UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

CAMILA CRUZ LIBARDI

Influence of etching time and adhesive system on shear bond strength and compression resistance of the reinforced leucite ceramic

Influência do tempo de condicionamento ácido e do sistema adesivo na resistência de união e compressão da cerâmica reforçada por leucita

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Orientadora: Profa. Dra. Juliana Fraga Soares Bombonatti

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FOLHA DE APROVAÇÃO

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ABSTRACT

Influence of etching time and adhesive system on shear bond strength and compression resistance of the reinforced leucite ceramic.

This in vitro study evaluated the bond and compression strength of cemented leucite reinforced glass ceramics in bovine tooth enamel, comparing three etching times with hydrofluoric acid 10% of the ceramic surface (20, 60 and 90 seconds) and two adhesive treatments (adhesive system + silane and universal adhesive system). For the bond strength test, 120 ceramic cylinders (2mm diameter x 2mm length; n=20) were etched and cemented (80µm thick) in enamel with a dual resin cement, varying the adhesive treatment, obtaining the groups: UEXC20s, UEXC60s, UEXC90s, USBU20s, USBU60s, USBU90s. After 24 hours, the shear bond strength test was performed on a universal test machine (0.5mm/min, 50kgf). For the compression test, 30 ceramic plates (5x5mm with 1mm thick, n=5) were etched, received the same adhesive treatments and were cemented (80µm) in enamel, obtaining the following groups: CEXC20s, CEXC60s, CEXC90s, CSBU20s, CSBU60s, CSBU90s. After 24 hours of cementation, the compression test (0.5mm/min, 500kgf) was performed. Statistical analysis was performed using two-way ANOVA and Tukey test (α=.05). For the shear bond strength test significant differences were found among the adhesives ($p<.05$). For the etching times there were no differences ($p=.059$). However, there was a significant interaction between the adhesives and the etching times ($p=.021$). At 60 seconds, the Silane + ExciTE F DSC Adhesive presented the highest bond strength values (47.53±16.70Mpa). And at 20 seconds, the Universal adhesive presented the lowest bond strength values (27.72±10.76Mpa). For the compression test there were no significant differences between the adhesives ($p=.571$) and between the times ($p=.154$). The group that presented the highest values of compression force was the Universal adhesive at 60 seconds (1757.89±200.47N). The lowest values were also the Universal adhesive, but at 90 seconds (1213.30±546.34N). The study concluded that the silane associated with the ExciTE F DSC adhesive showed the highest bond strength values at the etching time of 60 seconds with 10% hydrofluoric acid, without compromising the compressive strength of the leucite reinforced ceramic.

Key-words: Ceramics. Compressive Strength. Hydrofluoric Acid. Shear Bond Strength.

RESUMO

Influência do tempo de condicionamento ácido e do sistema adesivo na resistência de união e compressão da cerâmica reforçada por leucita

Este estudo *in vitro* avaliou a resistência de união e compressão da cerâmica reforçada por cimentada em esmalte de dentes bovinos, comparando-se três tempos de condicionamento com ácido fluorídrico 10% da superfície cerâmica (20, 60 e 90 segundos) e dois tratamentos adesivos (sistema adesivo + silano e sistema adesivo universal). Para o teste de resistência de união (U), 120 cilindros cerâmicos (2mm diâmetro x 2mm comprimento; n=20) foram condicionados e cimentados (80 µm de espessura) em esmalte, com cimento resinoso dual, variando-se o tratamento adesivo, obtendo-se os grupos: UEXC20s, UEXC60s, UEXC90s, USBU20s, USBU60s, USBU90s. Após 24h, foi realizado o teste de cisalhamento (0,5mm/min, 50kgf). Para o teste de compressão (C), 30 placas cerâmicas (5x5mm com 1mm de espessura; n=5) foram condicionadas, receberam os mesmos tratamentos adesivos e foram cimentadas (80µm) em esmalte, obtendo-se os grupos: CEXC20s, CEXC60s, CEXC90s, CSBU20s, CSBU60s, CSBU90s. Após 24h da cimentação, foi realizado o teste de compressão (0,5mm/min, 500kgf). Foi realizada a análise estatística por meio de ANOVA dois critérios e teste de Tukey ($P < 0,05$). Para o teste de cisalhamento diferenças significativas foram encontradas entre os adesivos ($p<0,05$). Para os tempos de condicionamento não houve diferenças significativas ($p=0,059$). No entanto, houve interação significativa entre os adesivos e os tempos de condicionamento ($p=0,021$). No tempo de 60 segundos, o silano + adesivo ExciTE F DSC apresentou os maiores valores de resistência de união (47,53±16,70Mpa). E no tempo de 20 segundos, o adesivo Universal apresentou os menores valores de resistência de união (27,72±10,76Mpa). Para o teste de compressão não houve diferenças significativas entre os adesivos ($p=0,571$) e entre os tempos ($p=0,154$). O grupo que apresentou maiores valores de força de compressão foi o adesivo Universal no tempo de 60 segundos (1757,89±200,47N). Já os menores valores, foi também o adesivo Universal, mas no tempo de 90 segundos (1213,30±546,34N). Pode-se concluir que, o Silano associado ao adesivo ExciTE F DSC, mostrou os maiores valores de resistência de união no tempo de condicionamento de 60 segundos com ácido fluorídrico a 10%, sem comprometer a resistência a compressão da cerâmica reforçada por leucita.

Palavras-chave: Ácido fluorídrico. Cerâmicas. Força compressiva. Resistência de União.

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1 INTRODUCTION

1 INTRODUCTION

Ceramics have good resistance and excellent optical properties, which produces aesthetic quality and satisfactory hardness (Gomes *et al*., 2008), besides having excellent biocompatibility (Zogheib *et al*., 2011). Such properties make them suitable for use in dentistry to replace one or more missing teeth.

In order to further improve its characteristics, ceramics have been reinforced by various materials, such as leucite, silica, lithium and alumina disilicate, among other elements.

Adhesive cementation of glass ceramic restorations can be optimized with acid etching, silane application and adhesive system. Acid etching stabilizes porcelain surface defects by reacting with the glass matrix of the ceramic and removing it, forming retentive microchannels (Sattabanasuk *et al.,* 2017). Some studies have proved that the time increase of hydrofluoric acid etching increases the roughness (Lyann *et al*., 2018, and the ensuing bonding of the ceramic to the surface of the enamel (Addison *et al*., 2006; Zogheib *et al*., 2011; Sundfeld *et al.,* 2018).

The use of the silane agent optimyses the porcelain adhesion to the dental enamel, as it forms a siloxane and silica network on the ceramic surface, increasing the union between the dental enamel and the porcelain (Colares *et al*., 2013). The organic fraction of the silane is capable of increase the surface wettability of the ceramic, contributing to better adhesion (Mattos *et al*., 2006; Yao *et al*., 2018).

It is recommended by the manufacturer the etching time with hydrofluoric acid at 60 seconds for leucite reinforced ceramics. However, this time can vary from 15 to 60 seconds, associated with the use of silane, adhesive and resin cement. The longest etching time seems to provide better bond strength results (Tostes BO, 2016), although it leaves room for doubt as whether it can compromise the compressive strength of the ceramic.

Clinically speaking, during the simultaneous surface acid treatment of several pure porcelain prosthetics prior to adhesive cementation to the prepared teeth, the acid etching time can be extended or decreased. This may lead to the increase or decrease of the mechanical properties of the leucite reinforced ceramic.

The acid etching of the ceramic surface is a very important step for the adhesive cementation of etched ceramic restorations, especially in those reinforced by leucite (Rigolin *et al.,* 2014). If not performed correctly, it can lead to problems such as bonding failure (ceramic to dentin or enamel), as well as problems in its mechanical properties (Sattabanasuk *et al.*, 2017).

The development of the universal adhesives systems with 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and silane in their composition also seems to be a viable option for adhesive cementation of the ceramics. However, there is little scientific evidence regarding the behavior of these adhesives for the cementation of the leucite reinforced ceramics.

The ceramic is still widely used in laboratories as a more accessible value option with excellent optical and mechanical properties. Thus, with this study, the clinician is expected to have more accurate to enable the professional to take this important and sensitive step in the cementation technique of pieces made from leucite reinforced glass ceramics.

PROPOSITION

Therefore, this *in vitro* study aimed to evaluate the influence on shear bond strength and compression resistance to the understanding of a leucite reinforced ceramic, by varying the etching time with hydrofluoric acid and using two adhesive treatments.
The null hypotheses tested were:

1. There would be no difference in bond strength between the acid etching times of the ceramic and the adhesive systems used.
2. There would be no difference in the compressive strength between the acid etching times of the ceramic and adhesive systems used.

2 Article

2 ARTICLE

Article - The article presented in this Dissertation was written according to The Journal of Prosthetic Dentistry instructions and guidelines for article submission.

Influence of etching time and adhesive system on shear bond strength and compression resistance of the reinforced leucite ceramic.

ABSTRACT

Statement of the Problem: Adhesive cementation of glass ceramic restorations can be optimized with acid etching, silane application and adhesive system. However, the establishment of an appropriate protocol is fundamental to avoid any failure in the union of the restoration and compromising of the mechanical properties of the ceramic leucite.

Objective: This in vitro study evaluated the shear bond strength and compression strength of cemented leucite reinforced glass ceramics in bovine tooth enamel, comparing three etching times (20, 60 and 90 seconds) with hydrofluoric acid 10% of the ceramic surface and two adhesive treatments (adhesive system + silane and universal adhesive system).

Materials and Methods: For the shear bond strength test, 120 ceramic cylinders (2mm diameter x 2mm length; n=20) and cemented (80µm thick) in enamel with a dual resin cement, varying the adhesive treatment and etching times, obtaining the groups: UEXC20s, UEXC60s, UEXC90s, USBU20s, USBU60s, USBU90s. After 24 hours, the shear bond strength test was performed on a universal test machine (0.5mm/min, 50kgf). For the compression test, 30 ceramic plates (5x5mm with 1mm thick, n=5) were etched, received the same adhesive treatments and were cemented (80µm) in enamel, obtaining the following groups: CEXC20s, CEXC60s, CEXC90s, CSBU20s, CSBU60s, CSBU90s. After 24 hours of cementation, the compression test (0.5mm/min, 500kgf) was performed. Statistical analysis was performed using two-way ANOVA and Tukey test (α=.05).

Results: For the shear bond strength test significant differences were found among the adhesives ($p<.05$). For the etching times there were no differences ($p=.059$). However, there was a significant interaction between the adhesives and the etching times ($p=.021$). At 60 seconds, the Silane + ExciTE F DSC adhesive presented the highest bond strength values (47.53±16.70Mpa). And at 20 seconds, the Universal adhesive presented the lowest bond strength values (27.72±10.76Mpa). For the compression test there were no significant differences between the adhesives ($p=.571$) and between the times ($p=.154$). The group that presented the highest values of compression force was the Universal adhesive at 60 seconds (1757.89±200.47N). The lowest values were also the Universal adhesive, but at 90 seconds (1213.30±546.34N).

Conclusions: The study concluded that the silane associated with the ExciTE F DSC adhesive showed the highest bond strength values at the etching time of 60 seconds with 10% hydrofluoric acid, without compromising the compressive strength of the leucite reinforced ceramic.

Key-words: Ceramics. Compressive Strength. Hydrofluoric Acid.
Shear Bond Strength.

CLINICAL IMPLICATIONS

According to this study, the clinician is expected to have more accurate information to enable the professional to takes this important and sensitive step in the technique of cementing pieces made with leucite glass ceramics. This ceramic is still widely used in laboratories, being an option with more accessible value and with excellent optical and mechanical properties.

INTRODUCTION

Ceramics have good resistance and excellent optical properties, which produces aesthetic quality and satisfactory hardness[1], besides having excellent biocompatibility[2]. Such properties make them suitable for use in dentistry to replace one or more missing teeth.

In order to further improve its characteristics, ceramics have been reinforced by various materials, such as leucite, silica, lithium and alumina disilicate, among other elements.

Adhesive cementation of glass ceramic restorations can be optimized with acid etching, silane application and adhesive system. Acid etching stabilizes porcelain surface defects by reacting with the glass matrix of the ceramic and removing it, forming retentive microchannels[3].

Some studies have proved that the time increase of hydrofluoric acid etching increases the roughness[4] and the ensuing bonding of the ceramic to the surface of the enamel[5,2,6].

The use of the silane agent optimyses the porcelain adhesion to the dental enamel, as it forms a siloxane and silica network on the ceramic surface, increasing the union between the dental enamel and the porcelain[7]. The organic fraction of the silane is capable to increase the surface wettability of the ceramic, contributing to better adhesion[8,9].

It is recommended by the manufacturer the etching time with hydrofluoric acid at 60 seconds for leucite reinforced ceramics. However, this time can vary from 15 to 60 seconds, associated with the use of silane, adhesive and resin cement. The longest etching time seems to provide better bond strength results[10], although it leaves room for doubt as whether it can compromise the compressive strength of the ceramic.

Clinically, during the simultaneous surface acid treatment of several pure porcelain prosthetics prior to adhesive cementation to the prepared teeth, the acid etching time can be extended or decreased. This may lead to the increase or decrease of the mechanical properties of the leucite reinforced ceramic.

The acid etching of the ceramic surface is a very important step for the adhesive cementation of etched ceramic restorations, especially in those reinforced by leucite[11]. If not performed correctly, it can lead to problems such as bonding failure (ceramic to resin cement), as well as problems in its mechanical properties[3].

The development of the universal adhesives systems with 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and silane in their composition also seems to be a viable option for adhesive cementation of the ceramics. However, there is little scientific evidence regarding the behavior of these adhesives for the cementation of the leucite reinforced ceramics.

Therefore, this in vitro study aimed to evaluate the influence on shear bond strength and compression resistance to the understanding of a leucite reinforced ceramic, by varying the etching time with hydrofluoric acid and using two adhesive treatments.

The null hypotheses tested were:

1. There would be no difference in bond strength between the acid etching times of the ceramic and the adhesive systems used.
2. There would be no difference in the compressive strength between the acid etching times of the ceramic and adhesive systems used.

MATERIALS AND METHODS

Trial Design

This in vitro study evaluated the following factors:

- Etching time with 10% hydrofluoric acid (in 3 levels: 20, 60 and 90 s)
- Adhesive Treatment (in 2 levels: silane + ExciTE F DSC adhesive and Single Bond Universal Adhesive)

The response variables were:

- Shear bond strength and compression resistance.

The materials used are in the table 1.

Table 1 – Commercial name, manufacturer and material composition.

COMERCIAL NAME	MANUFACTURER	COMPOSITION
Porcelain etching	Dentsply	10% hydrofluoric acid, water, thickener and dye.
Phosphoric acid 37%	Dentsply	37% phosphoric acid, thickener, dye and deionized water.
IPS Empress Esthetic	Ivoclar-Vivadent	SiO_2> 55% by weight, K_2O, Al_2O_3, Na_2O, B_2O_3, CaO, TiO_2, CeO_2 and dyes.
MonoBond Plus	Ivoclar-Vivadent	Alcoholic solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate.
ExciTE F DSC	Ivoclar-Vivadent	HEMA, dimethacrylate, phosphonic acid acrylate, highly dispersed silicon dioxide, primers, stabilizers and potassium fluoride in an alcoholic solution. The applicator is coated with primers.
Single Bond Universal	3M ESPE	MDP Phosphate monomer, dimethacrylate resin, HEMA, copolymer, polyalkenoic acid, ethanol, water, primers and silane.
Variolink N	Ivoclar-Vivadent	BisGMA, urethane dimethacrylate and triethyleneglycol dimethacrylate, barium glass, ytterbium trifluoride, barium and aluminum fluorsilicate glass and mixed spheroidal oxides, initiators, stabilizers and pigments.

Teeth selection and preparation

Sixty crowns of bovine incisors were used, cleaned with a curette, brush and running water, cut in a cutted root. The crowns were taken to the politrix (PLF DV; Fortel) to standardize the size (1.8cm maximum length) and to plan their Vestibular surface using metallographic sandpaper from the highest to the lowest granulation (24, 320, 600). All crowns were included in PVC pipes (¾ inch, Tiger S/A), with type IV stone gypsum (Durone; Dentsply), leaving the Vestibular face exposed and parallel to the surface of the supporting table. After inclusion, the specimens were returned to the politrix (fine-grained sandpaper, 600 and 1200) to level the surface, leaving the enamel polished and uniform.

The specimens that exhibited dentin exposure were discarded and replaced. The specimens were immersed in distilled water and kept in a refrigerator until they were used to make ceramic cementation.

Trial Design 1

SHEAR BOND STRENGTH OF LEUCITE REINFORCED GLASS CERAMICS

A.1 – Specimen preparation

Thirty crowns of bovine incisors were used, included with stone gypsum in PVC pipes, as previously described, divided into 6 groups with 5 teeth each, where each tooth was cemented with 4 ceramic cylinders (n=20), with uniform cementation thickness[10], according to the groups evaluated (Table 2).

Table 2 – Groups Division for the shear bond strength test.

SHEAR BOND STRENGTH	ETCHING TIME (in seconds)	ADHESIVE TREATMENT
UEXC20s	20	Silane MonoBond Plus + ExciTE F DSC
UEXC60s	60	Silane MonoBond Plus + ExciTE F DSC
UEXC90s	90	Silane MonoBond Plus + ExciTE F DSC
USBU20s	20	Single Bond Universal
USBU60s	60	Single Bond Universal
USBU90s	90	Single Bond Universal

Ceramic specimens were obtained through the lost wax technique and ceramic injection. For the cylinders production cylindrical wax sticks were used measuring 40mm in length x 2mm in diameter (Sprue of wax no.2, Kota). The cylindrical sticks and plates were weighed on a digital scale (PB303; Mettler Toledo) to quantify the value of inserts required for the ceramic injection. These were fixed to the ring base with sticky wax, sprinkled with anti-bubbles and then, the silicone ring was positioned. For inclusion, 100mg of powder and 16ml:11ml of coating liquid itself (IPS PressVEST; Ivoclar-Vivadent) were handled in a vacuum machine and the revest ring was filled.

After 25 minutes, it was inserted into a conventional preheated furnace to 850 °C to remove the wax for 45 minutes. Therefore, an area was created for its subsequent filling with the injection of the glass-ceramic. Still in this furnace, the leucite reinforced ceramic insert was positioned in the refractory opening (infeed channel). This set (refractory and ceramic) was placed in a furnace (EP 600; Ivoclar-Vivadent) and then the protocol recommended by the manufacturer was used. The initial temperature was 700 ºC with a heating rate of 60 ºC/min, with final temperature for the ceramic injection of 1075 ºC, maintaining injection time for 20 minutes. After ring cooling at room temperature, the ceramic specimens were carefully disinfected using carborundum discs for cutting and removal of the coating, blasting with aluminum oxide (100 µm, 2 bar pressure) to remove the adhered coating and 15 minutes in 1% hydrofluoric acid solution on ultrasound to complete the coating removal.

The IPS Empress Esthetic ceramic cylinders, after cleaning the coating, were taken to the precision cutting machine (IsoMet 1000; Buehler) and cut into smaller cylinders measuring 2mm in diameter x 2mm in height (checked with specimen) (Fig. 1). A total of 120 cylinders were stored in a pot with a lid until use.

Fig. 1 - Ceramic cylinders measuring 2mm in diameter x 2mm in height.

A.2 - Acid etching and Adhesive Treatment

The cylinders were placed in a specific device developed for a Universal Testing Machine (EMIC 23-10; EMIC/Instron) (TOSTES BO, 2016), but it was adapted for a new equipment developed in this study for manual standardization of the cementation line, which will be described in "A.3 – Cementation". In this way, the bases of the cylinders were exposed (Fig. 2) and etched with 10% hydrofluoric acid (HF).

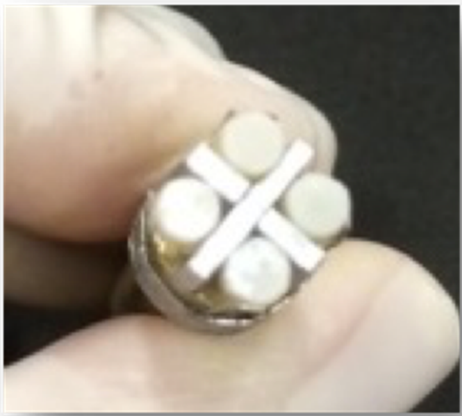


Fig. 2 – Bases of the cylinders were exposed when placed in a specific device.

The UEXC20s and USBU20s groups received HF for 20s. For UEXC60s and USBU60s, the HF application time was 60s (time recommended by the ceramic manufacturer) and the UEXC90s and USBU90s groups were etched by 90s. After the respective etching times, the specimens (cylinders) were washed with plenty of water (triple syringe) for 60s and dried with air jets for 30s. For the groups of the ExciTE F DSC (EXC) adhesive, the silane agent MonoBond Plus with a microbrush (KG Brush Fine; KG Sorensen) was previously applied in two layers: application of the first layer, waiting for 10s, the second layer, waiting for 60s and removal of excess with air jets. For the groups of the Single Bond Universal (SBU) adhesive, there was no application of the silane, as it is found in the composition of the adhesive. The application in both of them was performed with a disposable applicator, in a thin and uniform layer, sequenced from the application of soft air jets (triple syringe) for solvent evaporation and removal of excess with sterile absorbent paper. The adhesives were not prepolymerized in the cylinders.

The Vestibular faces of bovine teeth were etched with 37% phosphoric acid for 30s, washed with plenty of water for 30s and dried with air jets (triple syringe) for 15s. Each group received the respective single layer adhesive system applied with a microbrush, sequenced from soft air jets to evaporate the solvent, removing the excess with sterile absorbent paper. The adhesive was photopolymerized for 10s using the LED light-activating unit (DB 685; Dabi Atlante) with a power density of 1000mW per cm^2.

A.3 – Cementation

All cylinders were cemented with Variolink N dual resin cement, according to the manufacturer's guidelines. After mixing the equal portions of the base and catalyst, the cylinders received a small amount of the cement and were positioned on the tooth surface with the aid of the adapted device, which kept the cement line (80µm) uniform. In order to standardize the thickness of the cementation line and to cement more than one cylinder at a time, a specific device for fitting to the Universal Testing Machine EMIC was developed at the Integrated Research Center of FOB-USP. Such device presents 4 attachments for fixing 4 cylinders to be cemented simultaneously[10]. Such device has been adapted, with a specific part, for precise fitting to a new manual cementing equipment for standardization of the cement line (Fig. 3), also developed in FOB-USP. This equipment was developed with two linear guides, joined by a movable base, supported under a fixed base.

Fig. 3 - New manual cementing equipment for standardization of the cement line.

The adapted device fits perfectly into this movable base, bolted through a thread which descends to the base of the equipment.

In this way, the cement line remained constant without any pressure being exerted, since the device remained fixed in a previously calibrated position through a 0.8mm thick spacer. To stipulate the thickness of the cementation line, the specimen was first positioned in the adapted device which is threaded into the movable base of the equipment and lowered manually by manipulating the two side guides that descend parallel to the surface of the spacer located on the tooth surface and on the fixed base of the equipment (Fig. 4).

Fig. 4 – The spacer located on the tooth surface and on the fixed base of the equipment.

Once in contact with the spacer, the specimen was removed to then recede the guides at 0.8mm, and the area was filled with cement. The guides were in this position and for all specimens after being positioned, the cementation was performed. The photoactivation was complemented by further 20s in the upper portion, with the LED light-curing unit with a density power of 1000mW/cm^2. The specimens were stored in distilled water in an incubator at 98.6±33.8 ºF for 24 hours.

It is important to note that, in order to guarantee the accuracy of the cementing line, a specimen of the pilot test of the experiment was sanded on the politrix to expose the adhesive line and it was taken to the scanning electron microscope (SEM) (T220A; JEOL) to confirm the thickness standardization of the cementation line.

A.4 – Shear bond strength

After the cementation of the cylinders, the specimens were stored in distilled water at 37±1 ºC for 24 hours and then submitted to the shear bond strength test. The tests of shear bond strength were performed in a Universal Testing Machine (KE serie; Kratos) with a 50kg load cell and a displacement speed of 0.5mm per minute. The maximum load supported by each specimen was recorded in the Universal Testing Machine software in MPa.

The specimens were inserted into the posterior opening of the test metal device and secured with a bolted metal plate. The opening at the front made the tested cylinders visible. The device was coupled to the base of the Universal Testing Machine and an opening in the upper part allowed the passage of the steel wire (# 8; Morelli) in a handle form that descended by lacing the cylinders one at a time. The wire attached to the load cell at one end had its handle positioned at the adhesive joint (tooth/cylinder) so as to be close and parallel to the tooth surface. The test was started, pulling the wire and transmitting charge until the cylinder moved[12].

After completion of the shear bond strength test, the surfaces of the specimens were analyzed by using a digital **microscope (DINO-LITEplus digital microscope; AnMo Electronics Corp)** to determine the failure type involved. Failures were classified as adhesive, cohesive, or mixed.

Trial Design 2

COMPRESSIVE STRENGTH OF THE LEUCITE REINFORCED GLASS CERAMIC

B.1 – Specimen preparation

Thirty crowns of bovine incisors were used and included in PVC pips with special type lV stone gypsum, as previously described. These were divided into 6 groups, with 5 teeth in each one. Rectangular ceramic plates (1mm thick x 5mm wide x 18mm long) obtained from the inclusion of wax plates type 9 (Pink Wax; Lysanda) (lost wax technique), as described in item 1 of the trial design 1, were cut with diamond wheel (Superflex; Edenta) in squares of 5x5mm and 1mm thick (Fig. 5), totaling 30 square ceramic plates. Each tooth received 1 cemented plate, adding 5 per group (n=5) (Table 3).

Fig. 5 – Ceramic squares of 5x5mm and 1mm thick.

Table 3 – Groups division for the compression strength test.

COMPRESSION STRENGTH (C)	ETCHING TIME (in seconds)	ADHESIVE TREATMENT
CEXC20s	20	Silane MonoBond Plus + ExciTE F DSC
CEXC60s	60	Silane MonoBond Plus + ExciTE F DSC
CEXC90s	90	Silane MonoBond Plus + ExciTE F DSC
CSBU20s	20	Single Bond Universal
CSBU60s	60	Single Bond Universal
CSBU90s	90	Single Bond Universal

B.2 – Acid etching and adhesive treatment

The ceramic plates were etched with 10% hydrofluoric acid on all surfaces and the exposure time was adjusted according to the following groups: CEXC20s and CSBU20s - 20s, CEXC60s and CSBU90s - 60s (manufacturer recommended time) and CEXC90s and CSBU90s - 90s.

After the respective etching times, the ceramic plates were washed with plenty of water (triple syringe) for 60s and dried with air jets for 30s. For the groups of the EXC adhesive, the silane agent MonoBond Plus was applied in two-layers by a microbrush: first coat application, 10s, second coat applied, 60s and removed air jets. For the groups of the SBU adhesive, there was no silane application. The application of both adhesives was performed with a disposable applicator, in a thin and uniform layer, sequenced from the application of soft air jets (triple syringe) for solvent evaporation and removal of excess with sterile absorbent paper. The adhesives were not previously photopolymerized on the ceramic plates.

The teeth were etched with 37% phosphoric acid for 30s, washed with plenty of water for 30s and dried with air jets (triple syringe). Then, the enamel was hybridized with the respective adhesive system which was applied by a microbrush in a single layer. Soft air jets for solvent evaporation was used and the excess was removed with sterile absorbent paper. The photopolymerization for 10s was accomplished using a photoactivating unit of LED light with power density of 1000 mW/cm^2.

B.3 - Cementation

All plates were cemented in the same manual cylinder cementation equipment with Variolink N dual resin cement, following the manufacturer's guidelines. After mixing the equal portions of the slides, the slabs received a small amount of the plates and were applied to the teeth by being positioned on the slab surfaces and pressed with the same adapted cementation device for the shear bond strength test, keeping the cement line uniform (80µm). The photopolymerization was carried out with the same photoactivating device, for 60s, in three directions (20s behind and 20s in each frontal diagonal) and after the removal of the device, a new polymerization of 20s was performed on the upper face.

After cementation, the specimens were immersed in distilled water in an incubator at 37±1 ºC for 24 hours.

B.4 – Compression test

After 24 hours of cementation of the plates, the compression test was performed using a pencil-shaped device with a rounded tip of 1.5mm, positioned in the center of the previously marked plate[13,10].

The tests were also performed on the Kratos KE Series Universal Testing Machine, with a speed of 0.5mm/min and a 500kgf load cell, with printing compressive force. The test was interrupted when the first discontinuity of the graphs appeared, indicating an early crack or even a fracture. The compression force required to cause damage to the ceramic plates was recorded in Newtons (N).

Statistical analysis

The Statistical analysis was performed using a software (Statistica 10; Stat Soft Inc). The data of shear bond strength and compression were submitted to the Bartlet test to verify the homogeneity of the variations. When the homogeneity was verified, the data were analyzed using two-way ANOVA, considering the adhesive treatment and acid etching time as independent variable and shear bond strength (in MPa) and compressive strength (in N) as dependents variables. Multiple comparisons were performed using the Tukey test with results that presented significant difference ($P < .05$).

RESULTS

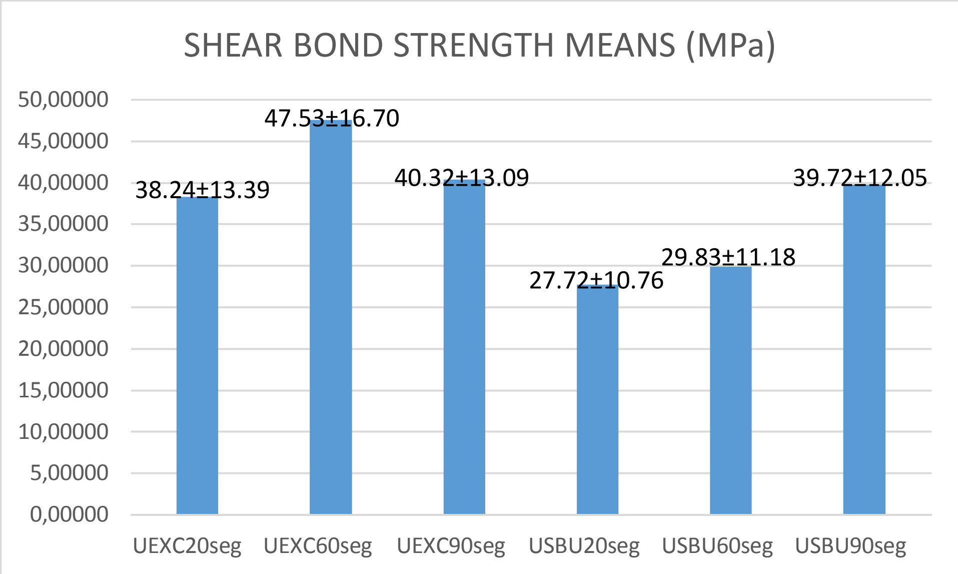
Shear bond strength

Means, standard deviations and statistical differences between the groups for the shear bond strength test are shown in **Table 4** and **Chart 1**.

TABLE 4 – Shear bond strength test results.

GROUPS	Shear bond strength (Mpa)
UEXC20s	38.24 ± 13.39^{ab}
UEXC60s	47.53 ± 16.70^{b}
UEXC90s	$40{,}32 \pm 13{,}09^{ab}$
USBU20s	27.72 ± 10.76^{a}
USBU60s	29.83 ± 11.18^{a}
USBU90s	39.72 ± 12.05^{ab}

Chart 1 – Shear bond strength test results



Significant differences were found among the adhesives ($p<.05$), and for the etching times there were no differences ($p=.059$). However, there was a significant interaction between the adhesives and the etching times ($p=.021$). At 60 seconds, the EXC adhesive presented the highest bond strength values (47.53±16.70Mpa). At 20 and 60 seconds, the SBU adhesive presented the lowest ones (27.72±10.76Mpa).

Failure modes were adhesive in almost all specimens (75%). Mixed failures occurred in 27 specimens (22.5%) and cohesive failures occured in 3 specimens (2.5%) (Table 5).

TABLE 5 – Failure type: cylinder/cement.

FAILURE TYPE	NUMBER OF SPECIMENS	PERCENTAGE
COHESIVE	3	2.5%
MIXED	27	22.5%
ADHESIVE	90	75%

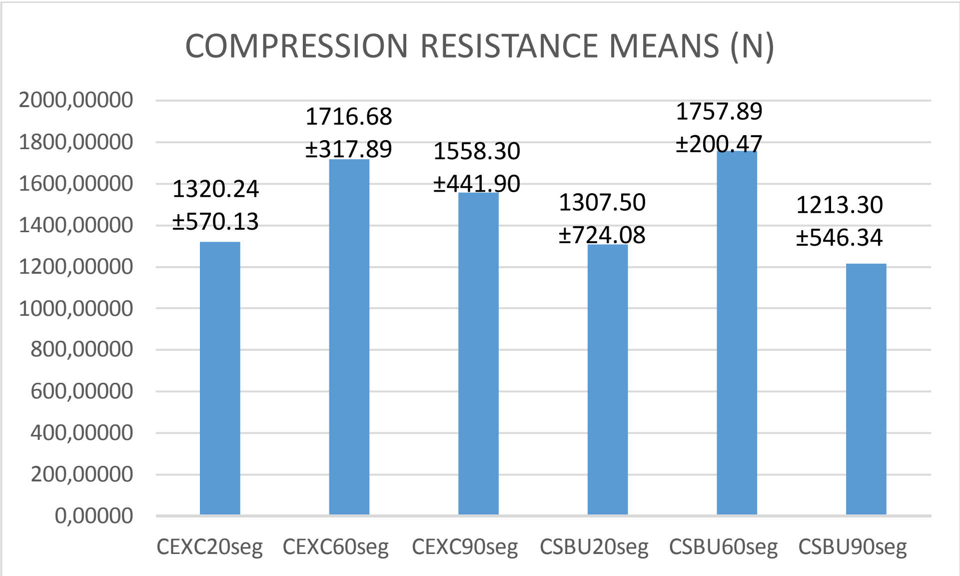
Compression resistance

Means, standard deviations and statistical differences between the groups for the compression test are shown in Table 6 and Chart 2.

TABLE 6 – Compression test results.

GROUPS	Compression (N)
CEXC20s	1320.24±570.13[a]
CEXC60s	1716.68±317.89[a]
CEXC90s	1558.30±441.90[a]
CSBU20s	1307.50±724.08[a]
CSBU60s	1757.89±200.47[a]
CSBU90s	1213.30±546.34[a]

Chart 2 – Compression test results.



There were no significant differences between the adhesives (p=.571) and the times (p=.154). The group that presented the highest values of compressive force was the SBU adhesive at 60 seconds (1757.89±200.47N). However, the one with the lowest values was also the SBU adhesive, but at 90 seconds (1213.30±546.34N).

DISCUSSION

The first null hypothesis was rejected because of the significant difference shown when the adhesives were compared with each other. The second null hypothesis was accepted due to the absence of significant differences between the factors of variation (time x adhesive).

Pre requisite for the success of ceramic restorations is the appropriate and long-lasting bonding between the ceramic and the dental substrate[14]. Currently, it is recommended that leucite reinforced ceramics be pretreated with hydrofluoric acid and in sequenced silane application[15]. Hydrofluoric acid attacks the glassy phase of the ceramic, creating microporosities and dissolving the surface to a micrometric depth[16]. The silane, in turn, presents a bifunctional characteristic that promotes a chemical interaction between the silica and the methacrylate groups of the resinous materials through the formation of siloxane bonds[7]. Therefore, the selection of the appropriate cementation protocol plays a crucial role in achieving long-lasting and clinical[17].

It is important to emphasize that the objective of this study was to simulate a clinical situation experienced by professionals, which consists in the simultaneous acid etching of several pieces. In this way, the etching time between one piece and another. With the advent of adhesive systems, clinicians often tend to choose more simplified systems, such as universal, in which silane is not needed.

Generally, two methods are used to evaluate shear bond strength: shear bond strength test and tensile test[18]. In this study, the shear bond strength was evaluated by the shear bond strength test, which is commonly performed in specimens ranging from 7 to 28mm^2 of adhesive interface surface[18]. Compared with the micro-shear bond strength test, which is performed on much smaller specimens (below 2mm^2), the area of the specimen is closer to the values of micro-shear (3.1mm^2).

However, both macro and micro tests have received much criticism in the literature due to the non-homogeneous distribution in the adhesive interface of the generated stresses[19]. The choice of the shear bond strength test type and its setting, as well as the distance between the load application and the adhesive interface, affect the stress distribution. The more distant the load is applied from the adhesive interface, the higher the tensile stress generated in the area[18]. In the case of shear stress, tensile stress is higher, suggesting that the tensile forces are responsible for the initial failure of the joint[20,18].

The steel wire test used in this study showed to have a better stress distribution at the edge of the adhesive interface area[21]. However, despite the disadvantages, the shear bond strength test is still widely used and accepted for the preliminary evaluation of bond strength, because it is relatively faster than the tensile test, easier to perform and allows for the use of multiple specimens on a single surface. In addition, in this study, the preparation of specimens smaller than $2mm^2$ would be a more expensive process and it would be difficult to obtain specimens with smaller surface areas by the technique of lost wax and injection.

Different treatment options of the ceramic surface have been tested. One of them is the application of silane without pretreatment of the glass ceramics with hydrofluoric acid and air-dried at 100 °C for 1 minute, reporting a significant increase of the bond strength in the tested group. The highest reported mean was ± 28.5MPa, values that are lower than those found in this study and many others[22]. Another tested alternative was the variation of ceramic etching time. It was concluded that, at a concentration of 9.6% acid and a time of 4 minutes, the bond strength was lower compared with the other groups with lower etching time and higher concentration.

Further attempts in the ceramic cementation field demonstrate that the ceramic component etching step is critical for good bond strength results and that the change in the ceramic surface etching time based on lithium disilicate increased the surface roughness, but did not demonstrate a significant difference in bond strength between 10s, 20s and 60s. In addition, it presented a slight decrease of the compressive strength in the greatest etching time[10]. Based on this, it is clear that the findings of the present study show that the time of 60 seconds, recommended by the ceramic manufacturer, is efficient, without the need to increase or decrease it. At 60 seconds there is no compromising of the properties of the ceramic.

Recently, the focus of ceramics research is the use of the universal adhesive system, in which is present in its silane formulation and a functional monomer, such as 10-methacryloyloxydecyldi-hydrogen phosphate (MDP). Its great repercussion is due to its versatility, which promises a new and simplified strategy for union between resins and ceramics[23]. These monomers can interact chemically with the ceramic surface, showing promising results to shear bond strength. However, it tends to decrease after aging treatments of the adhesive interface, causing a hydrolytic degradation in the area.

In this study, the groups that were treated with the universal adhesive system (SBU) presented the lowest bond strength values. This may be related to the complex chemistry in the composition of this adhesive material. However, in the groups where the silane adhesive system was separately used, they presented superior results. High resistance values may be related to the presence of the dimethacrylate monomer, which establishes chemical bonds with the methacrylate present in the resin cements, improving the bond strength[24].

Regarding the compression test, there was no statistical difference between the tested groups, regardless of the acid etching time of the ceramic and the adhesive treatment. It can be stated that even in the longest acid etching time, which would be the simulation of the worst clinical situation tested, the compressive strength, in spite of having the lowest resistance values, was not significantly affected in relation to the other times tested.

Similar results of mechanical properties of leucite reinforced ceramics have already been shown indicating that leucite is a resistant ceramic[25,26].

The appropriate surface treatment for leucite reinforced ceramics was obtained with the application of 10% hydrofluoric acid for 60 seconds without the need to increase or decrease this time, as recommended by the manufacturer.

For further studies, it would be interesting to improve the bond strength test, such as the use of the microtensile test, and to make the specimens aging be different from the results found in this study.

CONCLUSION

According to the results of this study, it can be concluded that the silane associated with the ExciTE F DSC adhesive showed the highest values of bond strength in the etching time of 60 seconds with 10% hydrofluoric acid, without compromising the compressive strength of the leucite reinforced ceramic.

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