UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

ALFREDO ESTEBAN LLERENA ICOCHEA

Influence of simplified surface treatments protocols on the bond strength between resin cements and reinforced glass-based ceramics

Influência de protocolos de tratamentos de superfície simplificados na resistência de união entre cimentos resinosos e cerâmicas vítreas reforçadas

BAURU
2021

ALFREDO ESTEBAN LLERENA ICOCHEA

Influence of simplified surface treatments protocols on the bond strength between resin cements and reinforced glass-based ceramics

Influência de protocolos de tratamentos de superfície simplificados na resistência de união entre cimentos resinosos e cerâmicas vítreas reforçadas

Tese constituída por artigos apresentada à Faculdade de Odontologia de Bauru da Universidade de São Paulo para obtenção do título de Doutor em Ciências no Programa de Ciências Odontológicas Aplicadas, na área de concentração Dentística.

Orientador: Prof. Dr. Adilson Yoshio Furuse

BAURU

2021

Llerena Icochea, Alfredo Esteban
Influência de protocolos de tratamentos de superfície simplificados na resistência de união entre cimentos resinosos e cerâmicas vítreas reforçadas / Alfredo Esteban Llerena Icochea. - Bauru, 2021.
64 p. : il. ; 31 cm.

Tese (Doutorado) -- Faculdade de Odontologia de Bauru, Universidade de São Paulo, 2021.

Orientador: Prof. Dr. Adilson Yoshio Furuse

ERRATA

FOLHA DE APROVAÇÃO

DEDICATÓRIA

À Deus

Pelo fôlego de vida, por iluminar sempre meu caminho e por me capacitar a cada dia.

À minha família

Os quais sempre amarei e honrarei pelo esforço que fizeram pela minha formação pessoal e acadêmica. Sempre que precisei, preciso e precisarei sei que posso contar com vocês.

À minha esposa e melhor amiga Jussara

Pelo amor incondicional, ajuda e por todas as alegrias juntos. Sem você isto não seria possível. Se tem alguém que merece este título mais do que eu, é só você mesma. Obrigado por tudo meu amor.

Ao meu filho Enzo

Você me ensinou que existe um sentimento além do amor, o que eu sinto por você é indescritível, todo meu amor, esforço e trabalho serão sempre por você.

AGRADECIMENTOS

Agradeço à toda a minha família Peruana e Brasileira pela torcida e apoio.

Ao meu orientador Prof. Dr. Adilson Yoshio Furuse pela orientação ao longo desta caminhada.

Ao meu querido Professor José Mondelli e esposa dona Marisa pelo exemplo de vida compartilhado no dia a dia e pelo carinho demonstrado à minha família.

Ao meu amigo Adriano Mondelli, por ser quem me impulsionou sempre a buscar mais pela minha vida acadêmica.

Aos professores do departamento de Dentística da FOB, Rafael Mondelli, Sérgio e Aquira Ishikiriama, Linda Wang, Maria Teresa Atta, Juliana Bombonatti, Paulo Franciscone, meus agradecimentos pela oportunidade de trabalhar com vocês, por todo o conhecimento passado á mim e pelos exemplos de dedicação à carreira e profissão.

À Professora Ana Flávia Sanches Borges por toda ajuda e suporte durante este trabalho.

Aos funcionários do departamento de Dentística, Audria, Sharlene, Natália, Rita, Elizeo, Nelson, Zuleika e Dona Bety por toda dedicação aos alunos.

À Faculdade de Odontologia de Bauru-USP, na pessoa do senhor diretor Prof. Dr. Carlos Ferreira dos Santos.

Ao Conselho Nacional de Pesquisa (CNPq), pelo apoio financeiro durante a realização destes estudos de Doutorado.

“A maior recompensa para o trabalho do homem não é o que ele ganha com isso, mas o que ele se torna com isso”.

John Ruskin

RESUMO

O correto protocolo de tratamento de superfície de cerâmicas é uma etapa muito importante no processo de cimentação e pode influenciar na resistência de união final. Nestes dois estudos, o objetivo foi avaliar a influência de diferentes tratamentos de superfície realizados em cerâmicas a base de sílica. Cortes de silicato de lítio reforçado com zircônia e dissilicato de lítio foram obtidos e submetidos a diferentes protocolos de alteração de superfície. O ácido fluorídrico (HF) e o polifluoreto de amônio (MBEP) foram utilizados em ambos os estudos para alteração topográfica da superfície e posteriormente utilizou-se diferentes materiais adesivos e protocolos para uma possível potencialização da resistência de união final. Um dos estudos usou a termociclagem para envelhecimento dos espécimes antes da avaliação da resistência de união por micro-cisalhamento (μSBS). O modo de falha foi analisado com uma lupa estereoscópica. No primeiro estudo, os grupos que utilizaram HF e MBEP resultaram em um μSBS significativamente maior do que o grupo sem nenhum tratamento. Houve diferenças significativas nos valores de μSBS entre grupos de diferentes cimentos resinosos para condicionamento com HF e silano ou MBEP e cimento resinoso (independentemente da aplicação do adesivo ou não) em superfícies cerâmicas. No segundo estudo, o foco principal foi a utilização ou não de uma camada de adesivo extra após a aplicação do silano e não foram encontradas diferenças com o uso ou não deste passo clínico. O polifluoreto de amônio (MBEP) parece ser estável e comparável ao protocolo convencional com ácido fluorídrico e silano em uma superfície de cerâmica à base de silicato de lítio reforçado com zircônio e a aplicação ou não de uma camada adesiva extra não influenciou positiva ou negativamente na resistência de união final entre um cimento resinoso fotopolimerizável e a superfície de cerâmica à base de sílica.

Palavras-chave: Cerâmica. Cimentos Dentários. Resistência ao Cisalhamento.

ABSTRACT

Influence of different simplified surface treatments protocols on the bond strength between resin cement agents and glass-based ceramics

A correct surface treatment protocol for ceramics is a very important step in the cementation process and it may influence the final bond strength performance.

In these two studies, the aim was to evaluate the influence of simplified silica-based ceramic surface treatments on the shear bond strength with resin cements.

Zirconium-reinforced lithium silicate and lithium disilicate slices were obtained and submitted to different surface treatments protocols. Hydrofluoric acid (HF) and ammonium polyfluoride (MBEP) were used in both studies for topographic surface alteration and then combined with different adhesive materials and protocols for a possible potentialization of the final bond strength. One of the studies used thermocycling before the microshear bond strength (μSBS) evaluation. Failure mode was analyzed with a stereoscopic loupe. In the first study, groups that used HF and MBEP resulted in a significantly higher μSBS than did the group without any treatment. There were significant differences in μSBS values between groups of different resin cements for HF-etched and silane or MBEP and resin cement (regardless of adhesive application) to ceramic surfaces. In the second study, the main focus were the utilization or not of an extra adhesive layer after silane application and there were not found differences with the use or not of this extra clinical step. Ammonium polyfluoride (MBEP) seems to be stable and comparable to the conventional protocol with hydrofluoric acid and silane on a ceramic surface based of zirconium-reinforced lithium silicate and the application or not of an extra adhesives layer did not influence positive or negatively the final bond strength between a light cured resin cement and the silica-based ceramic surface.

Keywords: Ceramics. Dental Cements. Shear Strength.

LIST OF ILLUSTRATIONS

- ARTICLE 1

- ARTICLE 2

LIST OF TABLES

- ARTICLE 1

- ARTICLE 2

TABLE OF CONTENTS

1 INTRODUCTION

1 INTRODUCTION

Nowdays, dental ceramics have an important place in cosmetic dentistry. Because of their nonmetallic, biocompatible, and esthetic properties, an increasing number of all-ceramic materials and systems are avaliable for clinical use. (CONRAD et al., 2007). Monolithic CAD-CAM materials are becoming more popular between clinicians because of the simplification on their processing. (COLDEA et al., 2013) Among these systems; Highly aesthetic dental ceramics are predominantly vítreous and ceramics materials with greater mechanical properties are generally polycristalline. (KELLY et al., 2008 e GRACIS et al., 2015) One of the vitreous ceramic most known and used is the lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) because of its high flexural strength, chemical stability, biocompability and a large amount of indications (anterior or posterior crowns, veneers, onlays, inlays and implant crowns). (RAUCH AH et al., 2018)

Recently introduced to the market, the monolithic CAD-CAM block of zirconia-reinforced lithium silicate (ZLS) is a clear example of evolution, since its proposes to mix an excellent aesthetics and very interesting mechanical properties, thanks to the chemistry structure behind this new material. (ELSAKA et al., 2016) Structurally, the ZLS block is composed of lithium metasilicate and zirconium dioxide, thus achieving a greater and correct indication for both anterior and posterior regions as well. (TRAINI et al., 2016).

In order to obtain success and longevity in treatments employing dental ceramics, it is necessary that the prosthetic pieces are properly adhered to both dental structure and resin cement. (DELLA BONA et al., 2008). Due to the differences with the ceramic's composition and microstructure, cementation protocol changes and the treatment of the inner surface varies greatly. The bonding of ceramics to tooth structure is very well researched, studied and documented, yielding strong, predictable and durable bonds. (PEUMANS et al., 2007)

Bond strength to ceramics relies on both chemical bonding and micromechanical interlocking to the ceramic surface. Common surface treatment methods include sandblasting with alumina, grinding, abrasion with diamond instruments and acid etching. (with HF solution or ammonium polyfluoride) (CHEN et al., 1998 & WOLF et al., 1992) Application of a silane coupling agent to pretreated ceramic surfaces provides a strong chemical covalent bond between the hydroxyl group of silanol (from the silane coupling agent) and that of the silica (from ceramics) (BARGHI et al., 2000). This chemical process guaranteed wettability between the resin cement and the pre-treated ceramic surface.

In general, this protocol is time consuming and generates some doubts between clinicians. Therefore, new alternative materials for shortened clinical protocols for ceramic surface conditioning are being studied. A single-step self-etching primer (Monobond Etch and Prime) has gained attention on the market. It claims to be a faster option for ceramic surface conditioning. This material could be a save time product reducing the conventional two-step approach to a one-step glass ceramic conditioning approach. (MAIER et al., 2019)

Finally, there always exist the doubt about the use or not of an extra adhesive layer for a better modification of the viscosity and wettability of the resin-based material for cementation. (NAVES et al., 2010) Withal, this particular step is still controversial in the literature. (NOGUEIRA et al., 2020) In this context, the aim of these two researches was to evaluate the bond strength between two glass-based ceramics systems and resin cement agents, using different simplified bonding protocols.

2 ARTICLES

2 ARTICLES

2.1 ARTICLE 1

Article formatted according to Operative Dentistry

Effect of the simplification of different surface treatments on the bond strength between two resin cements and a zirconium-reinforced lithium silicate ceramic.

Effect of the simplification of different surface treatments on the bond strength between two resin cements and a zirconium-reinforced lithium silicate ceramic.

Clinical Relevance

The simplified surface treatment with ammonium polyfluoride and without hydrofluoric acid etching can be used for cementing zirconium-reinforced lithium silicate ceramics. The use of a zirconia primer is not recommended for zirconium-reinforced lithium silicate ceramics.

SUMMARY

Objective: To evaluate the influence of simplified ceramic surface treatments on the shear bond strength of two resin cements to a zirconium-reinforced lithium silicate material.

Methods and Materials: Ninety zirconium-reinforced lithium silicate slices of 1.5mm thickness were obtained and submitted to different surface treatments protocols. Hydrofluoric acid (HF) and ammonium polyfluoride (MBEP) were used for surface treatment and then combined with different adhesive materials: Monobond N, Prosil, Ambar Universal, Signum Zirconia Bond, AllCem cement, and Multilink Automix cement. Resin composite cylinders made with Vitra APS were bonded to zirconium-reinforced lithium silicate with one of the evaluated protocols and were subjected to 6.000 cycles of thermocycling from 5ºC to 55ºC and a dwell time of 30 seconds at each temperature before the microshear bond strength (µSBS) evaluation. Failure mode was analyzed with a stereoscopic loupe. Statistical analyses were performed with one-way ANOVA and Tukey's HSD test (α=0.05). Bartlet test was used to verify the homogeneity of variances.

Results: Groups that used HF and MBEP resulted in a significantly higher µSBS than did the group without any treatment ($p<0.0001$). There were significant differences in µSBS values between groups of different resin cements (AllCem or Multilink Automix) for HF-etched and silane or MBEP and resin cement (regardless of adhesive application) to ceramic surfaces. The failure mode was adhesive for all specimens. Considering the protocol simplification factor, there was no significant differences on applying or not the adhesive layer on all groups.

Conclusions: Ammonium polyfluoride (MBEP) seems to be stable and comparable to the conventional protocol with hydrofluoric acid and silane on a ceramic surface based of zirconium-reinforced lithium silicate

INTRODUCTION

Recently introduced to the market, the monolithic CAD-CAM block of zirconia-reinforced lithium silicate (ZLS) is a clear example of evolution, since it proposes to mix the excellent optical properties of the predominantly vitreous ceramics reinforced with particles and the excellent mechanical properties of polycrystalline ceramics.[1] Structurally, the ZLS block is composed of lithium metasilicate and zirconium dioxide, thus achieving a greater and correct indication for both anterior and posterior regions.[2] In addition to these characteristics, this new material continues to be considered as acid sensitive due to the predominantly vitreous content (8 to 15% of Y-TZP in the composition).[3,4,5] This search of improvement on ceramics chemical composition and faster techniques of processing could be the reason for clinicians search for shortened and simplified clinical protocols, leading to gain time with fewer steps but focus on ensuring a correct adhesive interaction between the ceramic, the dental substrate and the cementing agent.[6]

Already studied, it is the reaction that exists between the hydrofluoric acid (HF) and the silica present in the glassy matrix of the conditioned ceramic systems; resulting in a topographic alteration of the surface, which would act as micromechanical retention to the cementing agent.[7] However, hydrofluoric acid concentration, the adequate etching time and problems associated with an over-conditioned surface and a possible negative effect on the mechanical properties of the ceramic material[7,8,9] are still controversial in the literature, besides its possible systemic toxic effect.[10]

Therefore, new alternative materials for shortened clinical protocols for ceramic surface conditioning are being studied. Among them, the introduction of a single-step self etching ceramic primer, (Monobond Etch and Prime) that claims to be a substitute for hydrofluoric acid and silane surface treatment. This new material could be a save time product reducing the conventional two-step approach to a one-step glass ceramic conditioning approach, with an easy protocol for use and a possible minimization of the potential toxic effects associated to HF acid.[11]

A different attempt of simplification could be performed trying to take advantage of the 8-15% of Y-TZP present in the ZLS composition and the possible reaction with different specifics zirconia primers (Signum Zirconia Bond) and universal adhesives based on 10-methacryloxydecyl dihydrogen phosphate (10-

MDP). It is known that this functional monomer is necessary for obtaining chemical adhesion with polycrystalline materials, and because they do not have as much sensitivity of the technique for their application, it would benefit to the clinical process.[12,13] Thus, the aim of this study was to evaluate the effects of the simplification of the different surface treatment protocols on the bond strength between two resin cements to a zirconia reinforced lithium silicate ceramic. The null hypothesis evaluated was that there would not be difference on the microshear bond strength produced by the technique simplification and the standard surface treatments protocols.

MATERIAL AND METHODS

The materials used in the study are described in Table 1.

SPECIMEN PREPARATION

Zirconium lithium silicate blocks (ZLS) (Celtra Duo, Dentsply Sirona, Bensheim, Germany) were sectioned in a cutting machine (Isomet 1000 Low Speed, Buehler, LakeBluff, IL, USA) with a diamond disc (15LC diamond No. 11-4254, Buehler, LakeBluff, IL, USA) at a speed of 300 rpm under constant water cooling to obtain 1.5-mm-thick slices (3.5 X 3.0 X 0.15mm). Slices were polished with sandpapers #800, #1000, and #1200 (K2000 Polishing Paper, Exact, Nordestedt, Scheleswing-Holstein, Germany) on a metallographic polishing machine (Exact, Nordestedt, Schleswing-Holstein, Germany) to standardize ceramic surfaces. These slices were then cut in four equal parts with a diamond disc (Mono Face Ref. 7010, KG Sorensen, Cotia, São Paulo, SP, Brazil) under constant water cooling. Afterwards, all specimens were sintered following the manufacturer's protocol with a temperature of 840°C for 8 minutes in a specific oven (Programat EP510, Ivoclar Vivadent, Schaan, Liechstenstein).

Next, resin composite cylinders (Vitra APS, FGM, Joinville, SC, Brazil) were made using surgical catheters with an internal diameter of 1.40 mm and 1 mm height and an LED device (VALO Cordless, Ultradent), operating in standard mode with an irradiance of 1.000mW/cm^2. After 10 minutes, the surgical catheters were removed with a No. 11 scalpel blade (Embramed, Jurubatuba, SP, Brazil) to expose the resin composite cylinders.

SURFACE TREATMENTS

After sintering, the specimens were randomly divided into 9 groups (n = 10). Afterwards, the conditioning of the surface corresponding to each group was carried out (Table 2) according to the guidelines of each manufacturer. For the groups that used hydrofluoric acid, 10% Condac Porcelana (FGM, Joinville, SC, Brazil) was applied for 20 seconds. Next, the acid was removed with copious air and water for 60 seconds. For the MBEP groups, Monobond Etch and Prime (Ivoclar Vivadent, Schaan, Liechstenstein) was actively applied with a microbrush for 20 seconds and allowed to react on the surface for another 40 seconds. The product was then removed with copious air and water for 10 seconds. Next, all the ceramic surfaces already treated received the application of the adhesives, metal primers and resin cements following the manufacturer's specifications (Table 2).

CEMENTATION PROCEDURE

For the cementation procedures, the resin composite cylinders were cemented onto the previously conditioned ceramic surfaces. Therefore, in order to attempt the most realistic clinical conditions, it was used a metallic device for photoactivation of the composite resin cylinders threw ceramic, trying to emulate a real clinical scenario (Figure 1). Two resin cements; AllCem (FGM, Joinville, SC, Brazil) and Multilink Automix (Ivoclar Vivadent, Schaan, Liechstenstein) were used according to the manufacturer's specifications and groups selection (Table 2). After removal of the excesses with the microbrush, photoactivation was carried out using an LED device (VALO Cordless, Ultradent Products, South Jordan, UT, USA) with an irradiance of 1.000mW/cm^2. After cementation, the specimens were stored for 48 hours in deionized water. Subsequently, all specimens were aged before the bond strength test. This was done by 6.000 cycles of thermocycling from 5ºC to 55ºC and a dwell time of 30 seconds at each temperature.

BOND STRENGTH EVALUATION

After thermocycling, the bond strength was evaluated on a universal testing machine (Instron 3342, Illinois Tool Works, Norwood, MA, USA). A 0.2-mm wire loop device was used to apply a shear stress as close as possible to the bonding interface at a speed of 0.5mm/min until breaking. The force measurement during the test was done through a load cell with a capacity of 50 kg (500 N). The bond strength was expressed in MPa, calculated by the dividing the maximum force in Newtons by the bonding area in mm^2. After performing the micro-shear evaluation, the surfaces of the specimens were analyzed in a stereoscopic loupe to determine the type of failure involved. The failures were classified as adhesive, cohesive or mixed.

STATISTICAL ANALISYS

After verifying the normality and homogeneity of variances, data were analyzed through one-way Anova and the Tukey's HSD. The significance level was set at 5%.

RESULTS

The mean bond strength and standard deviations of each group are detailed in Table 3. For group NT+SZB+RC, which used a zirconia primer (SZB) and did not receive any previous surface treatment (HF or MBEP) or any silane application (Monobond N or Prosil) or adhesive layer (Ambar Universal), mean μSBS were closed to zero. The group MBEP+S+RCAM showed the highest μSBS values where the use of MBEP was highlighted in conjunction with the silane agent Monobond N and Multilink Automix resin cement; however, it did not differ statistically from group HF+S+RCAM which used the standard surface treatment with fluoridric acid, silane and the Multilink Automix resin cement. Grupos that used Multilink Automix as dual resin cement agent resulted in a significantly higher mean μSBS than did All Cem resin cement. There were significant differences in μSBS values between groups of different resin cements for HF-etched and silane or MBEP and resin cement (regardless of adhesive application) to ceramic surfaces. The failure mode was adhesive for all specimens. Considering the protocol simplification factor, there was no significant differences on applying or not the adhesive layer on all groups.

DISCUSSION

The null hyphotesis, that the μSBS of the simplified surface treatment with MBEP does not significantly differ from the μSBS of the standard surface treatment with HF and silane, cannot be rejected. In the present study, the different groups that used the ammonium polyfluoride self-etching ceramic primer attained a mean μSBS that did not statiscally differ from those that used hydrofluoric acid and silane treatment.

It was also observed whether the use of different products containing 10-MDP (Ambar Universal and Signum Zirconia Bond Primer) would positively or negatively influence the bond strength of two resin cements (AllCem and Multilink Automix) to a zirconium-reinforced lithium silicate ceramic (ZLS). The purpose of these associations in Groups HF-S-UA-RC, MBEP-UA-RC, NT-SZB-RC, MBEP-SZB-RC and HF-S-SZB-RC was to try to potentiate the bond strength through a possible interaction of the bifunctional monomer 10-MDP and the polycrystalline part of the ZLS block. In other studies[14,15,13], is well emphasized the importance of the chemical interaction of the 10-MDP monomer with the Y-TZP. However, due to the higher amount of vitreous matrix in the composition of the ZLS block, it does not appear to react in the same way with this bi-functional monomer.

The action mechanism of hydrofluoric acid is very well detailed in the literature. It reacts with the silica, eliminating it selectively, thus achieving the exposure of the crystalline structure. After obtaining a change in surface topography, the ceramics can be functionalized with silane and infiltrated with some adhesive material. [16-19] This classic acid agent embrace some problems reported in the literature. These includes high toxicity, corrosive potential, possible burns on skin contact, possible absorption into the bloodstream through interaction with skin and bone tissue and irreversible damage if there is eye contact.[20,21,10] In addition to the sensitivity of the technique as still being questioned, with relation to the agent concentration and the time of action on the ceramic surface, and if this could be detrimental to the mechanical properties of the material.[22-24]

Therefore, Monobond Etch and Prime (MBEP) appear to be an alternative material that could minimize some of these problems. MBEP is a product that combines ammonium polyfluoride (less aggressive acid agent) and silane (trimethoxypropyl methacrylate) in a single bottle, thus reducing the number of steps and the clinical time used on the conventional technique (HF+S).[25,26] There are still few reports in the literature of the association of MBEP and zirconia reinforced lithium silicate (ZLS) ceramics. Other authors have found encouraging results reagarding the association of MBEP with other ceramic materials. Prado and others[27]; compared the two protocols (HF+S or MBEP) and concluded that there was no significant difference in surface treatments with lithium disilicate and feldspathic ceramics and the durability of the bond strength after aging by thermocycling was more stable for the MBEP. In the study of El-Damanhoury and Gaintantzopoulou[28], it was also concluded that the MBEP is efficient and thus, a dependent ceramic material, acting better on surfaces of lithium dissilicate and feldspathic ceramics. Also, in the study by Wille and others[29]; no significant differences were found in the microtensile bond strength with the use of the conventional method (HF + S) and MBEP on the surface of lithium disilicate.

In the conduction of the present study there were a few points to be addressed, the cementation procedure was conducted indirectly, with the influence of the ceramic thickness (1.5-mm) between the light source and the cemented resin composite cylinder. It is known that the influence of a ceramic material between the light source and the resin cement has the effect of decreasing the degree of conversion and the hardness of the resin agent. [30] For this purpose, it was used a metallic device with a disk hole with a compatible width of the active tip of the LED device. This appliance supports the ceramic slice and allowed the passage of light only from below of it. This does not allow the directly light activation of the resin cement, more accordingly to the clinical reality. [31]

In the present study, thermocyling procedure was performed to age the adhesive interface. Water storage time seems to affect the bond strength produced by the different surface treatment protocols. This exposure of the adhesive interface to an aggressive method seeks to degrade the possible bond strength. Correct and sufficient exposure is necessary to achieve the saturation of water with the resin or resin cement. Therefore, 6,000 thermocycling cycles were programmed from 5 ° C to 55 ° C as recommended by ISO (ISO / TS 11405 2015). [32]

One of this study's limitations was the debonding of the specimens before the mechanical test . These were much more observed in Group NT+SZB+RC which did not used any form of prior etching, relying in the possible interaction between the zirconia specific primer (SZB) and the polycrystalline content of the CAD-CAM block used. Therefore, it could be concluded that the Signum Zirconia Bond alone, did not have a correct interaction with the ceramic surface. Moreover, future studies are needed to understand if the protocol simplification would have a correct interaction between the MBEP, different adhesives and primers, different types of resin cements and other commercially available ceramics. As well, it could be important to test this different materials with more long term water storage and thermocycling periods.

CONCLUSION

It may be concluded that the conventional pre-treatment protocol; with hydrofluoric acid and silane and the more shortened protocol with the Monobond Etch and Prime on a ceramic surface based on zirconium-reinforced Lithium Silicate seems to be an effective simplified alternative.

REFERENCES

1. Elsaka SE, & Elnaghy AM (2016) Mechanical properties of zirconia reinforced lithium silicate glass-ceramic *Dental Materials* 32(7) 908-914.

2. Traini T, Sinjari B, Pascetta R, Serafini N, Perfetti G, Trisi P, & Caputi S (2016) The zirconia-reinforced lithium silicate ceramic: lights and shadows of a new material *Dental Materials Journal* 35(5) 748-755.

3. Sato TP, Anami LC, Melo RM, Valandro LF, & Bottino MA (2016) Effects of Surface Treatments on the Bond Strength Between Resin Cement and a New Zirconia-reinforced Lithium Silicate Ceramic *Operative Dentistry* 41(3) 284-292.

4. Bömickea W, Rammelsbergb P, Krisamc J, & Ruesd S (2019) The Effects of Surface Conditioning and Aging on the Bond Strength Between Composite Cement and Zirconia- reinforced Lithium-Silicate Glass-Ceramics *The Journal of Adhesive Dentistry* 21(6) 567-576.

5. Barchetta N, Amaral M, Prochnow C, Rodrigues F, Bottino M, Valandro, & Marques de Melo R (2019) Strength of a Zirconia-Reinforced Lithium Silicate Ceramic: Acid-Etching Time and Resin Cement Application Effects *The International Journal of Periodontics & Restorative Dentistry* 39(3) 431-437.

6. Silva LH, Lima E, Miranda RBP, Favero SS, Lohbauer U, & Cesar PF (2017) Dental ceramics: a review of new materials and processing methods *Brazilian Oral Research* 31(5) 133-146.

7. Yen TW, Blackman RB, & Baez RJ (1993) Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic *The Journal of Prosthetic Dentistry* 70(3) 224-233.

8. Della Bona A, Anusavice K, & Hood JA (2002) Effect of ceramic surface treatment on tensile bond strength to a resin cement *International Journal of Prosthodontics* 15(3) 248-255.

9. Della Bona A, & Van Noort R (1995) Shear *vs*. tensile bond strength of resin composite bonded to ceramic *Journal of Dental Research* 74(9) 1591-1596.

10. Ozcan M, Allahbeickaraghi A, & Dündar M (2012) Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review *Clinical Oral Investigations* 16(1) 15-23.

11. Maier E, Bordihn V, Belli R, Taschner M, Petschelt A, Lohbauer U, & Zorzin J (2019) New Approaches in Bonding to Glass-Ceramic: Self-Etch Glass-Ceramic Primer and Universal Adhesives *The Journal of Adhesive Dentistry* 21(3) 209-217.

12. Hanabusa M, Yoshihara K, Yoshida Y, Okihara T, Yamamoto T, Momoi Y, & Van Meerbeek B (2016) Interference of functional monomers with polymerization efficiency of adhesives *European Journal Oral of Science* 124(2) 204-209.

13. Llerena-Icochea AE, Costa RM, Borges A, Bombonatti J, & Furuse AY (2017) Bonding Polycrystalline Zirconia With 10-MDP–containing Adhesives *Operative Dentistry* 42(3) 335-341.

14. Yoshida K, Tsuo Y, & Atsuta M (2006) Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler *Journal of Biomedical Materials Research Part B: Applied Biomaterials* 77(1) 28-33.

15. Ozcan M, Nijhuis H, & Valandro LF (2008) Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia after thermal aging *Dental Materials Journal* 27(1) 99-104.

16. Stangel I, Nathanson D, & Hsu CS (1987) Shear strength of the composite bond to etched porcelain *Journal of Dental Research* 66(9) 1460-1465.

17. Horn HR (1983) Porcelain laminate veneers bonded to etched enamel *Dental Clinics of North America* 27(4) 671-684.

18. Shahverdi S, Canay S, Sahin E, & Blige A (1998) Effects of different surface treatment methods on the bond strength of composite resin to porcelain *Journal of Oral Rehabilitation* 25(9) 699-705.

19. Tian T, Tsoi JK, Matinlinna JP, & Burrow MF (2014) Aspects of bonding between resin luting cements and glass ceramics materials *Dental Materials* 30(7) 147-162.

20. Bertolini JC (1992) Hydrofluoric acid: a review of toxicity *The Journal of Emergency Medicine* 10(2) 163-168.

21. Blatz MB, Sadan A, & Kern M (2003) Resin-ceramic bonding: A review of the literature. *The Journal of Prosthetic Dentistry* 89(3) 268-274.

22. Menees TS, Lawson NC, Beck PR, & Burgess JO (2014) Influence of particle abrasion or hydrofluoric acid etching on lithium disilicate flexural strength *The Journal of Prosthetic Dentistry* 112(5) 1164-1170.

23.Zogheib LV, Bona AD, Kimpara ET, & McCabe JF (2011) Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. *Brazilian Dental Journal* 22(1) 45-50.

24. Hooshmand T, Parvizi S, & Keshvad A (2008) Effect of surface acid etching on the biaxial flexural strength of two hot-pressed glass ceramics *Journal of Prosthodontics* 17(5) 415-9.

25. Román-Rodríguez JL, Perez-Barquero JA, Gonzalez-Angulo E, Fons-Font A, & Bustos-Salvador JL (2017) Bonding to silicate ceramics: Conventional technique compared with a simplified technique *Journal of Clinical and Experimental Dentistry* 9(3) 384-386.

26. Dapieve K, Machry R, Pilecco R, Kleverlaan C, Pereira G, Venturini A, & Valandro L (2020) One-step ceramic primer as surface conditioner: Effect on the

load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations *Journal of the Mechanical Behavior of Biomedical Materials* 10(4) 1-8.

27. Prado M, Prochnow C, Marchionatti AME, Baldissara P, Valandro LF, & Wandscher VF (2018) Ceramic Surface Treatment with a Single-component Primer: Resin Adhesion to Glass Ceramics *The Journal of Adhesive Dentistry* 20(2) 99-105.

28. El-Damanhoury HM, & Gaintantzopoulou MD (2018) Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance *Journal of Prosthodontic Research* 62(1) 75-83.

29. Wille S, Lehmann F, & Kern M (2017) Durability of Resin Bonding to Lithium Disilicate and Zirconia Ceramic using a Self-etching Primer *The Journal of Adhesive Dentistry* 19(6) 491-496.

30. Watanabe H, Kazama R, Asai T, Kanaya F, Ishizaki H, Fukushima M, & Okiji T (2015) Efficiency of dual-cured resin cement polymerization induced by high-intensity LED curing units through ceramic material Operative Dentistry 40(2) 153-162.

31. Calgaro PA, Furuse AY, Correr GM, Ornaghi BP, & Gonzaga CC (2013) Influence of the interposition of ceramic spacers on the degree of conversion and the hardness of resin cements Brazilian Oral Reserch 27(5) 403-409.

32. ISO/TS 11405 (2003) Testing of adhesion to tooth structure Geneva: International Organization for Standardization *Dental Materials.*

FIGURES

Figure 1 - Metallic Device for Photoactivation of composite resin cylinders threw ceramic.

TABLES

Table 1 - – Materials used and their main characteristics.

Material	Manufacturer	Composition
Ambar Universal	FGM	Active Ingredients: MDP (10-Methacryloyloxydecyl dihydrogen phosphate), methacrylic monomers, photoinitiators, coinizers and stabilizer. Inactive Ingredients: inert load (silica nanoparticles) and vehicle (ethanol).
All Cem	FGM	Base paste: TEGDMA, BisEMA, BisGMA, camphoroquinone barium-aluminum-silicate microglass, silica nanoparticles; Catalyst paste: methacrylic monomers, dibenzoyl peroxide and stabilizers, barium-aluminum-silicate microparticles of 66-67 wt% over the mixture.
Celtra Duo	Dentsply	85% Lithium Metasilicate, 15% Zirconium dioxide
Signum Zirconia Bond	Heraeus Kulzer	Signum zirconia bond I: acetone, bifunctional components based on methacrylate. Signum zirconia bond II: MMA, initiators.
Prosil	FGM	3-Methacryloxypropyltrimethoxysilane tenor. <5% Tenor Ethanol >85% Water <10%
10% Condac Porcelain	FGM	Hydrofluoric acid at 4, 5 or 10%, water, thickener, surfactant and dye.
Monobond Etch & Prime	Ivoclar Vivadent	Ammonium polyflouride, trimethoxypropyl methacrylate, alcohols, water
Multilink Automix	Ivoclar Vivadent	Dimethacrylate, HEMA (2-hydroxyethyl methacrylate), barium cement composite glass, Ba-Al-fluorosilicate glass, ytterbium trifluoride, silica, catalysts, stabilizers, pigments
Monobond N	Ivoclar Vivadent	Ethanol, 3-trimethoxysilylpropyl methacrylate, methacrylated phosphoric acid ester (10-MDP), disulfide acrylate

Table 2 - Groups and surface treatment protocols

Groups	Surface Treatment Protocol
Hydrofluoric Acid (HF) + Silane (S) + Ambar Universal (UA) + All Cem Dual Resin Cement (RC)	1. Apply the hydrofluoric acid to the surface to be treated for 20 seconds. 2. Wash and dry the surface for 20 seconds. 3. Application of silane for 60 seconds. 4. Application of adhesive agent. 5. Photoactivation for 20 seconds.
Monobond Etch and Prime (MBEP) + Ambar Universal (UA) + All Cem Dual Resin Cement (RC)	1.Wash and dry the surface. 2.Actively apply the MBEP for 20 seconds. 3.Wait 40 seconds. 4. Wash and dry the surface for 10 seconds. 5.Application of the adhesive. 6.Photoactivation for 20 seconds.
Monobond Etch and Prime (MBEP) + All Cem Dual Resin Cement (RC)	1. Wash and dry the surface. 2. Actively apply the MBEP for 20 seconds. 3. Wait 40 seconds. 4. Wash and dry the surface for 10 seconds.
No Treatment (NT) + Signum Zirconia Bond (ZSB) + All Cem Dual Resin Cement (RC)	1. Wash and dry the surface. 2. Application of SZB I and wait to evaporate for 15 seconds. 3. Application of SZB II on the ceramic surface for 20 seconds. 4. Photoactivation for 20 seconds.
Monobond Etch and Prime (MBEP) + Signum Zirconia Bond (ZSB) + All Cem Dual Resin Cement (RC)	1. Wash and dry the surface. 2. Actively apply the MBEP for 20 seconds. 3. Wait 40 seconds. 4. Wash and dry the surface for 10 seconds. 5. Application of SZB I and wait to evaporate for 15 seconds. 6. Application of SZB II on the ceramic surface for 20 seconds. 7. Photoactivation for 20 seconds.

Hydrofluoric acid (HF) + Silane (S) + Signum Zirconia Bond (SZB) + All Cem Dual Resin Cement (RC)	1. Apply the hydrofluoric acid to the surface to be treated for 20 seconds. 2. Wash and dry the surface for 20 seconds. 3. Application of silane for 60 seconds. 4. Application of SZB I and wait to evaporate for 15 seconds. 5. Application of SZB II on the ceramic surface for 20 seconds. 6. Photoactivation for 20 seconds.
Monobond Etch and Prime (MBEP) + Multilink Automix Dual Resin Cement (RCAM)	1. Wash and dry the surface. 2. Actively apply the MBEP for 20 seconds. 3. Wait 40 seconds. 4. Wash and dry the surface for 10 seconds. 5. Application of cement.
Monobond Etch and Prime (MBEP) + Monobond N (S) + Multilink Automix Dual Resin Cement (RCAM)	1. Wash and dry the surface. 2. Actively apply the MBEP for 20 seconds. 3. Wait 40 seconds. 4. Wash and dry the surface for 10 seconds. 5. Application of Monobond N for 60 seconds. 6. Application of cement.
Hydrofluoric Acid (HF) + Monobond N (S) + Multilink Automix Dual Resin Cement (RCAM)	1. Apply the hydrofluoric acid to the surface to be treated for 20 seconds. 2. Wash and dry the surface for 20 seconds. 3. Application of Monobond N for 60 seconds. 4. Application of cement.

Table 3 - Mean values and standard deviations (in parenthesis) of bond strength for the different groups. Different letters mean statistically significant differences ($p < 0.05$).

Group	**Bond Strength (Mpa)**
HF+S+UA+RC	12.7 (6.1) b
MBEP+UA+RC	10.6 (7.0) b
MBEP+RC	14.6 (5.6) bc
NT+SZB+RC	0.4 (0.5) a
MBEP+SZB+RC	8.6 (5.7) ab
HF+S+SZB+RC	15.8 (7.3) bc
MBEP+RCAM	22.0 (3.5) cd
MBEP+S+RCAM	29.5 (10.0) d
HF+S+RCAM	27.7 (5.3) d

2.2 ARTICLE 2

Influence of the use of an adhesive layer on the bond strength between a light cured resin cement and two etchable ceramic CAD-CAM materials.

SUMMARY

Objective: To evaluate the influence of the used of an extra adhesive layer on the shear bond strength of one resin cement between two glass-based ceramics.

Methods and Materials: Sixty zirconium-reinforced lithium silicate and sixty lithium disilicate slices of 1.0mm thickness were obtained and submitted to different surface treatments and adhesive protocols. Hydrofluoric acid (HF) associated with a silane agent (RelyX Ceramic Primer) or the one step ceramic primer, ammonium polyfluoride (MBEP) were used for surface treatment and then associated with the application or not of an adhesive layer of two bonding agents: All Bond Universal and Scothbond Multipurpose (Hydrophobic part) Cylinders made with Variolink Esthetic LC resin cement were bonded to both glass-based ceramic surfaces with one of the evaluated protocols and subjected to the microshear bond strength (μSBS) evaluation. Failure mode was analyzed with a stereoscopic loupe. Statistical analyses were performed with two-way ANOVA and Tukey's HSD test (α=0.05). Bartlet test was used to verify the homogeneity of variances.

Results: There were significant differences between adhesive protocols on the Lithium Disilicate groups (F=8.9708; p<0.00001) and no significant differences between Zirconium Lithium Silicate groups (F=1,3797; p=0,24646). The failure mode was adhesive for all specimens. Considering the protocol simplification factor, there was no significant differences on applying or not the adhesive layer on all groups.

Conclusions: The application or not of an extra adhesive layer did not influence the bond strength between two glass-based CAD-CAM ceramic materials and the light cured resin cement.

INTRODUCTION

The constantly search for high strength and aesthethics indirect restorations have led to an evolution of all ceramic materials.[1] Monolithic CAD-CAM materials are becoming more popular between clinicians because of the simplification on their processing.[2] Among these systems, one of the most known and used is the lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) because of its high flexural strength, chemical stability, biocompability and a large amount of indications (anterior or posterior crowns, veneers, onlays, inlays and implant crowns).[3]

Nowadays, a newly monolithic block has gained attention in the dental industry. It is the zirconia-reinforced lithium silicate (ZLS) and it is a clear example of new developments in dentistry, since it proposes to mix the excellent optical properties of the predominantly vitreous ceramics reinforced with particles and the excellent mechanical properties of polycrystalline ceramics.[4] Thanks to this particular structure the ZLS block can be indicated for anterior and posterior indirect restorations.[5]

Due to the glass-based main composition of these two ceramic systems, they are considered as acid sensitive. Therefore, it is very well described in the literature the standard procedures and the reaction that exists between the hydrofluoric acid and the ammonium polyfluoride with the silica present in the glassy matrix of the ceramic surface; enhancing a topographic alteration, which would act as a micromechanical retention to the cementing agent. [6,7,8,9,10]

In addition, because of its bifunctional characteristics, an application of silane coupling agent provides a strong chemical covalent bond between the hydroxyl group of silanol and silica between the methacrylate groups of the resin through siloxane bonds. [11,12,13] This chemical process guaranteed wettability between the resin cement and the pre-treated ceramic surface.

Application of an adhesive layer after silane application is a very common conduct between clinicians. This additional step and function of the adhesives remains controversial in the literature[14], since a few studies suggest that only with the association of surface topography alteration[15] (hydrofluoric acid or ammonium polyfluoride) and chemical adhesion promoted by the silane coupling agent is quite enough to guarantee a durable final bond strength with the resin cement.[16] Thus, the objective of the present study is to evaluate if the reduction of this step compromise or not the bond strength between one light cure resin cement and two glass-based ceramic systems.

The null hypotheses evaluated were: 1) the application or not of an extra adhesive layer would not influence the microshear bond strength; 2) there would be no differences between the surface treatment protocols and the two glass-based ceramics.

MATERIAL AND METHODS

The materials and compositions used in this study are described in Table 1.

SPECIMEN PREPARATION

The CAD-CAM blocks, lithium disilicate (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechstenstein) and Zirconium lithium silicate (Celtra Duo, Dentsply, Sirona, Bensheim, Germany) were sectioned in a cutting machine (Isomet 1000 Low Speed, Buehler, LakeBluff, IL, USA) with a diamond disc (15LC diamond No. 11-4254, Buehler, LakeBluff, IL, USA) at a speed of 300 rpm under constant water cooling. Thus 10 slices of 1.0mm thickness were obtained of each CAD CAM block (3.5 X 3.0 X 0.10mm). Next, these slices were polished through a # 800, # 1000 and # 1200 sandpapers sequence (K2000 Polishing Paper, Exact, Nordestedt, Scheleswing-Holstein, Germany) on a metallographic polishing machine (Exact, Nordestedt, Schleswing-Holstein, Germany), achieving a standardization of the ceramic surfaces. Then, these slices were cut in four equal parts with a single-phase manual-cut diamond disc (Mono Face Ref. 7010, KG Sorensen, Cotia, São Paulo, Brazil) under constant water-cooling. Afterwards, all specimens were sintered following the manufacturer's protocol.

The slices were then randomly divided in 6 groups for each CAD CAM block and embedded in acrylic resin (JET, Campo Limpo Paulista, São Paulo, Brazil). After that, all the slices were polished once again with sequential sandpaper discs (grit sizes #1000 to #1200; K2000 Polishing Paper, Exact, Nordestedt, Scheleswing-Scheleswing-Holstein, Germany) to remove any acrylic resin that might have covered the samples.

An acid resistant, double-sided adhesive tape (Scotch Permanent Double Sided Tape, 3M, St Paul, MN, USA) compatible with the CAD-CAM slices dimensions, was perforated with one 1.40mm diameter hole and laid over the ceramic surface. Twelve groups (n=10) were made using the following protocols:

SURFACE TREATMENT

Afterwards, the conditioning of the surface corresponding to each group was carried out according to the guidelines of each manufacturer. For the groups that used hydrofluoric acid (HF), 10% Condac Porcelain Etching Gel (FGM, Joinville, Santa Catarina, Brazil) was applied for 20 seconds. Next, the acid was removed with copious air/water spray for 60 seconds. For the groups that used ammonium polyfluoride, Monobond Etch and Prime (Ivoclar Vivadent, Schaan, Liechstenstein) was actively applied with a microbrush for 20 seconds and allowed to react on the surface for another 40 seconds. The product was then removed with copious air and water for 10 seconds. Afterwards, all the specimens were cleaned ultrasonically in distilled water for 4 mins.

SILANE SOLUTION

In all groups that used HF as surface treatment independent of the type of CAD CAM block (IPS e.max CAD or ZLS Celtra Duo) a prehydrolyzed silane-based primer was used. (Rely X Ceramic Primer, 3M, St Paul, MN, USA) It was applied with a microbrush and allowed to react for 60 seconds. Then, it was used a stream of air for the solvent evaporation. In the other hand, all the groups that used Monobond Etch and Prime (Ivoclar Vivadent, Schaan, Liechtensein) as a surface conditioner went threw the manufacters recommendations because of its self-etching single-component glass-ceramic nature.

ADHESIVE LAYER

For the Universal Adhesive (All Bond Universal, Bisco, Schaumburg, IL, USA), it was applied with an active application for 20 seconds and air-dried for 20 seconds to achieve the solvent evaporation. On the other half, it was used the hydrophobic part of Scotchbond Multi-purpose (3M, St Paul, MN, USA) and it was applied according to the manufacturer’s instructions. Adhesives were light cured for 10 seconds with an LED device (VALO Cordless, Ultradent Products, South Jordan, UT, USA) operating at an irradiance of 1000 mW/cm^2.

RESINOUS CEMENTATION

For the cementation process, surgical catheters with an internal diameter of 1.40 mm and a height of 1 mm were used for the resin cement cylinders (Variolink Esthetic LC, Ivoclar Vivadent, Schaan, Liechstenstein). One cylinder per ceramic quarter of surface was prepared, resulting in ten for each group. The resin cement was inserted into the catheters and after removal of the excesses with a microbrush, light-activation was carried out using a light-emitting device (VALO Cordless, Ultradent Products, South Jordan, UT, USA) with an intensity of 1000mW / cm^2. After light curing, the surgical catheters were kept untouched for 10 minutes before being removed with #11 scalpel blades (Embramed, Jurubatuba, SP, Brazil) to expose the resin cement cylinders. The samples were stored in deionized water for 24 hours at 37°C.

BOND STRENGTH EVALUATION

All specimens were submitted to the universal test machine micro-shear test (Instron 3342, Illinois Tool Works, Norwood, MA, USA). The force measurement during the test was done through a load cell with a capacity of 50 kg (500 N). A 0.2mm diameter steel wire device was used at a speed of 0.5mm/min until breaking. The bond strength was expressed in MPa, calculated by the dividing the maximum force in Newtons by the bonding area in mm^2.

FAILURE TYPE ANALYSIS

After performing the micro-shear test, the surfaces of the specimens were then analyzed in a stereoscopic loupe to determine the type of failure involved. The failures were classified as adhesive, cohesive or mixed.

STATISTICAL ANALYSIS

Bond strength data were submitted to the Bartlet test to verify the homogeneity of variances. Data were analyzed through two-way A global significance level of 5% was adopted.

RESULTS

Mean values of the bond strength and standard deviation of each group are summarized in Table 2. Two-way ANOVA test revealed significant differences between protocols on the Lithium Disilicate groups HF+SIL+AMP / HF+SIL+AU (F=8.9708; p<0.00001) compared to the others (MBEP+AMP/ MBEP+AU/ HF+SIL/ MBEP). On the other hand, there were no significant differences between Zirconium lithium silicate groups (F=1,3797; p=0,24646). The highest µSBS values on the Lithium Disilicate group were obtained with the MBEP+AU surface protocol and on the Zirconium lithium silicate group were obtained with the MBEP surface protocol alone. With the extra adhesive layer application or not, results showed no statistical differences between these study factor in all groups of the both ceramic surfaces. The failure mode was adhesive for all specimens.

DISCUSSION

The first null hypothesis was accepted since no statistically significant difference was found between the application or not of an extra adhesive layer. The second null hypothesis was rejected, since there were differences between surface treatments protocols and ceramics used in the study. In the present study, the choice of the two glassy matrix ceramics was based on the well-known and reliable surface treatments protocols described in the literature, as these two systems are considered as acid sensitive.[17, 18, 19] Therefore, the need for hydrofluoric acid and silane or ammonium polyfluoride treatments before the application of the resin-bonding agents are very important.[20, 21]

Monobond etch and prime is a product that combines a less aggressive acid agent (ammonium polyfluoride) and silane (trimethoxypropyl methacrylate) in a single bottle, thus reducing the number of steps and the clinical time used on the conventional technique (hydrofluoric acid + silane). [22,23]

After the ceramic surface modification, a very common clinical step between the clinicians looking to enhance the bond strength is the use of an adhesive layer attending for the modification of the viscosity and wettability of the resin-based material for cementation. [24] Withal, this conduct is still controversial and debatable in the literature. [16, 25,26] Most of the feasible adhesives in the market are chemistry based with either hydrophilic or hydrophobic monomers.[16] It could be expected a better performance of hydrophobic based adhesives because of the superior stability of this kind of monomers, as the water challenge to penetrate the adhesive interface could be a major task.[25]

Moreover, this specific field of comparison between the chemistry composition of adhesives and the glass-based ceramic interaction are still unclear in the literature and future studies should evaluate how the different adhesive compositions could impact negative or positively the ceramic-resin bond strength.[26] In the present study, the application of the two selected adhesives; All bond Universal (AU) and the hydrophobic part of the Scotchbond Multipurpose system (AMP) did not improve or aggravate the micro-shear bond strength between the ceramic surfaces and the resin cement agent. These results are in agreement with other studies.[14,16,27] where they used different acid sensitive ceramics, but obtained similar results with the application or not of an extra adhesive layer. However, it is important to remark that in the present study, no long-term storage or aging of the specimens was conducted. This could be consider as a study limitation, because of the possible instability of the bonding agents after aging.[28] Another important fact to be pointed out is that in the present study the light activation of the resin cement was conducted directly, without any influence of a specific ceramic thickness between the light source and the light activated resin cement. This conduct could increase the degree of conversion and hardness of the bonding agent and it is not accurately what happens in real clinical procedures.[29]

Moreover, the protocol simplification, reductions of clinical steps without affecting the bond strength quality are always alluring in dentistry procedures. Therefore future studies with different ceramic compositions, surface treatments and bonding materials should be conducted.

CONCLUSION

It may be concluded that the application or not of an extra adhesive layer will not influence positive or negatively the bond strength between a light cured resin cement and the glass-based CAD-CAM ceramic surfaces.

REFERENCES

1. Guess PC, Schultheis S, Bonfante EA, Coelho PG, Ferencz JL. All- ceramic systems: laboratory and clinical performance. Dent Clin North Am. 2011;55(2):333–352.

2. Coldea A; Swain MV; Thiel N. Mechanical properties of polymer-infiltrated-ceramic-network materials. Dent Mater 2013;29:419–26.

3. Rauch A, Reich S, Dalchau L, & Schierz O. Clinical survival of chair-side generated monolithic lithium disilicate crowns:10-year results. Clin Oral Invest 2018; 22: 1763-1769.

4. Elsaka S, Elnaghya. Mechanical properties of zirconia reinforced lithium silicate glass-ceramic. Dent Mater 2016; 32: 908-914.

5. Rinke, S., Pabel, A.-K., Rödiger, M., Ziebolz, D., 2016a. Chairside fabrication of an all- ceramic partial crown using a zirconia-reinforced lithium silicate ceramic. Case Rep. Dent. http://dx.doi.org/10.1155/2016/1354186.

6. Chen HJ, Matsumura H, Atsuta M. Effect of etchant, etching period and silane priming on bond strength to porcelain of composite resin. Oper Dent. 1998; 23(5):250-257.

7. Yen TW, Blackman RB, Baez RJ. Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic. J Prosthet Dent 1993; 70: 224-233.

8. Tian T, Tsoi J, Matinlinna J, & Burrow M. Aspects of Bonding Between Resin Luting Cements and Glass Ceramic Materials. Dent Mater 2014; 30:147-162.

9. Román-Rodríguez JL et al. Bonding to silicate ceramics: Conventional technique compared with a simplified technique. J Clin Exp Dent 2017;9:e384–e386.

10. Maier E, Bordihn V, Belli R, Taschner M, Petschelt A, Lohbauer U, & Zorzin J. New Approaches in Bonding to Glass-Ceramic: Self-Etch Glass-Ceramic Primer and Universal Adhesives. J Adhes Dent 2019; 21: 209-217.

11. Barghi N. To silanate or not to silanate: making a clinical decision. Compend Contin Educ Dent. 2000; 21(8):659-62, 664.

12. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. Dent Mater 2004;20:338-344.

13. Soderholm KJ, & Shang SW (1993) Molecular orientation of silane at the surface of colloidal silica Journal of Dental Research 72(6) 1050-1054.

14. Peumans M, Hikita K, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P, van Meerbeek B. Effects of ceramic surface treatments on the bond strength of an adhesive luting agent to CAD-CAM ceramic. J Dent 2007;35: 282-288.

15. Shimada Y, Yamaguchi S, Tagami J. Micro-shear bond strength of dual- cured resin cement to glass ceramics. Dent Mater 2002;18: 380-388.

16. Passos SP, Valandro LF, Amaral R, Ozcan M, Bottino MA, Kimpara ET. Does Adhesive Resin Application Contribute to Resin Bond Durability on Etched and Silanized Feldspathic Ceramic? J Adhes Dent 2008; 10: 455-460.

17. Shahverdi S, Canay S, Sahin E, & Blige A (1998) Effects of different surface treatment methods on the bond strength of composite resin to porcelain *Journal of Oral Rehabilitation* 25(9) 699-705.

18. Tian T, Tsoi JK, Matinlinna JP, & Burrow MF (2014) Aspects of bonding between resin luting cements and glass ceramics materials *Dental Materials* 30(7) 147-162.

19. Sato TP, Anami LC, Melo RM, Valandro LF, & Bottino MA (2016) Effects of Surface Treatments on the Bond Strength Between Resin Cement and a New Zirconia-reinforced Lithium Silicate Ceramic *Operative Dentistry* **41(3)** 284-292.

20. El-Damanhoury HM, Gaintantzopoulou MD. Self-etching ceramic primer versus hydrofluoric acid etching: etching efficacy and bonding performance. J Prosthodont Res 2018; 62: 75-83.

21. Farias DC, Gonçalves LM, Walter R, Chung Y, & Blatz MB (2019) Bond strengths of various resin cements to different ceramics *Brazilian Oral Research* 33(95) 1-10.

22. Román-Rodríguez JL, Perez-Barquero JA, Gonzalez-Angulo E, Fons-Font A, & Bustos-Salvador JL (2017) Bonding to silicate ceramics: Conventional technique compared with a simplified technique *Journal of Clinical and Experimental Dentistry* **9(3)** 384-386.

23. Dapieve K, Machry R, Pilecco R, Kleverlaan C, Pereira G, Venturini A, & Valandro L (2020) One-step ceramic primer as surface conditioner: Effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations *Journal of the Mechanical Behavior of Biomedical Materials* **10(4)** 1-8.

24. Naves LZ, Soares CJ, Moraes RR, Gonc¸alves LS, Sinhoreti MA, & Correr-Sobrinho L (2010) Surface/ interface morphology and bond strength to glass ceramic etched for different periods Operative Dentistry 35(4) 420-427.

25. El Zohairy AA, De Gee AJ, Hassan FM, Feilzer AJ. The effect of adhesives with various degrees of hydrophilicity on resin ceramic bond durability. Dent Mater 2004;20:778-87.

26. Nogueira IO, Gomes de Oliveira PF, Magno MB, Ferreira DM, Maia LC & Rabello TB (2020) Does the application of an adhesive layer improve the bond strength of etched and silanized glass ceramics to resin-based materials? A systematic review

and meta-analysis The Journal of Prosthetic Dentistry doi: 10.1016/j.prosdent.2019.12.005.

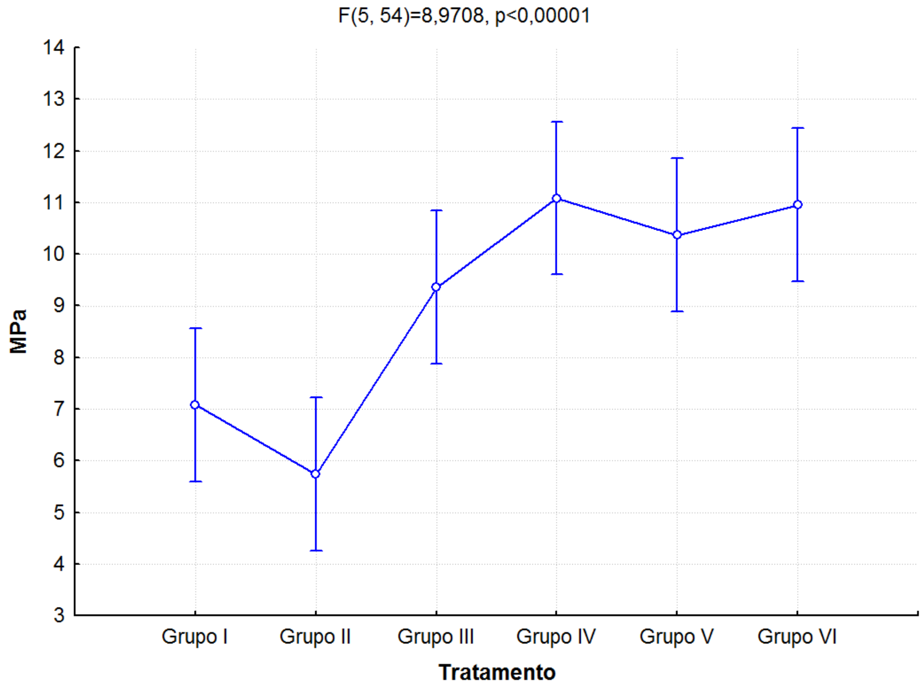
27. Lise DP, Perdigão J, Van Ende A, Zidan O, Lopes GC. Microshear bond strength of resin cements to lithium disilicate substrates as a function of surface preparation. Operative Dentistry 2015;40:524-32.

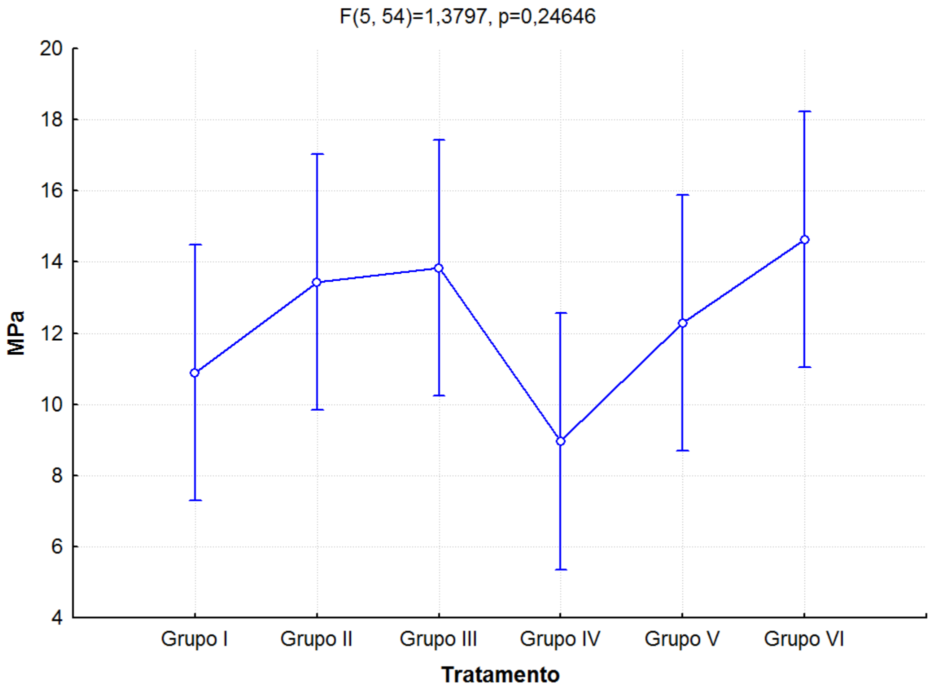
28. Lise DP, Ende A, De Munck J, Vieira LCC, Baratieri LN, Van Meerbeek B (2017) Microtensile Bond Strength of Composite Cement to Novel CAD/ CAM Materials as a Function of Surface Treatment and Aging Operative Dentistry 42(1), 73-81.

29. Calgaro PA, Furuse AY, Correr GM, Ornaghi BP, & Gonzaga CC (2013) Influence of the interposition of ceramic spacers on the degree of conversion and the hardness of resin cements Brazilian Oral Reserch 27(5) 403-409.

FIGURES

Figure 1 –





TABLES

Table 1 - – Materials used and their main characteristics.

Material	Manufacturer	Composition
IPS e.max CAD	Ivoclar Vivadent	Lithium disilicate glass-ceramic
Celtra Duo	Dentsply	85% Lithium Metasilicate 15% Zirconium dioxide
10% Condac HF	FGM	Hydrofluoric acid at 10% water, thickener, surfactant and dye.
Monobond Etch & Prime	Ivoclar Vivadent	Alcohol aqueous solution of ammonium polyfluoride, silane methacrylate and dye.
AllBond Universal	BISCO	Bisphenol A Dilycidylmethacrylate, ethanol, 10-MDP, 2-Hydroxyethyl Methacrylate
Hidrophobic part Scotchbond Multipurpose	3M	Adhesive: BisGMA (60–70 Wt%); HEMA (30–40 Wt%)
Rely X Ceramic Primer	3M	Ethyl Alcohol (70–80 wt%), water (20–30 wt%), methacryloxypropyltrimethoxysilane (<2 Trade Secret).
Variolink Esthetic LC	Ivoclar Vivadent	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, boroaluminofluorosilicate glass, spheroidal mixed oxide, benzoylperoxide, stabilizers, pigments

Table 2 - Groups & Surface conditioning protocol & Mean values and standard deviations (in parenthesis) of bond strength for the different groups.

Groups	Ceramics	
	Lithium Disilicate Reinforced Silicate	Zirconium
HF+SIL+AMP	7.07 (3.1)	10.88 (2.9)
HF+SIL+AU	5.73 (2.1)	13.43 (7.8)
MBEP+AMP	9.36 (2.0)	13.83 (6.8)
MBEP+AU	11.08 (2.4)	8.95 (6.6)
HF+SIL	10.37 (2.3)	12.29 (4.7)
MBEP	10.95 (1.5)	14.62 (2.9)

3 Discussion

3 DISCUSSION

Adhesion is both mechanical and chemical in nature. The bonding of ceramics to tooth structure is well researched and documented, yielding strong, predictable and durable bonds. (CHEN et al; 1998) A strong bond to ceramic relies on micromechanical interlocking to the ceramic surface coupled with chemical adhesion to exposed hydroxyl groups.

Adhesion requires roughening to create sufficient surface activation. Some of these methods could be sandblasting with alumina (LACY et al; 1988), grinding (SEMMELMAN et al; 1968), abrasion with diamond instruments and acid etching (HF solution or ammonium bifluoride). (WOLF et al; 1992)

The action mechanism of hydrofluoric acid is very well detailed in the literature. It reacts with the silica, eliminating it selectively, thus achieving the exposure of the crystalline structure. After obtaining a change in surface topography, the ceramics can be functionalized with silane and infiltrated with some adhesive material. (STANGEL et al; 1987, HORN; 1983, SHAHVERDI et al; 1998, TIAN et al; 2014) This classic acid agent embrace some problems reported in the literature. These includes high toxicity, corrosive potential, possible burns on skin contact, possible absorption into the bloodstream through interaction with skin and bone tissue and irreversible damage if there is eye contact. (BERTOLINI; 1992, BLATZ et al; 2003, OZCAN et al; 2012) In addition to the sensitivity of the technique as still being questioned, with relation to the agent concentration and the time of action on the ceramic surface, and if this could be detrimental to the mechanical properties of the material. (MENEES et al; 2014, ZOGHEIB et al; 2011, HOOSHMAND et al; 2008)

Therefore, Monobond Etch and Prime (MBEP) appear to be an alternative material that could minimize some of these problems. MBEP is a product that combines ammonium polyfluoride (less aggressive acid agent) and silane (trimethoxypropyl methacrylate) in a single bottle, thus reducing the number of steps and the clinical time used on the conventional technique (HF+S). (ROMÁN-RODRÍGUEZ et al; 2017, DAPIEVE et al; 2020)

In the conduction of the first study there were a few points to be addressed, the cementation procedure was conducted indirectly, with the influence of the ceramic thickness (1.5-mm) between the light source and the cemented resin composite cylinder. It is known that the influence of a ceramic material between the light source and the resin cement has the effect of decreasing the degree of conversion and the hardness of the resin agent. (WATANABE et al; 2015) For this purpose, it was used a metallic device with a disk hole with a compatible width of the active tip of the LED device. This appliance supports the ceramic slice and allowed the passage of light only from below of it. This does not allow the directly light activation of the resin cement, more accordingly to the clinical reality. (CALGARO et al; 2013)

In the first study as well, thermocyling procedure was performed to age the adhesive interface. Water storage time seems to affect the bond strength produced by the different surface treatment protocols. This exposure of the adhesive interface to an aggressive method seeks to degrade the possible bond strength. Correct and sufficient exposure is necessary to achieve the saturation of water with the resin or resin cement. Therefore, 6,000 thermocycling cycles were programmed from 5 ° C to 55 ° C as recommended by ISO (ISO / TS 11405 2015). (ISO/TS 11405; 2003)

After the ceramic surface modification and search for different protocol simplification, the second study focused more on a very common clinical step between the clinicians looking to enhance the bond strength with the use of an adhesive layer attending for the modification of the viscosity and wettability of the resin-based material for cementation. (NAVES et al; 2010) Withal, this conduct is still controversial and debatable in the literature. (PASSOS et al; 2008, EL ZOHAIRY et al; 2004, NOGUEIRA et al; 2020) Most of the feasible adhesives in the market are chemistry based with either hydrophilic or hydrophobic monomers. (PASSOS et al; 2008) It could be expected a better performance of hydrophobic based adhesives because of the superior stability of this kind of monomers, as the water challenge to penetrate the adhesive interface could be a major task. (EL ZOHAIRY et al; 2004)

In the study, the application of the two selected adhesives; All bond Universal (AU) and the hydrophobic part of the Scotchbond Multipurpose system (AMP) did not improve or aggravate the micro-shear bond strength between the ceramic surfaces and the resin cement agent. These results are in agreement with other studies (PEUMANS et al; 2007, PASSOS et al; 2008, LISE et al; 2015) where they used different acid sensitive ceramics, but obtained similar results with the application or not of an extra adhesive layer. However, it is important to remark that in the study, no long-term storage or aging of the specimens was conducted. This could be consider as a study limitation, because of the possible instability of the bonding agents after aging. (LISE et al; 2017) Another important fact to be pointed out is that in the present study the light activation of the resin cement was conducted directly, without any influence of a specific ceramic thickness between the light source and the light activated resin cement. This conduct could increase the degree of conversion and hardness of the bonding agent and it is not accurately what happens in real clinical procedures. (CALGARO et al; 2013)

Moreover, in both of the studies, reductions of clinical steps without affecting the bond strength quality were the main idea. Both using silica-based ceramics systems. The first study, trying to use some 10-MDP containing products and different protocol applications to potentialize the bond strength between the resin cement agent and the second one, reducing one extra step in the standard protocol for ceramics cementation. Therefore future studies with different ceramic compositions, surface treatments and bonding materials should be conducted.

REFERENCES

REFERENCES

Barghi N (2000) To silanate or not to silanate: making a clinical decision. **Compend Contin Educ Dent**; 21(8):659-62, 664.

Bertolini JC (1992) Hydrofluoric acid: a review of toxicity **J. Emerg. Med**; 10(2) 163-168.

Blatz MB, Sadan A, & Kern M (2003) Resin-ceramic bonding: A review of the literature **J Prosthet Dent**; 89(3) 268-274.

Calgaro PA, Furuse AY, Correr GM, Ornaghi BP, & Gonzaga CC (2013) Influence of the interposition of ceramic spacers on the degree of conversion and the hardness of resin cements **Braz Oral Res**; 27(5) 403-409.

Chen JH, Matsumura H, Atsuta M (1998) Effect of etchant, etching period, and silane priming on bond strength to porcelain of composite resin **Oper Dent**; 23(5):250-257.

Coldea A; Swain MV; Thiel N (2013) Mechanical properties of polymer-infiltrated-ceramic-network materials **Dent Mater**; 29:419–26.

Conrad HJ, Seong WJ, Pesun IJ (2007) Current ceramic materials and systems with clinical recommendations: a systematic review **J Prosthet Dent**; 98(5): 389-404.

Dapieve K, Machry R, Pilecco R, Kleverlaan C, Pereira G, Venturini A, & Valandro L (2020) One-step ceramic primer as surface conditioner: Effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations **J Mech Behav of Biomed Mater**; 10(4) 1-8.

Della Bona A, Kelly JR (2008) The clinical success of all-ceramic restorations **J Am Dent Assoc**;139(suppl): 8S-13S.

El Zohairy AA, De Gee AJ, Hassan FM, Feilzer AJ (2004) The effect of adhesives with various degrees of hydrophilicity on resin ceramic bond durability **Dent Mater**; 20:778-87.

Elsaka SE, & Elnaghy AM (2016) Mechanical properties of zirconia reinforced lithium silicate glass-ceramic **Dent Mater**; 32(7) 908-914.

Gracis S, Thompson VP, Ferencz JL, Silva NRFA, & Bonfante EA (2015) A new Classification System for All-Ceramic and Ceramic-like Restorative Materials **Int J Prosthodont**; 28:227–235.

Hooshmand T, Parvizi S, & Keshvad A (2008) Effect of surface acid etching on the biaxial flexural strength of two hot-pressed glass ceramics **J Prosthodont**; 17(5) 415-9.

Horn HR (1983) Porcelain laminate veneers bonded to etched enamel **Dent Clin N Am**; 27(4) 671-684.

ISO/TS 11405 (2003) Testing of adhesion to tooth structure Geneva: International Organization for Standardization **Dent Mater**.

Kelly JR (2008) Dental Ceramics: What Is This Stuff Anyway? **J Am Dent Assoc**; 139;4S-7S.

Lacy AM, LaLuz J, Watanabe LG, Dellinges M (1988) Effect of porcelain surface treatment on the bond to composite **J Prosthet Dent**;60(3):288-291.

Lise DP, Ende A, De Munck J, Vieira LCC, Baratieri LN, Van Meerbeek B (2017) Microtensile Bond Strength of Composite Cement to Novel CAD/ CAM Materials as a Function of Surface Treatment and Aging **Oper Dent;** 42(1), 73-81.

Lise DP, Perdigão J, Van Ende A, Zidan O, Lopes GC (2015) Microshear bond strength of resin cements to lithium disilicate substrates as a function of surface preparation **Oper Dent**; 40:524-32.

Maier E, Bordihn V, Belli R, Taschner M, Petschelt A, Lohbauer U, & Zorzin J (2019) New Approaches in Bonding to Glass-Ceramic: Self-Etch Glass-Ceramic Primer and Universal Adhesives **J Adhes Dent;** 21(3) 209-217.

Menees TS, Lawson NC, Beck PR, & Burgess JO (2014) Influence of particle abrasion or hydrofluoric acid etching on lithium disilicate flexural strength **J Prosthet Dent**; 112(5) 1164-1170.

Naves LZ, Soares CJ, Moraes RR, Gonc¸alves LS, Sinhoreti MA, & Correr-Sobrinho L (2010) Surface/ interface morphology and bond strength to glass ceramic etched for different periods **Oper Dent**; 35(4) 420-427.

Nogueira IO, Gomes de Oliveira PF, Magno MB, Ferreira DM, Maia LC & Rabello TB (2020) Does the application of an adhesive layer improve the bond strength of etched and silanized glass ceramics to resin-based materials? A systematic review and meta-analysis **J Prosthet Dent**; doi: 10.1016/j.prosdent.2019.12.005.

Ozcan M, Allahbeickaraghi A, & Dündar M (2012) Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review **Clin Oral Investig**; 16(1) 15-23.

Passos SP, Valandro LF, Amaral R, Ozcan M, Bottino MA, Kimpara ET (2008) Does Adhesive Resin Application Contribute to Resin Bond Durability on Etched and Silanized Feldspathic Ceramic? **J Adhes Dent**; 10: 455-460.

Peumans M, Hikita K, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P, Van Meerbeek B (2007) Effects of ceramic surface treatments on the bond strength of an adhesive luting agent to CAD-CAM ceramic **J Dent**; 35: 282-288.

Rauch A, Reich S, Dalchau L, & Schierz O (2018) Clinical survival of chair-side generated monolithic lithium disilicate crowns:10-year results **Clin Oral Invest**; 22: 1763-1769.

Román-Rodríguez JL, Perez-Barquero JA, Gonzalez-Angulo E, Fons-Font A, & Bustos-Salvador JL (2017) Bonding to silicate ceramics: Conventional technique compared with a simplified technique **J Clin Exp Dent**; 9(3) 384-386.

Semmelman JO, Kulp PR (1968) Silane bonding porcelain teeth to acrylic. **J Am Dent Assoc**; 76(1):69-73.

Shahverdi S, Canay S, Sahin E, & Blige A (1998) Effects of different surface treatment methods on the bond strength of composite resin to porcelain **J Oral Rehabil**; 25(9) 699-705.

Stangel I, Nathanson D, & Hsu CS (1987) Shear strength of the composite bond to etched porcelain **J Dent Res**; 66(9) 1460-1465.

Tian T, Tsoi JK, Matinlinna JP, & Burrow MF (2014) Aspects of bonding between resin luting cements and glass ceramics materials **Dent Mater**; 30(7) 147-162.

Traini T, Sinjari B, Pascetta R, Serafini N, Perfetti G, Trisi P, & Caputi S (2016) The zirconia-reinforced lithium silicate ceramic: lights and shadows of a new material **Dent Mater J**; 35(5) 748-755.

Watanabe H, Kazama R, Asai T, Kanaya F, Ishizaki H, Fukushima M, & Okiji T (2015) Efficiency of dual-cured resin cement polymerization induced by high-intensity LED curing units through ceramic material **Oper Dent**; 40(2) 153-162.

Wolf DM, Powers JM, O'Keefe KL (1992) Bond strength of composite to porcelain treated with new porcelain repair agents. **Dent Mater**; 8(3):158-161.

Zogheib LV, Bona AD, Kimpara ET, & McCabe JF (2011) Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. **Braz Dent J**; 22(1) 45-50.