santo Tirso numa perspetiva de prevenção e promoção de saúde oral.

As atividades realizadas ao longo deste estágio encontram-se descritas na tabela seguinte:

Data	Atividade
14/09/18	Elaboração Tarefa 1/ Projeto de intervenção comunitária na área da saúde oral. Estabelecimento Prisional do norte de Portugal.
02/10/18	Deposito em do plataforma projetos de atividade Tarefa1
10/11/18	Elaboração Tarefa 2/ Hospital da Misericórdia implementar um Projeto de Intervenção Comunitária na área da Saúde Oral.
17/11/18	Deposito em do plataforma projetos de atividade Tarefa 2.
29/11/18 a 13/06/19	Projeto de intervenção comunitária no Hospital de Santo Tirso. Atendimento em medicina dentária e promoção a saúde oral
17/12/18	Elaboração da Tarefa 3 /Projetos de atividade de intervenção comunitária de rua na área da Saúde Oral.
20/12/18	Deposito em do plataforma projetos de atividade da Tarefa 3 /intervenção comunitária de rua na área da Saúde Oral.

31/01/19	Elaboração Tarefa 3/Projetos de atividade de intervenção comunitária de rua na área da Saúde Oral.
15/03/2019	Elaboração Tarefa 4/ demonstrar ter conhecimento, reciclar ou adquirir o conhecimento sobre a temática: "Patologias sistémicas com repercussões na cavidade oral. Conhecer e saber como proceder"
01/03/2019	Deposito em do plataforma projetos de atividade da Tarefa 4 /" Patologias sistémicas com repercussões na cavidade oral. Conhecer e saber como proceder"
21/03/19	Deposito Tarefa 5/ comprovativo da inscrição, o comprovativo do seu diploma e o comprovativo da validação da sua presença e assistência a 100% da temática em questão "Patologia benigna dos tecidos moles em Odontopediatria. Diagnóstico e terapêutica em ambulatório"
29/03/19	Deposito Tarefa 6/ comprovativo da inscrição, o comprovativo do seu diploma e o comprovativo da validação da sua presença e assistência a 100% da temática em questão e "Relatório em forma de Guedelines de diagnóstico e terapêutica"
13/06/19	Ultima clinica intervenção comunitária no Hospital de Santo Tirso