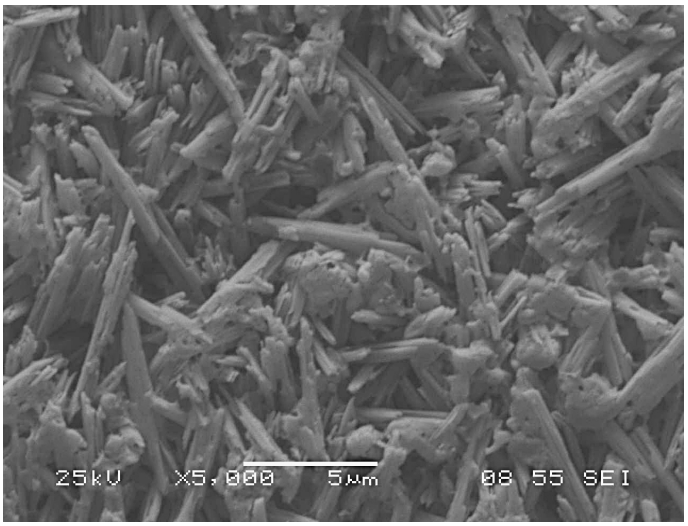
A STUDY INTO MECHANICAL, AESTHETIC AND ADHESIVE ASPECTS OF LITHIA SILICA-BASED GLASS CERAMICS



Riccardo Fabian Fonzar

Università di Firenze

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COORDINATORE
Prof.ssa Gabriella Pagavino

"A study into mechanical, aesthetic and adhesive aspects of lithia silica-based glass ceramics"

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Dottorando

Dott. Riccardo Fabian Fonzar

Tutori

Prof. Marco Ferrari

Prof. Alessandro Vichi

Coordinatore

Prof.ssa Gabriella Pagavino

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...to my Mother, for whom I feel a love that is deeper than words can ever express...

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Chapter 1: Introduction

1.1 General introduction

In the ongoing trend toward more esthetically-pleasing and biologically-compatible restorations, ceramics have gained significant popularity over the last decade (Beuer et al., 2009). The high in-vivo stability, together with the excellent estetic and optical properties, explain their widely use among the clinicians (O'Brien, 1985). Their development is aimed to extend their clinical applications from the partial- and full-coverage single-unit restoration to the fixed partial dentures (Kurbad, 2002). To resist the masticatory loads, some mechanical parameters, such as the flexural strength, have been improved by structural modifications in the material composition (Sedda et al., 2014; Vichi et al., 2013). In fact, the reduction of the glassy matrix in favor of greater crystalline content is associated with more resistant and opaque ceramics.

However, the fracture resistance does not depend on the material composition only. Further physical aspects play a key-role in the success of ceramics. The restoration thickness, together with its superficial texture and cementation technique, contribute to determine the mechanical behaviour (Carvalho et al., 2014; Magne et al., 2015). Proper height, smooth surfaces and adhesively luting procedures prevent the

restoration from failures. Since the material composition influences its mechanical and optical properties, polishability and bonding ability, it was on interest to evaluate these parameters on different lithia silica-based glass ceramics.

This thesis consists of three investigations about lithia silica-based glass ceramics. In the first, the mechanical properties of CAD-CAM and heat-pressed lithium disilicate were compared with regard to the translucency of the material and the processing method. In the second, the efficacy of three finishing and polishing systems was evaluated on the milled lithia silica-based ceramics. In the third, the efficacy of different etching protocols was investigated in terms of bonding ability to the milled lithia silica-based ceramics.

1.2 Classification of dental ceramics

Classification of dental ceramics may be made according to the fusion temperature, fabrication method, crystalline phase and hydrofluoric acid sensitivity.

1.2.1 Classification by fusion temperature

There are four classifications of ceramics according to fusion temperature: ultra-low fusing (<870), low fusing (870-1065), medium fusing (1090-1290), and high fusing (1315-1370). The low fusing interval is utilized for lithia silica-based restorations.

1.2.2 Classification by fabrication method

Classifications according to fabrication method are numerous, and consist of conventional layering, hot pressing, slip casting and machining. All of these are described below.

1.2.2.1 Conventional layering

Conventional layering refers to the technique that is typically employed to layer veneer onto the prosthetic core. Porcelain powder and modeling liquid are mixed according to the manufacturer's instructions and applied to the core with a brush in multiple applications and firings.

1.2.2.2 Heat-pressing

Heat-pressing is used to fabricate monolithic all-ceramic crowns, inlays, onlays and veneers. A wax pattern is produced and then invested in a

refractory die material. The wax is then burnt out to create the space necessary for the injected ceramic ingot. As the glass-ceramic comprises a certain volume of glassy phase, the material can be pressed into the mould using the principle of viscous flow (Plengsombut et al., 2009).

Lithium disilicate ceramic is available on the market as heat-pressable ingots:

- IPS e.max Press is a crystallized lithium disilicate ceramic. Depending on the translucencies (HT, MT, LT, MO, HO), IPS e.max Press ingots are supplied in different shades and two dimensions. By pressing at 920° C, crystals complete their growth and the material is tough enough (400 MPa) to resist the masticatory forces as anterior or posterior monolithic crown.

1.2.2.3 Slip casting

Slip casting is a fabrication technique that involves the utilization of a ceramic slip that is poured into a negative mold of the desired framework, which is typically made of gypsum. When the walls of the mold material wick water away from the slip, powder particles near the walls of the mold become compacted and form a layer of ceramic that will become the framework. After the remaining slip is discarded the framework is removed and infused with molten glass. Veneering porcelain is then applied (Griggs, 2007; Pallis et al., 2004). Some

examples of ceramics that can be used for slip casting are zirconia toughened alumina, alumina-based, and spinel based ceramics.

1.2.2.4 Machining (CAD-CAM)

All-porcelain crowns may be fabricated using computer aided design and computer aided manufacturing (CAD-CAM). Partially- or fully-crystallized ceramic blocks are subtractivelly milled using computer-guided tools.

Different lithia silica-based blocks are available for milling:

- IPS e.max CAD (Ivoclar Vivadent) is a partially crystallized lithium disilicate glass ceramic. At the pre-sintering stage, IPS e.max CAD blocks are supplied in a bluish color and exhibit a flexural strength of 120 MPa to 150 MPa. Two block sizes (I12 and C14), four translucencies (HT, MT, LT and MO), sixteen A-D shades and four bleach BL shades are available for IPS e.max CAD. After crystallization, the flexural strength increases to 360 MPa, which makes it suitable for the fabrication of anterior and posterior crowns.
- VITA Suprinity (Vita Zahnfabrik) is a partially crystallized zirconia-reinforced (approx. 10% by weight) lithium silicate ceramic. Pre-sinterized blocks (C14) are available in two translucencies (HT and T) and seven shades (A1, A2, A3, A3.5,

B2, C2 and D2). Once sinterized, the fine-grained microstructure improves the flexural strength from 180 MPa to 420 MPa, which allows the material to be used for anterior and posterior crowns, suprastructure on implants abutments, veneers, inlays and onlays.

- CELTRA CAD FC (Dentsply) is a fully crystallized zirconia-reinforced (approx. 10% by weight) lithium silicate ceramic. Blocks (C14) are available in two translucencies (HT and LT) and five shades (A1, A2, A3, A3.5, B2). By glazing, the milled material exhibits a flexural strength of 370 MPa, thus it can be used for anterior and posterior crowns, suprastructure on implant abutments, veneers, inlays and onlays.

A thermal cycle is mandatory for IPS e.max CAD and VITA Suprinity (Bischoff et al., 2011; Plengsombut et al., 2009). Heating at approximatelly 840-850°C allows the immature crystals to reach the desired volume and dimension. This last step is known as crystallization and plays an important role in defining the mechanical and optical characteristics of lithia silica-based ceramics, since either the flexural strength or the translucency are controlled by the crystals' shape, dimension and content (Chung et al., 2009).

VITA Suprinity and CELTRA CAD FC represent the newest generation of zirconia-reinforced lithia silica-based glass ceramics with improved

fracture resistance through crack interruption (Aboushelib & Sleem, 2014).

1.2.3 Classification by crystalline phase

1.2.3.1 Predominantly glassy ceramics

Feldspathic porcelain, so named because of the presence of silica- and alumina-based feldspar, has the most tooth-like appearance, making it the most esthetically-pleasing ceramic for dental restorations. The primary weakness of feldspathic porcelain is its low flexural strength (Kelly, 2004).

1.2.3.2 Particle-filled glasses

It is possible to improve the mechanical properties of feldspathic porcelain-based glass ceramics by adding crystalline fillers such as leucite, crystalline mica or lithia-silicate crystals to the glass. With the addition of lithium silicate or disilicate crystals to the glass phase, the ceramic become more resistant despite the esthetic is maintained unchanged.

The first lithium disilicate glass ceramic available into the market was IPS Empress II (Ivoclar Vivadent AG, Schaan, Liechtenstein). Its crystals content, size and distribution ensured higher flexural strength (360 MPa) and fracture toughness when compared to leucite-reinforced and low fusing conventional ceramics (Drummond et al., 2000; Höland et al.,

2000; Oh et al., 2000). Despite that, the clinical applications were considered to be the same of IPS Empress I with the addition of 3-unit FPDs extended at maximum to the second premolar (Albakry et al., 2003; Conrad et al., 2007).

The IPS Empress era ended as soon as IPS e.max Press (Ivoclar Vivadent) was launched into the market in 2005 (Conrad et al., 2007). This new lithium disilicate glass-ceramic consists of approximately 70% fine-needle-like lithium disilicate crystals (Li_2Si2O_5) embedded in a glassy matrix. Its excellent clinical performances are comparable to metal-ceramics (Gehrt et al., 2013; Kern et al., 2012; Pieger et al., 2014; Silva et al., 2011; Zhang et al., 2013), insomuch as the multiple applications of IPS e.max encompass single anterior and posterior full-coverage restorations, single anterior and posterior partial-coverage restorations, anterior FPDs extending to premolars, Meryland bridges in anterior region and veneers (Guess et al., 2013; Silva et al., 2011; Sun et al., 2013).

1.2.3.3 Polycrystalline ceramics

Polycrystalline ceramics are stronger and tougher than glass ceramics because of the lack of glassy components. Aluminum and zirconium oxide are classified as single-phase materials free of glass. Despite the strength, high crystalline content makes polycrystalline ceramics rather

opaque, thus their use should be limited to substructures. Recently, high-translucent zirconia were brought to the market for monolithic multi-units restorations but the optical properties are still far away from the optimal esthetic of glass ceramics (Vichi et al., 2014).

1.2.4 Classification by hydrofluoric acid sensitivity

In order to achieve the desired bonding between the intaglio surface of all-ceramic crowns and the tooth structure, a strong resin bond is required (Buonocore, 1955; Della Bona et al., 2004; Fusayama et al., 1979). Unlike policrystallines, silica-based ceramics are sensitive to the hydrofluoric acid action (Borges et al., 2003; Salvio et al., 2007; Spohr et al., 2003). Its application leads to the dissolution of the glassy matrix and the exposure of the crystalline phase. The surface becomes rough and micro-retentive and the luting agent can penetrare into the irregularities to form an interlocked complex, which improves the bonding ability of silica-based ceramics to resin cements (Roulet et al., 1995).

1.3 CAD-CAM technology in dentistry

The development of ceramic materials for dental applications was strictly correlated to the development of new processing technologies (Lee et al., 2008). The computer-assisted manufacturing was already present in different industrial fields and during the seventies was modified and applied to dentistry (Andersson et al., 1996; Aoki et al., 1986). The acronyms CAD-CAM indicating "Computer Aided Design - Computer Aided Manufacturing" was firstly introduced by Dr. Duret with the Sopha System (Duret et al., 1991). This system consisted of an optical impression, a software for the restoration design and a milling machine (Duret et al., 1988).

The first CEREC, whose name derived from "CERamic REConstruction", was introduced in 1980 by Dr. Mörmann at the University of Zurich. This system was composed by an intraoral camera for optical impression, a 2D design software for the functionalization of a single restoration in maximum intercuspidation, and a compact milling machine for the pre-sintered feldspathic blocks processing. In 1986 Siemens brought to the market CEREC system in combination with VITA Mark I feldspathic blocks. This system, which was considered as "Chairside", offered the innovative possibility to fabricate the in-office prosthetic restoration in a single appointment. In 2003 with the

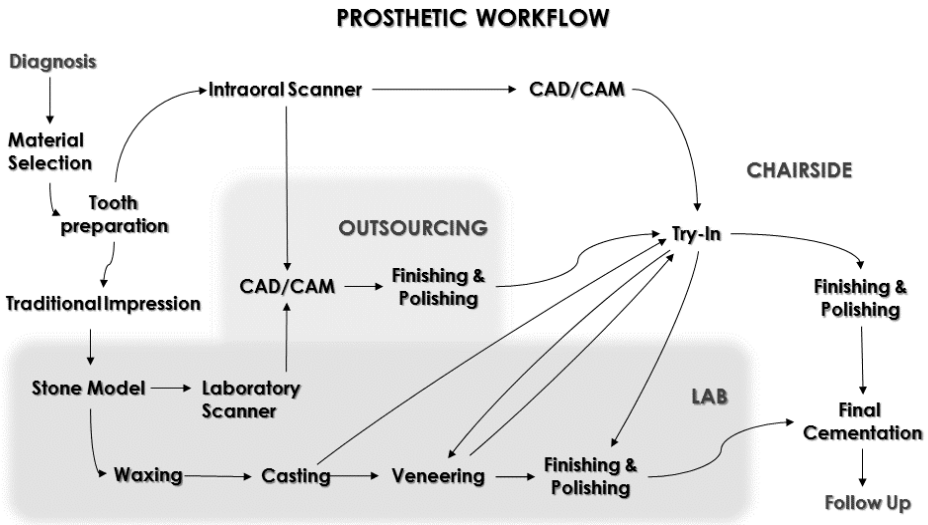
development of the 3D software and the processor, the chairside system was integrated with optical laboratory scanners based on the intraoral camera technology (Mörmann, 2006).

Parallel to the chairside technology, Dr. Andresson introduced a new CAD-CAM system for the metal alloys milling. Procera system was born as an economic alternative to the conventional precious alloys used in prosthodontic up to that moment. Due to the low biocompatibility of the first nickel-chrome alloy, new metals based on titanium, densely sintered alumina and chrome-cobalt were employed. The system basically consisted of three steps. The gypsum model was firstly scanned with a laboratory optical scanner. Afterwards, the substructure was digitally designed and sent to a delocalized milling center. After milling, the substructure was sent back to the laboratory for finalizing (Andersson et al., 1993). This centralized system was defined as an “Outsourcing” system (McLaren et al., 2002).

Since the eighties, the commercialized CAD-CAM dental systems have mainly followed the "chairside" and the "outsourcing" workflows (Poticny et al., 2010). Henceforward, industries have invested always more resources in the CAD-CAM technology and improved systems are nowadays available for multiple applications in fixed and removable prosthesis (Boitelle et al., 2014; Lima et al., 2014), maxillofacial and

implant surgeries (Vercruyssen et al., 2014; Wilde et al., 2014), and orthodontic therapies (Kwon et al., 2014).

The first application of CAD-CAM technology aimed to redefine the traditional prosthetic workflow preserving or improving the restorations quality (Torsello et al., 2008).



Several advantages result from the digital workflow (Fasbinder, 2013; Liu, 2005):

- Reduced operative time since restorations are quickly designed and fabricated.
- High predictability since it is a less operator-sensitive process.

- Larger material spectrum, especially for outsourcing.
- Use of industrially produced stable materials.
- Low materials waste.
- Reproducibility.
- Cheaper cost.
- Rapid learning curve.

Among the disadvantages of CAD-CAM systems:

- High costs for the in-office system purchase.
- The need for continuous software updating.
- Lack of long-term clinical trials for full arch restorations.
- Production parameters uncontrolled for outsourcing.

1.4 In-office CAD-CAM systems

The advantages of CAD-CAM systems in terms of reduced production-costs and working-time are emphasized in "chairside" systems (Miyazaki et al., 2011). These systems have become less expensive, more practical and precise, improving the clinical performance of the restorations (Bernhart et al., 2009; Bindl et al., 2003; Fasbinder, 2013). Besides CEREC (Sirona, Bernsheim, Germany) other CAD-CAM systems are available for the in-office workflow, which confirms the increased interest of the clinicians toward this technology: KaVo ARCTICA (KaVo Dental GmbH, Biberach, Germany), Planmeca Planscan/Planmill (Planmeca Oy, Helsinki, Finland), Carestream CS solutions (Carestream Dental, Atlanta, GA, USA). These systems basically consist of an intraoral scanner, a software for designing and a milling unit.

At the present time, Sirona CEREC System offers two different intraoral scanners. The first one is CEREC Bluecam which was introduced in 2009. Its optical reading technology is powder-dependent and based on short-wave blue light. Single pictures are combined for the 3D model reconstruction, thus its use is indicated for scanning up to a quadrant. The second one is CEREC Omnicam which was introduced in 2012. Its optical reading technology is powder-free and based on continuous data acquisition for the 3D model fabrication. Even though no higher

precision is reported compared to the CEREC Bluecam, CEREC Omnicam is also indicated for full-arch scanning.

Despite conventional impressions still remain the gold-standard for the full-arch reproduction (Ender & Mehl, 2013; Nedelcu & Persson, 2014), traditional and digital workflow exhibit similar repeatability and accuracy in replicating short-span prosthesis (Ting-Shu & Lian, 2014). The main advantage of intraoral scanning is related to the more comfortable experience and the more efficient workflow for either the patient and the dentist (Fasbinder, 2010; Patzelt et al., 2014).

Softwares are divided in two categories depending on the processing file. Closed-files are processed with the proprietary software only (CEREC InLab SW 4.3), whilst STL open-files do not need any exclusive software (KaVo multiCAD, Planmeca PlanCAD, the Carestream CS Restore). Thanks to the virtual articulator and the digital smile design, CEREC 4.2 software allows the operator to design and virtually simulate the prosthetic treatment with different restorative solutions.

Once the gypsum model is scanned and the restoration is digitally waxed-up, the proper restorative material can be selected for the milling process. CEREC MC-XL is four-axial milling system with four electric motors and four water-cooled burs able to mill up to 12-units zirconia

bridge. Its widely recognized application regards the fabrication of single tooth- or implant-supported monolithic restorations.

Since the milled restoration has to be crystallized and finalized before delivering, lithia silica-based ceramics are not considered as pure in-office materials. Thus, finishing and luting procedures were evaluated to better understand the polishability and the bonding ability of lithia silica-based restorations.

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Chapter 2: Mechanical properties

2.1 Flexural resistance of heat-pressed and CAD-CAM lithium disilicate with different translucencies.

Introduction

Metal-free restorations are largely used for prosthodontic rehabilitations. As a result of the increased esthetic demand, ceramic biomaterials have become more popular in the daily practice (Vichi et al., 2011). According to their composition, ceramic biomaterials can essentially be divided into two categories: glass- and polycrystalline-ceramics. As a general rule, glass matrix bestows esthetic characteristics, whereas crystals improve mechanical properties (Fischer et al., 2008).

Since the introduction by McLean & Hughes (McLean, 1965) of the first high strength ceramic in which alumina (Al_2O_3) was added to feldspathic porcelain, research has pursued the objective of obtaining an ideal restorative material, which would combine natural tooth-like appearance and high mechanical performances (Höland et al., 2006). With this aim lithium oxide was added to the glass matrix, and a new material based on SiO_2-Li_2O-P_2O_5-Al_2O_3-K_2O-ZrO_2 was used to fabricate dental restorations (Bischoff et al., 2011).

Among the lithium disilicates, IPS e.max Press and IPS e.max CAD are available for the heat-pressed and CAD-CAM technique respectively. For IPS e.max Press, ingots are already crystallized. By heating, the ingots become viscous and pressable (Plengsombut et al., 2009). Conversely, for IPS e.max CAD the blocks exhibit an intermediate status (Li_2SiO_3), necessary for the milling procedures. After milling, the blocks undergo a heat-mediated chemical reaction, resulting in the lithium disilicate crystallization ($Li_2Si_2O_5$) (Bischoff et al., 2011; Plengsombut et al., 2009). This crystallization process consists of two major events, nucleation and crystals growth (Apel et al., 2007; Huang et al., 2015). Due to the nucleating agents, the reaction is controlled and the final crystals shape, size, and content are determined (Wang et al., 2010). By using different concentrations of different agents, the microstructure is modified and the glass ceramic changes its mechanical and esthetic properties (Anusavice & Zhang, 1997; Hasselman & Fulrath, 1966). Particularly, in order to render the desired shade and translucency, some oxides are used, acting as co-nucleating agents. These oxides interact with the described nucleation and crystallization processes, thus affecting the size of the crystals and, consequently the mechanical and physical properties (Anusavice et al., 1994a; Anusavice et al., 1994b). It can therefore be expected that for IPS e.max changes in translucency reflect

into modifications of the mechanical properties. Nevertheless, the information provided by the manufacturer does not discriminate on this regard.

Then, it seemed of interest to assess whether IPS e.max Press and IPS e.max CAD with different translucencies perform differently in terms of flexural strength.

Particularly, a study with a twofold objective was conducted. The first objective was to verify the manufacturer's claim that IPS e.max Press has higher flexural resistance than IPS e.max CAD. The tested null hypothesis was that IPS e.max Press and CAD measure similar flexural strengths. The second objective was to assess the flexural resistance of specimens of IPS e.max Press and IPS e.max CAD with different translucencies. The tested null hypothesis was that no statistically significant difference in flexural strength exists among the different translucencies available for IPS e.max Press or CAD.

Materials and methods

Specimen preparation

For the heat-pressed technique (Group A), acrylate polymer blocks (IPS AcrylCAD, Ivoclar Vivadent, Schaan, Liechtenstein) were perpendicularly cut with a low-speed water-cooled diamond saw (ISOMET 1000, Buehler, Lake Bluff, Illinois), in order to obtain 60 bar-shaped specimens with dimensions of 4.0 in width, 1.2 mm in thickness and 16.0 mm in length (ISO 6872:2015). The specimens were randomly divided into 4 subgroups (n=15).

Sprueing, investing, preheating, pressing, and finishing procedures were carried out according to the manufacturer's recommendations. In particular, for each press cycle, 6 specimens were fixed to the ring base (IPS Investment Ring System 200 gr., Ivoclar Vivadent, Schaan, Liechtenstein) with a 3-mm long, 3-mm diameter extra-smooth wax wire (S-U-Wax wire colourless, Schuler-Dental, Ulm, Germany). Bars were kept at least 3 mm away from each other and oriented in such a way that a distance of 10 mm from the silicone ring was ensured laterally and upwards. The hole on the ring's base was finally filled with red modelling wax (Tenatex, Kemdent, Wiltshire, United Kingdom). The investment procedure were there carried on following manufacturer's instruction and then the selected IPS e.max Press ingot (Ivoclar Vivadent,

Schaan, Liechtenstein) was processed for pressing. Based on the translucency of the pressed ingots, 4 subgroups were defined: Subgroup A.1 = HT (High Translucency) shade A3; Subgroup A.2 = MT (Medium Translucency) shade A3; Subgroup A.3 = LT (Low Translucency) shade A3; Subgroup A.4 = MO (Medium Opacity) shade 2. After cooling, rough and fine divestments (Microjet, Simed, Baranzate, Italy) were carried out using polishing beads, respectively at 4-bar and 2-bar pressure. The reaction layer was removed by submerging specimens in a <1% hydrofluoric acid ultrasonic bath (IPS e.max Invex Liquid, Ivoclar Vivadent, Schaan, Liechtenstein) for 15 min, followed by polishing beads at 2 bar pressure. Finally, specimens were separated by cutting the base of the sprue with a low-speed water-cooled diamond disc.

For the CAD formulation (Group B), 4 lithium disilicate blocks (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were selected: Subgroup B.1 = HT A3; Subgroup B.2 = MT A3; Subgroup B.3 = LT A3; Subgroup B.4 = MO 2. Specimens (n=15) were obtained by cutting the blocks with the low-speed water-cooled diamond saw. Firing paste (IPS Object Fix Putty, Ivoclar Vivadent, Schaan, Liechtenstein) was used to avoid contact of the specimen with the firing tray. The final crystallization was performed with the EP 600 Combi furnace following manufacturer's instructions.

Both CAD-CAM and heat-pressed specimens were polished and finished with water-cooled silica carbide papers of #600, #1200 and #2400 grit, until obtaining the desired dimensions of 4.0 ± 0.2 mm in width, 1.2 ± 0.2 mm in thickness and 16.0 ± 0.2 mm in length (ISO 6872:2015). A 45° edge was made at each major sharp edge, by keeping the specimens at 45° with metal tweezers (ISO 6872:2015).

Test method

A three-point bending test (3PBT) appliance was used (ISO 6872:2015; Sedda et al., 2014; Vichi et al., 2013). A support milled from a stainless steel block (A.I.S.I. type 316L), with two Cobalt-HSS (high speed steel) roller supports (∅ 2 mm, 13.00 span width) was used. Tests were performed in a universal testing machine (Triax 50, Controls, Milano, Italy), equipped with a Cobalt-HSS (high speed steel) loading tip, and operating at a cross-head speed of 1 mm/min. Specimens were tested in dry conditions and at room temperature. The fracture load was recorded in N and the flexural strength (σ) was calculated in MPa by using the following equation:

$$\sigma = 3Pl / 2wb^2$$

where: P in the fracture load in N, l is the span (distance between the center of the supports) in mm, w is the width in mm, and b is the height in mm of the specimen.

The Weibull characteristic strength (σ_0) and the Weibull modulus (m) were calculated according to the following equation:

$$P_f = 1 - exp[-(\sigma/\sigma_0)^m]$$

where: P_f is the probability of failure between 0 and 1, σ is the flexural strength in MPa, σ_0 is the Weibull characteristic strength in MPa (the value at the 63.2% of the specimens fail), and m is the Weibull modulus.

Statistical analysis of flexural strength data

In order to test the first formulated null hypothesis, all the Pressed specimens (Group A) were cumulatively compared with all the CAD specimens (Group B), using the t-test for Independent Samples, having verified that in either group data distribution was normal (Kolmogorov-Smirnov test), and variances were homogeneous (Levene test).

In order to test the second formulated null hypothesis, within each group a Kruskall-Wallis Analysis of Variance, followed by the Dunn's Multiple Range test for post hoc comparisons, was run to compare the flexural strengths measured by the different available translucencies (Subgroups A.1-A.4; Subgroups B1-B4). The choice of non-parametric tests was

dictated by the finding that the data did not meet the requirement of normal distribution according to the Kolmogorov-Smirnov test.

In all the analyses the level of significance was set at $\alpha = 0.05$ and PASW Statistic 18.0 software (SPSS, Chicago, IL, USA) was used.

SEM evaluation

IPS e.max Press and IPS e.max CAD were qualitatively evaluated before and after the furnace-mediated heat treatment. Specimens (n=2) were polished and finished with water-cooled silica carbide papers of #600, #1200 and #2400 grit, etched for 60 seconds with 4.9% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent AG, Schaan, Liechtenstein), rinsed out with running water for the acid removal, ultrasonically vibrated in a 95% alcohol solution for 3 minutes (CP104, CEIA, Italy), and air dried with an oil-free stream. Each bar was secured to SEM (JSM-6060LV, JEOL, Tokyo, Japan) tabs with gold conducting tape, and gold coated in a vacuum sputter coater (SC7620 Sputter Coater, Polaron Range, Quorum Technologies, Newhaven, UK). Crystals morphology and orientation were observed at x5000 magnifications.

Results

Flexural strength

The mean and standard deviation values of flexural strength (σ), the Weibull characteristic strength (σ_0), and the Weibull modulus (m) are reported in Table 1.

According to the t-test, the overall means of Press and CAD specimens did not differ significantly ($p>0.05$, Table 1). The power of the test was calculated to be 0.80.

With regard to the comparison among translucency subgroups, within the Press group different translucencies were found to have similar flexural strengths ($p>0.05$, Table 1).

Within the CAD group, statistically significant differences emerged among the tested translucencies ($p<0.001$). Specifically, the post-hoc test demonstrated that MT had significantly higher flexural strength than HT and MO. Also, LT exhibited significantly higher flexural strength than MO.

Table 1. Flexural strength (σ), Weibull characteristic strength (σ0) and Weibull modulus (m) of heat-pressed and CAD-CAM lithium disilicate. Different letters label statistically significant differences in flexural strength.

Materials		σ					m	σ0
		Mean	SD	Median	Interquartile range	Sig. p<0.05		
PRESS[A] Mean 344.35 SD 65.94	HT	316.91	60.38	299.41	274.99-349.69	a	6.39	340.32
	MT	379.70	76.26	374.51	310.17-452.60	a	5.89	409.63
	LT	316.48	35.32	327.63	283.26-341.25	a	10.45	331.93
	MO	364.32	64.88	353.86	310.61-425.72	a	6.68	390.22
		σ					m	σ0
		Mean	SD	Median	Interquartile range	Sig. p<0.05		
CAD[A] Mean 345.74 SD 68.00	HT	346.21	35.14	315.20	306.46-358.78	bc	9.21	359.12
	MT	397.46	62.61	402.50	368.23-431.97	a	7.40	423.39
	LT	381.04	42.02	364.07	352.91-401.14	ab	11.65	398.66
	MO	281.19	47.94	254.21	240.55-296.99	c	6.77	298.11

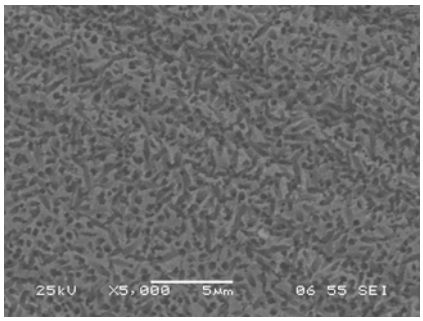
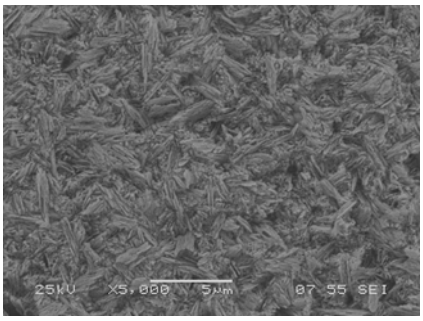
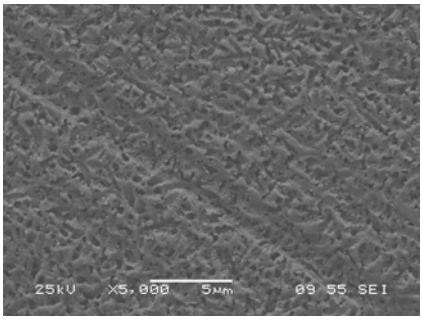
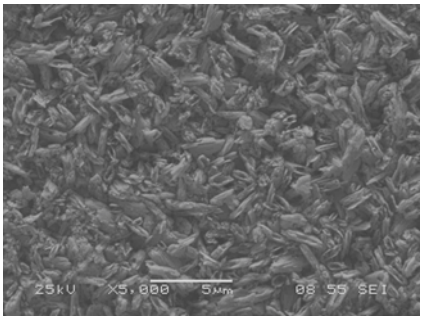
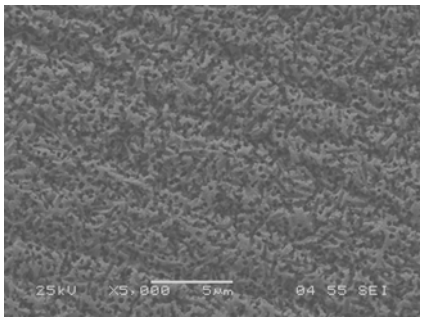
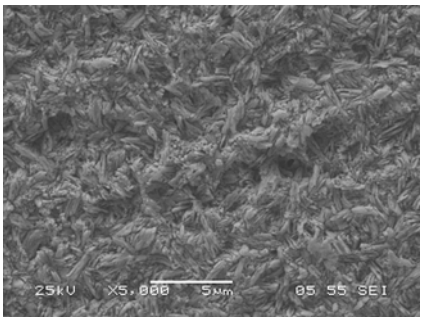
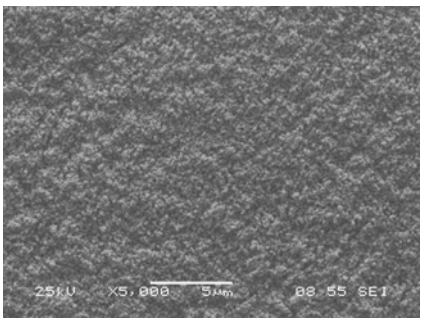
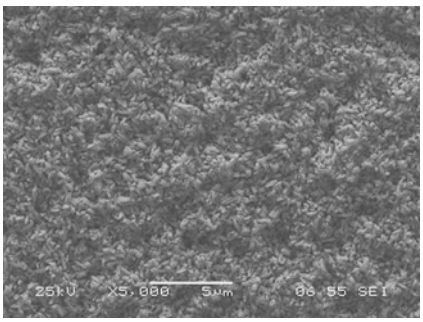
SEM evaluation

For Group A, crystals did not vary in dimension and orientation within the tested translucencies. However, the elongated crystals became much longer after heat-pressing.

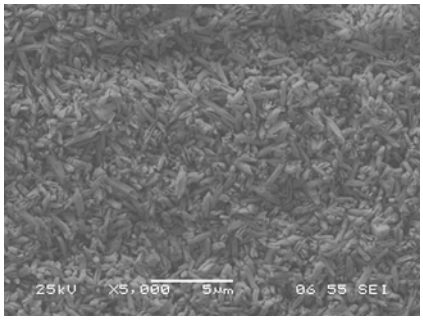
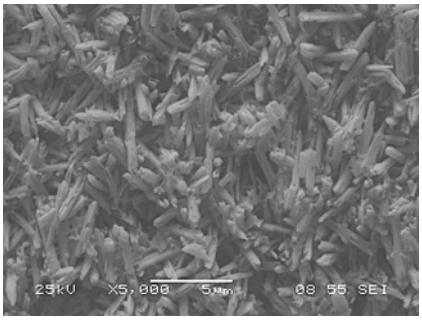
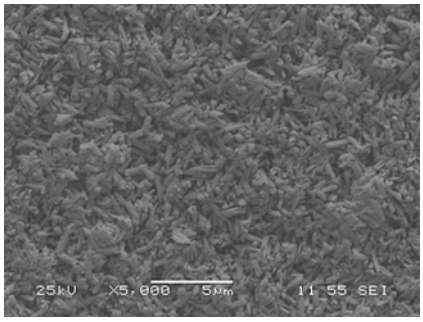
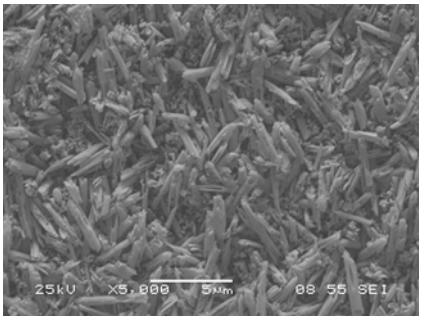
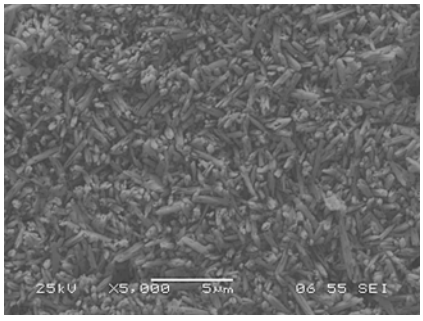
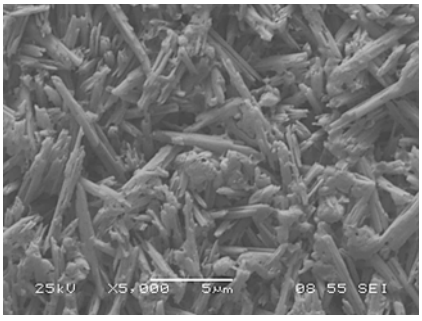
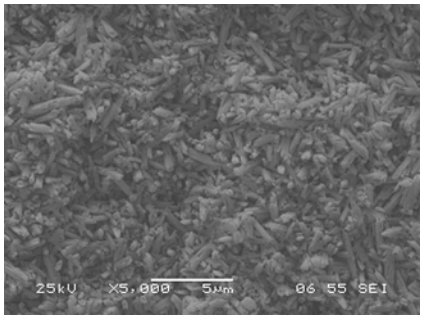
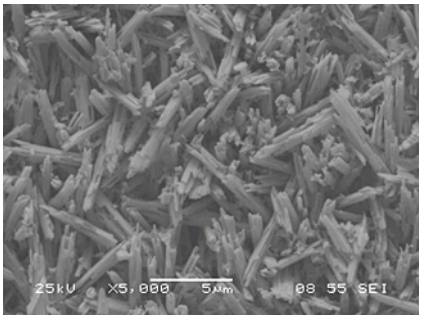
For Group B, the pre-crystallized specimens showed the presence of metasilicate crystals dispersed into an amorphous phase. By increasing the opacity of the material, the amount of metasilicates increases, and the glassy matrix decreases. After processing, crystals became longer even if smaller than those observed for IPS e.max Press. Unlike the other

translucencies, IPS e.max CAD MO showed shorter densely-distibuted round-shaped crystals.

IPS e.max CAD

Pre-crystallization		Post-crystallization
	**HT**	
	**MT**	
	**LT**	
	**MO**	

IPS e.max Press

Pre-pressing		Post-pressing
	**HT**	
	**MT**	
	**LT**	
	**MO**	

Discussion

No statistically significant difference was found between Press and CAD material formulations, therefore the first null hypothesis was accepted. Conversely, based on the outcome of the statistical analysis, the second null hypothesis had to be accepted for IPS e.max Press and rejected for IPS e.max CAD.

IPS e.max Press is marketed in 5 different translucency degrees, high translucency (HT), medium translucency (MT), low translucency (LT), medium opacity (MO) and high opacity (HO). In IPS e.max CAD, HO opacity is not present, therefore IPS e.max Press HO was not included in the study.

The finding of a relatively low flexural strength for the MO translucency in the CAD group is in accordance with a previous study in which a value of 273±52 MPa was recorded for this material (Sedda et al., 2014). A difference in the composition and processing of the two formulations can account for the outcome of a superior flexural resistance of MO Press specimens in comparison with the CAD counterpart.

Whilst for IPS e.max Press the controlled crystallization is industrially accomplished, for IPS e.max CAD it consists of two crystallization processes. The first one is industrially mediated and yields the materials to the metasilicate phase (Li_2SiO_3). The second one is completed in the

clinic or in the laboratory. By heat treatment, metasilicate phase dissolves and lithium disilicate crystallizes ($Li_2Si_2O_5$) (Zheng et al., 2008). This reaction is controlled by some nucleating agents (Fernandes et al., 2008; Headley & Loehman, 1984). At high concentrations of agents, the microstructure is denser and crystals become smaller and spherically-shaped (Wen et al., 2007). This "round" morphology does not allow the crystals to form an interlocked microstructure (Cramer von Clausbruch et al., 2000), thus the mechanical properties of the ceramic decrease (Thompson et al., 1995). At the same time, the higher density makes the material more opaque as the light scattering is reduced (Anusavice et al., 1994; Vichi et al., 2011; Vichi et al., 2014). Although the opacity can be improved by adding some pigments to the glass frit without modifying the flexural strength of the ceramic (Anusavice et al., 1994; Yuan et al., 2014), the final result seems to be more dependent on the volume and the size of the crystals than on the effect of a specific compound added on purpose. Thus, the nucleating agents proportions might be essential to determine the translucency degree and the resistance of the glass ceramic. The high density of the crystalline phase, along with the reduced dimension of the crystals, might explain the lower mechanical performances obtained for IPS e.max CAD MO. Further investigations are needed to confirm this hypothesis.

Unlike IPS e.max CAD MO, IPS e.max Press MO showed flexural strengths similar to the other 3 translucencies. The high crystalline content, together with the disposition of the crystals along the pressing direction, might explain the capability of IPS e.max Press MO to combine a higher degree of opacity with mechanical performances similar to those of the more translucent formulations.

In the present investigation the values obtained for flexural strength were generally below those claimed by the manufacturer, i.e. 400 MPa for IPS e.max Press and 360 MPa for IPS e.max CAD. Although the manufacturer declares a crystalline volume of approximately 70% for both IPS e.max Press and IPS e.max CAD, the two materials show different crystals distribution and size. According to Oh and Zhang (Oh et al., 2000; Zhang et al., 2013) for IPS e.max Press crystals are ≈ 4 μm in length, ≈ 0.6 μm in width, and about parallel, whereas for IPS e.max CAD they are ≈ 1 μm in length, ≈ 0.4 μm in width, and more randomly oriented. This interlocking microstructure of club-like crystals, together with their alignment along the direction of pressing (Denry & Holloway, 2004), is reported to play an important role in hindering crack propagation, thus improving the flexural strength of pressable lithium disilicate (Cramer von Clausbruch et al., 2000; Höland et al., 2000).

However, this statement is not confirmed by the outcome of our study, where the overall performance of Press was not superior to that of CAD.

Few data are available in the literature for a direct comparison with this study's outcome. For CAD formulation, the flexural strengths recorded in the present study are similar to those reported by Sedda et al. (Sedda et al., 2014) (336.06 ± 40.09 for HT, 376,85 ± 39.09 for LT and 272.61 ± 51.95 for MO) and by Lien et al. (Lien et al., 2015) (367 ± 44 MPa). In the latter, the opacity of IPS e.max CAD tested is not indicated. Concerning with Press formulation, Xiaoping et al. (Xiaoping et al., 2014) reported for IPS e.max Press HT higher flexural strength (384 ± 33 MPa) than that obtained in the present investigation (316 ± 60 MPa). However, in general the data set of the present study falls within the flexural strength range (251 ± 30 MPa - 407 ± 45 MPa) (Albakry et al., 2003; Albakry et al., 2004; Cattell et al., 2002; Höland et al., 2000; Lien et al., 2015; Nakamura et al., 2002; Oh et al., 2000; Sedda et al., 2014; Xiaoping et al., 2014) found in the literature for lithium disilicate glass ceramic non exclusively related to e.max (Table 2).

Table 2. Flexural strength of IPS Empress 2, IPS e.max Press and IPS e.max CAD.

Authors	Material	Test method	Flexural Strength (MPa)
Albakry et al.	Heat pressing IPS Empress 2	Biaxial flexural strength	σ_{BFT} = 407 ± 45 MPa
Albakry et al.	Heat pressing IPS Empress 2	Biaxial flexural strength	σ_{BFT} = 340 ± 40 MPa
Cattell et al.	Heat pressing IPS Empress 2	Biaxial flexural strength	σ_{BFT} = 251 ± 30 MPa
Hölland et al.	Heat pressing IPS Empress 2	Three-point bending test	σ_{3PBT} = 400 ± 40 MPa
Oh et al.	Heat pressing IPS Empress 2	Three-point bending test	σ_{3PBT} = 357 ± 28 MPa
Nakamura et al.	Heat pressing IPS Empress 2	Four-point bending test	σ_{4PBT} = 329 ± 43 MPa
Xiaoping et al.	Heat pressing IPS e.max Press HT	Three-point bending test	σ_{3PBT} = 384 ± 33 MPa (HT)
Sedda et al.	CAD-CAM IPS e.max CAD	Three-point bending test	σ_{3PBT} = 336 ± 40 MPa (HT) σ_{3PBT} = 377 ± 39 MPa (LT) σ_{3PBT} = 273 ± 52 MPa (MO)
Lien et al.	CAD-CAM IPS e.max CAD	Three-point bending test	σ_{3PBT} = 367 ± 44 MPa

The finding of a large standard deviation confirms that the mechanical properties of glass-based materials are markedly affected by the methods and devices used for the test (Wen et al., 2007). It is noticeable that the flexural strength values reported in literature for IPS e.max Press do not show a progress in comparison with IPS Empress 2 data. Indeed, in the evolution from IPS Empress 2 to IPS e.max Press, the crystalline content was improved from 60% to 70%, which should make IPS e.max Press more effective (Oh et al., 2000).

Based on the obtained results, the clinical indications can be outlined with reference to the ISO 6872:2015 specification. All the materials tested except IPS e.max CAD MO were above the threshold of 300 MPa and below that of 500 MPa, thus they meet the ISO requirements for Classes 1, 2 and 3. This means that three-unit (Class 4) and four or more units (Class 5) restorations (ISO 6872:2015), both monolithic and partially or fully covered substructures involving molars, are not recommended. In this view, it should be pointed out that, even if in the present test the flexural strength values were below those declared by the manufacturer, the clinical indications based on the ISO classification would not differ. IPS e.max CAD MO showed in the present investigation flexural strength values below the threshold of 300 MPa and thus should be classified in ISO Class 2. This means that based on

ISO 6872:2015 its clinical use should be restricted to single-unit restorations (anterior and posterior, monolithic or covered). Anyway, the mean value obtained (281 MPa) is close to the threshold, thus it might be speculated that, even if from an experimental viewpoint this combination did not fulfill the ISO Class 3 requirements, from a clinical viewpoint the difference is indeed limited. ISO standard clinical indications in fact refer to the flexural strength values obtained *in vitro*, that is to the mechanical property of the material. Moreover the flexural values reported in the ISO 6872:2015 are not related to a specific flexural test and it is known that the test systems (bi-axial, 3PBT, 4PBT) influence the outcome (Wen et al., 2007). Furthermore, from a clinical viewpoint the thickness has a significant influence on the capability of glass-ceramics to withstand clinical forces and resist the fracture (Sasse et al., 2015). Consequently, the MPa measurement alone, although important to compare the materials from a mechanical viewpoint, gives only a basic information that has necessarily to be correlated with the clinical thickness.

It is also noticeable that the adhesive cementation of lithium disilicate glass ceramic onto the tooth structure reinforces (Fleming et al., 2006) the restoration. It has recently been reported that crowns produced without respecting the minimum occlusal height (Magne et al., 2015; Sasse et al., 2015) are anyway capable to withstand clinical forces. The

relationship between resistance and strengthening after cementation deserves further attention, as it is still controversial particularly concerning the adhesive procedures and cements used. In fact, on the one hand the fracture resistance of CAD-CAM lithium disilicate onlays was seen to be higher using a conventional resin cement (2205.95 ± 515.39 N) rather than a self-adhesive resin cement (1869 ± 593.30 N) (Yildiz et al., 2013). On the other, Esquivel-Upshaw et al. (Esquivel-Upshaw et al., 2008) did not report different fracture rate among adhesive and non-adhesive procedures.

As in the present investigation IPS e.max Press and IPS e.max CAD showed similar mechanical properties, the choice between the two materials should be based on further considerations. Among them precision and workflow are certainly relevant. While some recent studies have reported substantial analogies in precision between the two fabrication modalities (Anadioti et al., 2014; Anadioti et al., 2015), with regard to workflow, heat-pressing has been reported to require more complex working procedures than CAD-CAM, thus resulting in a more time-consuming and expensive technique (Joda et al., 2015; Patzelt et al., 2014).

Conclusion

On the basis of the outcome and within the limitations of this study, the following conclusions can be drawn:

- IPS e.max Press and IPS e.max CAD showed similar flexural strengths. The processing method did not affect the mechanical properties of lithium disilicates.
- Translucency significantly influenced flexural strength only for IPS e.max CAD.

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Chapter 3: Surface properties

3.1 Effect of finishing and polishing on the surface roughness and gloss of lithium disilicate (IPS e.max CAD) and Zr reinforced lithium silicate (VITA Suprinity) glass ceramics for CAD-CAM systems.

Introduction

Computer-aided design and computer-aided manufacturing (CAD-CAM) technology represents an important part of the present-day prosthetic dentistry (Fasbinder & Neiva, 2016). CAD-CAM follows two main streams. On the one hand, digital procedures can be carried on by the technician in the laboratory, with a workflow that can somehow resemble the traditional one. On the other, it can be performed entirely in the office. The so-called "chairside" procedure enables the single-unit restorations to be fabricated and delivered by the dentist in a reasonable time-consuming single appointment. CEREC system, the first and leading system for chairside, was launched in 1985, and since then it has been developed in hardware, software and material options (Mörmann, 2006; Sadowsky, 2006). Among the several materials available (Sedda et al., 2014; Vichi et al., 2013; Wittneben et al., 2009) lithia silica-based

glass ceramics have a relevant place. IPS e.max CAD (Ivoclar Vivadent AG, Schaan, Liechtenstein) plays a leading role while VITA Suprinity (VITA Zahnfabrik, Bad Sackingen, Germany) is a more recent proposal, with a different chemistry but with similar clinical indications. In a general tendency toward monolithic restorations, both silicates are of interest especially for single-unit restorations, as they combine tooth-like appearance and high mechanical performances (Magne et al., 2015; Stawarczyk et al., 2015). Once the block is milled, the restoration is coarse in texture (Corazza et al., 2015; Fasbinder & Neiva, 2016; Song et al., 2015), thus polishing and finishing are mandatory before delivery (Silva et al., 2014). These procedures render the surfaces smoother (Fasbinder & Neiva, 2016) and more lustrous (Lawson & Burgess, 2015), and improve the biocompatibility (Bollen et al., 1996; Oh et al., 2002; Quirynen et al., 1993) of the restoration by minimizing the incidence of biological complication, such as plaque retention and antagonist-tooth wearing. In addition, well-finished surfaces lead to less technical and esthetic problems, thus the material become more tough (de Jager et al., 2000; Lohbauer et al., 2008), glossy (Heintze et al., 2006) and stable in translucency (Awad et al., 2015) and color (Motro et al., 2012). For glass ceramics, finishing and polishing procedures can be manually carried out with a sequence of burs, or by the use of heat-

mediated glazing systems. As manual polishing and glazing differently affect the surface smoothness and appearance of dental ceramic (Brunot-Gohin et al., 2013; Fasbinder & Neiva, 2016; Heintze et al., 2006; Odatsu et al., 2013; Silva et al., 2014) it has been considered of interest to evaluate whether the roughness and the gloss of IPS e.max CAD and VITA Suprinity vary according to the finishing treatments used.

The purpose of the present study was to evaluate *in vitro* the effect of the recommended manual finishing and polishing system, and the dedicated glazing paste and glazing spray on the roughness and gloss of IPS e.max CAD and VITA Suprinity. Particularly, a study with a twofold objective was conducted. The first objective was to verify whether a difference in the ability of decreasing the roughness and increasing the gloss exists between the two materials. The tested null hypothesis was that IPS e.max CAD and VITA Suprinity achieve the same final roughness and gloss on equal finishing and polishing system. The second objective was to test the efficacy of the manual and the furnace-mediated recommended systems to finish and polish IPS e.max CAD and VITA Suprinity. The tested null hypothesis was that no statistically significant differences in roughness and gloss exist among the tested systems for IPS e.max CAD and VITA Suprinity, respectively.

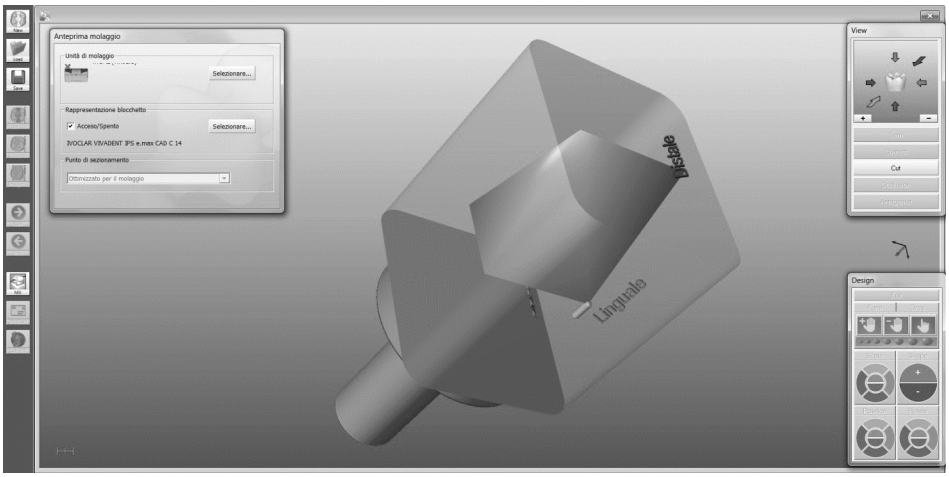
Materials and Methods

Specimen preparation

Pre-crystallized blocks of zirconia-reinforced lithium silicate (ZLS) (VITA Suprinity, HT A3, VITA Zahnfabrik, Bad Sackingen, Germany) and lithium disilicate (LD) (IPS E.max CAD, HT A3, Ivoclar Vivadent AG, Schaan, Liechtenstein) for CEREC® CAD-CAM (Sirona Dental, Bernsheim, Germany) were selected for this study. Twenty-four blocks were used, respectively, for each of the two tested materials.

A model for a 30º wedge-shape specimen was designed with CEREC InLab software v3.88 (Sirona Dental, Bernsheim, Germany) (Figure 1).

Figure 1. Software image of the 30° wedge milled from VITA Suprinty and IPS e.max CAD blocks.



Specimens were milled in a InLab MC-XL milling machine (Sirona Dental, Bernsheim, Germany). In order to standardize to the best extent the milling procedures, the diamond burs of the milling unit (Step Bur 12S, Sirona Dental, Bernsheim, Germany; Cyl. Pointed Bur 12S, Sirona Dental, Bernsheim, Germany) were replaced before starting the milling procedure and every 10 milling cycles. Milled wedges were finally separated from the block's base by means of a low-speed-water-cooled diamond disc. Final crystallization was performed following manufacturer's instructions (VITA Vacumat 6000, VITA Zahnfabrik, Bad Sackingen, Germany).

After crystallization, the 24 VITA Suprinity wedges (Group A) were randomly divided into 4 subgroups according to the finishing procedure (Table 1): Group A.1 = VITA Suprinity Polishing Set Clinical used for 30 seconds (VITA Zahnfabrik, Bad Sackingen, Germany); Group A.2 = VITA Suprinity Polishing Set Clinical used for 60 seconds; Group A.3 = VITA AKZENT Plus PASTE (VITA Zahnfabrik, Bad Sackingen, Germany); Group A.4 = VITA AKZENT Plus SPRAY (VITA Zahnfabrik, Bad Sackingen, Germany). Similarly to VITA Suprinity, the 24 IPS e.max CAD wedges (Group B) were randomly divided into 4 subgroups according to the finishing procedure: Group B.1 = Optrafine Ceramic Polishing System (Ivoclar Vivadent AG, Schaan, Liechtenstein)

used for 30 seconds; Group B.2 = Optrafine Ceramic Polishing System used for 60 seconds; Group B.3 = IPS e.max CAD Crystall./Glaze paste (Ivoclar Vivadent AG, Schaan, Liechtenstein); Group B.4 = IPS e.max CAD Crystall./Glaze spray (Ivoclar Vivadent AG, Schaan, Liechtenstein).

Table 1. Tested Groups, Materials and Treatments.

Groups	**Blocks**	**Finishing & Polishing Systems**	**Treatments and abbreviations**	
A1	VITA Suprinity (ZLS)	SUPRINITY Polishing Set Clinical	Manual finishing and polishing with contrangled handpiece Firs Tip: 30 seconds, 10.000 rpm Second Tip: 30 seconds, 6.000 rpm	30MFP
B1	IPS e.max CAD (LD)	Optrafine Ceramic Polishing System	Manual finishing and polishing with contrangled handpiece Firs Tip: 30 seconds, 10.000 rpm Second Tip: 30 seconds, 6.000 rpm	
A2	VITA Suprinity (ZLS)	SUPRINITY Polishing Set Clinical	Manual finishing and polishing with contrangled handpiece Firs Tip: 60 seconds, 10.000 rpm Second Tip: 60 seconds, 6.000 rpm	60MFP
B2	IPS e.max CAD (LD)	Optrafine Ceramic Polishing System	Manual finishing and polishing with contrangled handpiece Firs Tip: 60 seconds, 10.000 rpm Second Tip: 60 seconds, 6.000 rpm	

A3	VITA Suprinity (ZLS)	VITA AKZENT Plus PASTE	Laboratory finishing Gently applied on the surface with a brush and fired	GP
B3	IPS e.max CAD (LD)	IPS e.max CAD Crystall./Glaze paste	Laboratory finishing Gently applied on the surface with a brush and fired	
A4	VITA Suprinity (ZLS)	VITA AKZENT Plus SPRAY	Laboratory finishing Gently sprayed on the surface and fired	GS
B4	IPS e.max CAD (LD)	IPS e.max CAD Crystall./Glaze spray	Laboratory finishing Gently sprayed on the surface and fired	

For each group, 5 wedges were used for roughness and gloss measurements. As both sides of wedges underwent the test, a total of 10 surfaces for group were treated (n=10). One extra specimen for each subgroup was prepared for the SEM observation.

For the manual finishing procedure (subgroups A.1, A.2, B.1, B.2) rubber cups were used and replaced every two specimens. Finishing was carried out following the manufacturers' instructions with an angled hand-piece (Kavo INTRAmatic 20CN, Kavo, Biberach, Germany) under water-cooling. All the manual finishing and polishing procedures were performed by the same operator. Before and during the procedure, the operator was calibrated using a precision scale, taking into account a

reference force of 40 g for the light pressure replication. The operator calibration was repeated for each subgroup (Antonson et al., 2011).

For the furnace-based finishing procedures (subgroups A.3, A.4, B.3, and B.4), the glazing material was applied and fired (VITA Vacumat 6000) following the manufacturers' instructions.

Roughness (Ra) and Gloss (Gu) measurement

Before testing, specimens were ultrasonically cleaned in 95% alcohol solution for 3 minutes (CP104, CEIA, Italy).

A profilometer (Mitutoyo SJ-201P, Mitutoyo Corp., Kanagawa, Japan) set with a cutoff value of 0.8 mm, a stylus speed of 0.5 mm/s and a tracking length of 5.0 mm (The European Standard, 2004) was used to assess the surface roughness. The instrument reading track was standardized by means of a custom silicon mold that hold the specimen. Mean Ra (μm) was recorded.

A glossmeter (Novo-Curve, Rhopoint Instruments Ltd., Bexhill-on-Sea, U) with a 60° angle was used for gloss evaluation following ISO 2813 specifications for ceramic materials (ISO 2813, 1999). Gloss Units (GU) were recorded. A custom-made opaque silicone mold was used in order to avoid any ambient light and control the position of the specimens during measuring.

Ra and GU values were recorded three times for each specimen and the means were calculated.

Since pooled data for all the tested groups passed the normality test (P = 0.077) and the equal variance test (P = 0.545), the results were analyzed applying two separate 2-Way ANOVA for gloss and roughness measurement respectively, followed by Tukey's t-tests ($\alpha \leq 0.05$) to determine the level of significance between groups.

SEM evaluation

VITA Suprinity and IPS E.max CAD were processed for qualitative evaluation according to the aforementioned finishing and polishing/glazing procedures adopted. For each group, 1 extra specimen was prepared. After crystallization, specimens were ultrasonically cleaned in 95% alcohol solution for 3 minutes and air-dried with an oil-free stream. Specimens were then secured to SEM (JSM-6060LV, JEOL, Tokyo, Japan) slab with gold conducting tape, and gold coated in a vacuum sputter coater (SC7620 Sputter Coater, Polaron Range, Quorum Technologies, Newhaven, UK). The surfaces were observed at x500 magnification.

Results

Roughness (Ra)

The surface roughness of VITA Suprinity and IPS e.max CAD measured after 30 seconds and 60 seconds polishing, glazing paste and glazing spray is reported in Table 2.

Table 2. Means (SD) for surface roughness of VITA Suprinity and IPS e.max CAD after 30 seconds and 60 seconds polishing, glazing paste and glazing spray and statistical significance (Sign.). Different letters indicate statistically different groups.

Treatment	**Roughness (µm)**						**Sign.**
	VITA Suprinity			**IPS e.max CAD**			
	Mean	**SD**	**Sign.**	**Mean**	**SD**	**Sign.**	
30 s Polishing	0.69	0.15	b	0.62	0.21	a	BC
	a			a			
60 s Polishing	0.37	0.08	a	0.53	0.13	a	A
	a			b			
Glazing paste	0.42	0.12	a	0.66	0.15	a	AB
	a			b			
Glazing spray	0.64	0.31	b	0.91	0.21	b	C
	a			b			
Sign.	A			B			

Statistical significant differences were detected for both the tested variables. VITA Suprinity (ZLS) showed a significant lower roughness compared to IPS e.max CAD (LD). Materials resulted statistically different for all the tested treatments except that for the 30 seconds polishing group.

The 60 seconds polishing and the glazing paste groups obtained the highest level of significance. No statistically significant differences were found between the glazing paste and the 30 seconds polishing group. Furthermore lowest level of significance was reported for the glazing spray and the 30 seconds polishing groups.

Statistically significant differences were also found for treatment within the same materials.

VITA Suprinity obtained the lowest roughness in the 60 seconds polishing group (0.37 ± 0.08 μm) and glazing paste group (0.42 ± 0.12 μm). These groups statistically significant differ from the 30 seconds polishing (0.69 ± 0.15 μm) and the glazing spray (0.64 ± 0.31 μm) groups.

For IPS e.max CAD, the glazing spray group resulted in the roughest surface (0.91 ± 0.21 μm) and this was reported to be statistically significant compared to 30 seconds (0.62 ± 0.21 μm) and 60 seconds (0.53 ± 0.13 μm) polishing, and glazing paste (0.66 ± 0.15 μm) groups.

Gloss (GU)

The gloss of VITA Suprinity and IPS e.max CAD measured after 30 seconds and 60 seconds polishing, glazing paste and glazing spray is presented in Table 3.

Table 3. Means (SD) for surface gloss of VITA Suprinity and IPS e.max CAD after 30 seconds and 60 seconds polishing, glazing paste and glazing spray and statistical significance (Sign.). Different letters indicate statistically different groups.

Treatment	**Gloss (GU)**						**Sign.**
	VITA Suprinity			**IPS e.max CAD**			
	Mean	**SD**	**Sign.**	**Mean**	**SD**	**Sign.**	
30 s Polishing	49.05	6.17	c	63.14	12.13	a	B
	b			a			
60 s Polishing	85.02	12.94	a	65.77	12.36	a	A
	a			b			
Glazing paste	72.24	10.60	ab	48.28	9.53	b	B
	a			b			
Glazing spray	69.86	9.40	b	54.89	13.91	a	B
	a			b			
Sign.	A			B			

As described before for roughness both the variables shown statistically significant differences. VITA Suprinity (ZLS) reported a statistically significant higher gloss compared to IPS e.max CAD (LD). All the treatments reported a significant higher Gloss for ZLS. The only exception was 30 seconds polishing group where the gloss reported for LD was significantly higher compared to ZLS.

The 60 seconds polishing group showed the higher level of significance compared to the other treatments that did not statistically differ one from the other in terms of significance.

For VITA Suprinity, the 60 seconds polishing (85 ± 13 GU) and the glazing paste (72 ± 11 GU) groups showed the highest gloss. No statistically significant differences were found between the glazing paste and the glazing spray (70 ± 9 GU) groups. The 30 seconds polishing group (49 ± 6 GU) obtain the lowest gloss and this difference was statistically significant.

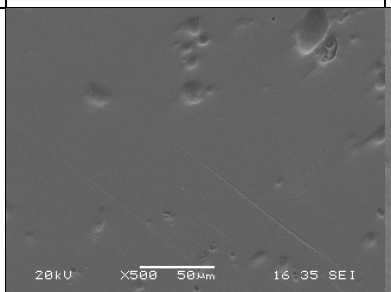
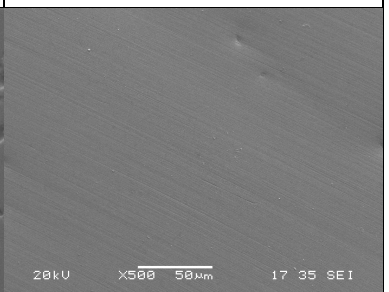
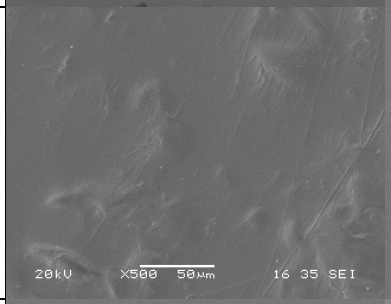
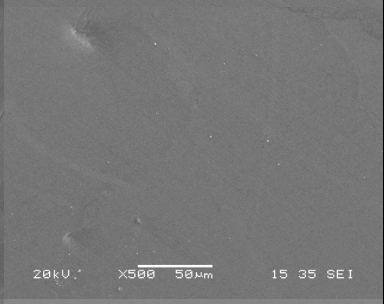
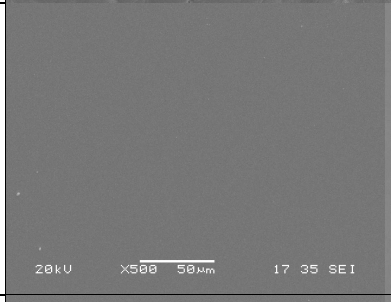
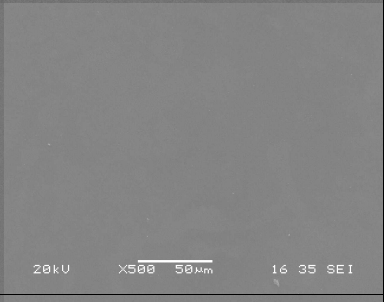
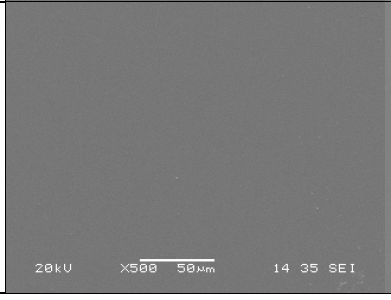
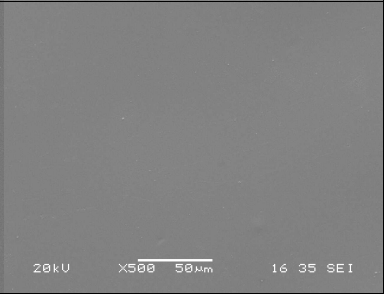
For IPS e.max CAD, the glazing paste group (48 ± 10 GU) resulted in the lowest gloss surface compared to the 30 (63 ± 12 GU) and the 60 seconds (66 ± 12 GU) polishing, and glazing spray (55 ± 14 GU) groups and this difference was reported to be statistically significant.

SEM evaluation

Different superficial topographies were observed for the manual and heat-mediated polishing systems (Figure 3). For VITA Suprinity, some irregularities were still present after 30 seconds manual finishing and polishing. The 60 seconds polishing group resulted in a more homogeneous surface with local small defect and scratches. For IPS e.max CAD, scratches resulting from the abrasive action of the polishing rubber cups, were particularly evident after 30 seconds manual polishing, without any substantial superficial defect. Instead, a more uniform surface was found for the 60 seconds manual polishing group.

For both materials, free-of-defect and smooth surfaces were observed after the application of the two different heat-mediated additive glazing systems.

Figure 3. SEM analysis of VITA Suprinity and IPS e.max CAD at 500x after 30 (30MFP) and 60 (60MFP) seconds manual finishing and polishing, glazing paste (GP) and glazing spray (GS).

Treatment	VITA Suprinity	IPS e.max CAD
30MFP		
60MFP		
GP		
GS		

Discussion

As statistical significant differences were detected between IPS e.max CAD and VITA Suprinity in the ability of decreasing roughness and increasing gloss, the first null hypothesis has been rejected. Statistical significant differences were also found in roughness and gloss among the different finishing and polishing systems tested, for both IPS e.max CAD and VITA Suprinity. Thus, the second null hypothesis was rejected as well.

Roughness and gloss allow glass ceramics to be superficially analyzed and screened with regard to their surface characteristics after finishing (Heintze et al., 2006). Roughness can be described by several linear (Ra, Rq, Rz) or three-dimensional (Sa,Sq,Sz) parameters (Fasbinder & Neiva, 2016; Odatsu et al., 2013; Zinelis et al., 2010). For the present investigation, Ra, which is defined as the mean arithmetical value of all the absolute distances of the profile inside of the measuring length (Silva et al., 2014), was assessed since it is the most common combination for evaluating the effect of finishing protocols on dental ceramics (Flury et al., 2010; Sasahara et al., 2006; Wang et al., 2009; Whitehead et al., 1995; Yilmaz & Ozkan, 2010).

Gloss (GU) represents the amount of specular reflection from a surface (Lawson & Burgess, 2015; Vichi et al., 2011) calculated by comparing

the magnitude of incident light travelling toward a surface at a 60º angle to the magnitude travelling away from the surface at an equal and opposite angle. Gloss is determined by both the optical properties (Refraction Index) and the surface topography (Lawson & Burgess, 2015) in such an extent that the coarser the texture is, the lower the reflectivity (Covey et al., 2011; Heintze et al., 2006; Kakaboura et al., 2007; O'Brien et al., 1984; Ohara et al., 2009).

The first aim of the present study was to compare the combination of silica-based glass ceramic and finishing set as proposed by the manufacturer. As the proposed systems are specific for each of the two material, the differences observed in roughness and gloss between VITA Suprinity and IPS e.max CAD might be due to the differences of either the microstructure of the two materials, or the properties of the polishing and glazing systems used. At present, no data are available in literature for VITA Suprinity. The data available in literature for IPS e.max CAD indicate similar performances of the various manual finishing and polishing systems tested, suggesting that the ability of obtaining smooth surfaces is more material-related than based on the finishing and polishing system selected (Lawson et al., 2014; Tholt de Vasconcellos et al., 2006).

Likewise, similar outcomes were recorded by comparing polishing and glazing. Indeed, IPS e.max CAD surface polished with OptraFine for 30 seconds and 60 seconds, and finished using glazing paste showed comparable roughness. Although Lawson et al. (Lawson et al., 2014) reported less efficacy of glazing paste than 60 seconds manual polishing, most of the studies agree on the matching efficacy of the two procedures (Akar et al., 2014; Amer et al., 2015; Preis et al., 2013), even if dissimilar values are reported in the cited studies. This dissimilarity might be explained with the different baseline roughness of the specimens. Since the after-milling roughness is usually replicated by grinding and polishing the specimens with silicone carbide papers (Fraga et al., 2015; Kawai et al., 2000) and the grit set differs among the studies, the baseline roughness varies and might not effectively match with that of the milled surface. For the present study, the flat specimens were directly milled with the CEREC MC XL milling unit, therefore the high reproducibility and the measurement of the real after-milling surface were guaranteed.

Despite the minor differences in composition between VITA Suprinity Polishing set and Optrafine manual abrasives (Table 4), polishing time has played an important role in the final smoothness and luster (Heintze et al., 2006).

Table 4. Polishing systems specifications as declared by the manufacturer.

Instrument	Grit	Contents	Manufacturer
VITA Suprinity Polishing Set clinical (Pink)	Diamond powder 500/600	Polyurethane-rubber/caoutchouc Diamond grains Pigments	VITA Zahnfabrik Bad Sackingen Germany
OptraFine F (Coarse)	NR	Synthetic rubber Diamond granulate Titanium dioxide	Ivoclar Vivadent AG Schaan Liechtenstein
VITA Suprinity Polishing Set clinical (Grey)	Diamond powder 3000	Polyurethane-rubber/caoutchouc Diamond grains Pigments	VITA Zahnfabrik Bad Sackingen Germany
OptraFine P (Fine)	NR	Synthetic rubber Diamond granulate Titanium dioxide	Ivoclar Vivadent AG Schaan Liechtenstein
NR = not reported by the manufacturer			

In the present study, polishing time affected the roughness and gloss of VITA Suprinity more than those of IPS e.max CAD. Whilst VITA Suprinity became smoother and glossier after 60 seconds polishing, IPS e.max CAD did not change. This might be explained by differences in the microstructure of the two materials (Kelly et al., 1996; Sasahara et al., 2006; Silva et al., 2014). VITA Suprinity is a zirconia-reinforced lithium silicate glass ceramic with a mean crystals size of approximately 0.5 μm, whilst IPS e.max CAD is a lithium disilicate glass ceramic with a mean crystals size of 1.5 μm. Since ceramic crystals removed from the surface during polishing might become part of the abrasive system and contribute to characterize the surface topography (Al-Wahadni & Martin,

1999), the finer microstructure of VITA Suprinity might explain its capability to be better smoothed after 60 seconds polishing than IPS e.max CAD (Yilmaz & Ozkan, 2010). In addition, SiO_2 concentration varies between IPS e.max CAD (57.0-80.0) and VITA Suprinity (56.0-64.0). As the more the concentration of SiO_2 is, the greater the crystalline phase (Goharian et al., 2010), the higher crystalline content of IPS e.max CAD might also explain its worse capability to be smoothed after 60 seconds polishing. Moreover, the higher content of ZrO_2 in VITA Suprinity (8.0-12.0) might contribute to justify its lower superficial roughness after 60 seconds polishing, as zirconia allows the material to be more efficaciously polished (Kou et al., 2006).

The timings tested in the present study refer to manufacturer instructions and are in the range of clinical common use. It is to be supposed that both materials, especially VITA Suprinity, might display higher superficial smoothness and gloss at longer polishing times (Heintze et al., 2006). However, longer polishing times might cause higher substance loss, as polishing is a subtractive procedure, and this has to be taken into account from a clinical viewpoint.

Comparing the efficacy of the furnace-based glazing systems, VITA Suprinity showed lower roughness and higher gloss after paste glazing rather than spray glazing. Different trends were observed for IPS e.max

CAD as roughness and gloss were lower for paste glazing rather than for spray. The different composition and characteristic density of IPS e.max CAD Crystall./Glaze Paste compared to VITA AKZENT Plus Paste resulted in a reduced glaze spread ability on the ceramic surface. These differences might explain the lower gloss of IPS e.max CAD rather than VITA Suprinity, as the less smooth glaze coat might have caused a variation in the superficial refraction index and, therefore, in the gloss.

As reported by Vo et al. (Vo et al., 2015), IPS e.max CAD treated with glazing spray obtained the worst superficial roughness among the tested finishing systems. Owing to the rougher baseline surfaces of the milled wedges, glazing spray was not able to uniformly coat all the irregularities, which would justify its worst smoothening efficacy (Addison et al., 2012; Asai et al., 2010).

By comparing the furnace-based systems efficacy on the two tested materials, it was observed that both glazing paste and spray were more effective on VITA Suprinity rather than on IPS e.max CAD in term of smoothness and luster. As previously reported about roughness, a possible reason of this finding might be due to the different microstructure of these glass ceramics. Because of the lower crystalline volume and the smaller crystals size, VITA Suprinity might have exhibited a lower baseline roughness, which might have led to lower

roughness after glazing (Al-Shammery et al., 2007; Dalkiz et al., 2009; Fasbinder & Neiva, 2016; Sasahara et al., 2006). As roughness and gloss have an inverse proportional trend (Lawson & Burgess, 2015), the lower roughness of VITA Suprinity specimens' surfaces may explain their higher luster.

To better understand the outcome of the present study the data collected have to be correlated with clinical needs. Some in vivo studies (Bollen et al., 1996) suggest an ideal threshold surface roughness of 0.2 μm above which the bacterial retention is facilitated and the incidence of biological complications increases. In addition, superficial roughness greater than 0.5 μm can be detected by the sensorial fibers of the tongue, resulting in a discomfort for the patient (Jones et al., 2004). Nevertheless, natural enamel roughness is reported to range between 0.64 μm and 0.90 μm with regard to the tooth type, location and patient age (Preis et al., 2013; Willems et al., 1991). By evaluating the clinical acceptability of the finished surfaces, all the Ra values were far away from the abrasive wearing threshold (1.5 μm) (Lawson et al., 2014). Furthermore, 60 seconds polishing and glazing paste allowed VITA Suprinity to be imperceptible by the tongue, whilst all the other groups fall into the enamel roughness range, with the exception of IPS e.max CAD after glazing spray.

Conversely to roughness, a clinically accepted threshold for gloss has not been established yet. Natural enamel gloss is reported to range between 40 and 52 GU (Barucci-Pfister & Göhring, 2009; Mörmann et al., 2013). VITA Surpinity polished for 30 seconds and IPS e.max CAD finished with glazing paste displayed similar gloss than that of enamel, while all the other procedures gave higher values for both materials.

Conclusion

With the limitations of the present study, the following conclusions can be drawn:

- VITA Suprinity displayed higher polishability rather than IPS e.max CAD.

- Manual finishing and polishing for 60 seconds and furnace-based glazing with paste are the most effective procedures in lowering the roughness of CAD-CAM lithia silica-based glass ceramics.

- Manual finishing and polishing for 60 seconds is the most effective procedures in improving the lustrous of CAD-CAM lithia silica-based glass ceramics, which confirms the inversely proportional relationship between roughness and gloss.

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Chapter 4: Adhesive properties

4.1 Acid concentration and etching time as variables in bonding to lithia silica-based glass ceramics.

Introduction

By modifying glass and crystalline content, dental ceramics can be produced with different esthetic and mechanical properties (Kern, 2015). When silicates concentration is above 15%, ceramics can be classified as "glass ceramic" (Kern, 2015). Within this category, feldspathic, leucite-reinforced, lithium disilicate and zirconia reinforced lithium silicates are available for indirect restorations (Hu et al., 2016; ISO 6872:2015; Kern, 2015; Sedda et al., 2014; Vichi et al., 2013). Due to the high crystalline content, lithia silica-based ceramics show mechanical properties that make them suitable for single unit restorations (Magne et al., 2015; Pjetursson et al., 2007; Stawarczyk et al., 2015).

These materials can be processed either by heat-pressing or CAD/CAM technology. By using CAD-CAM, monolithic crowns can be fabricated with accelerated work-flow and reduced costs (Fasbinder, 2006; Mörmann, 2006; Patzelt et al., 2014; Sadowsky, 2006). Once crystallized and finished, lithia silica-based ceramics can be cemented with either non-adhesive or adhesive procedures (Gehrt et al., 2013; Heintze et al.,

2008; ISO 6872:2015; Kern et al., 2012; Ozcan & Vallittu, 2003). To overcome the low crown-abutment retention (da Silveira et al., 2005), resin cements are successfully used to adhere to tooth structure and restorative materials (Albert & El-Mowafy, 2004; Cavalcanti et al., 2009; Diaz-Arnold et al., 1992; Gehrt et al., 2013; Heintze et al., 2008; Kern et al., 2012; Koutayas et al., 2009; Mörmann, 2006).

Adhesion to the substrates can be performed with different systems. Practically, cements can be either mediated by adhesives or not in the case of self-adhesive agents. For the latter, bonding to dentin can be ensured without any superficial pre-treatment (Holderegger et al., 2008; Vrochari et al., 2009). For bonding to the glass ceramic, the intaglio surface has to be mechanically and chemically modified (Borges et al., 2003; Della Bona et al., 2004; Erdem et al., 2014; Tian et al., 2014). To promote micro-retention, several systems have been suggested, such as sandblasting, Er:YAG lasering, acid etching and silica coating (Della Bona et al., 2004; Filho et al., 2004; Gökçe et al., 2007; Kato et al., 2000; Matinlinna et al., 2004; Moharamzadeh et al., 2008; Parker, 2004). Among the conditioners, hydrofluoric acid is widely recognized as an efficient agent (Addison et al., 2007; Della Bona et al., 2004; Filho et al., 2004; Güler et al., 2006; Hooshmand et al., 2008; Klosa et al., 2009; Salvio et al., 2007; Spohr et al., 2003). Because of the glass phase, lithia

silica-based ceramics are hydrofluoric acid-sensitive (Valandro et al., 2005). By etching, the glass matrix dissolves and the surface becomes rough, thus the luting agent can penetrate and mechanically interlock to form a complex with the crystals (Gökçe et al., 2007; Roulet et al., 1995). Furthermore, to maximize the affinity to polymers (Bailey, 1989; Horn, 1983; Jardel et al., 1999; Nakabayashi, 1997), a silane solution can be spread on the etched surface, improving the physico-chemical interaction between resins and glass ceramics (Addison et al., 2007; Güler et al., 2006; Hooshmand et al., 2008; Klosa et al., 2009; Myerson, 1969; Newburg & Pameijer, 1978; Paffenbarger et al., 1967; Roulet et al., 1995; Semmelman & Kulp, 1968).

The optimal bonding protocol to lithia silica-based glass ceramics is still controversial, and several combination of hydrofluoric acid etching times and concentrations have been proposed (Addison et al., 2007; Barghi et al., 2006; Chaiyabutr et al., 2008; Chen et al., 1998a; Chen et al., 1998b; Della Bona et al., 2002; Güler et al., 2006; Naves et al., 2010; Tian et al., 2014). Since the size and number of irregularities created on the intaglio surface as a result of etching were seen to be associated with the acid formulation, dilution (Canay et al., 2001; Kukiattrakoon & Thammasitboon, 2007; Sundfeld Neto et al., 2015) and application time (Addison et al., 2007; Barghi et al., 2006; Chen et al., 1998; Della Bona

& Anusavice, 2002; Guler et al., 2006; Naves et al., 2010), it was considered of interest to evaluate whether different conditioning protocols might affect the bond strength of lithia silica-based glass ceramics.

On the basis of these assumptions, the objective of the present study was to test the effect of different hydrofluoric acid etching times and concentrations on the bond strength of lithia silica-based glass ceramics to a dual-curing self-adhesive resin cement.

Three null hypotesis were formulated: i) hydrofluoric acid etching time does not influence the bond strength of VITA Suprinity and IPS e.max CAD; ii) hydrofluoric acid concentration does not influence the bond strength of VITA Suprinity and IPS e.max CAD; iii) no differences can be identified between VITA Suprinity and IPS e.max CAD according to the hydrofluoric acid etching protocol.

Methods and Materials

Microshear test

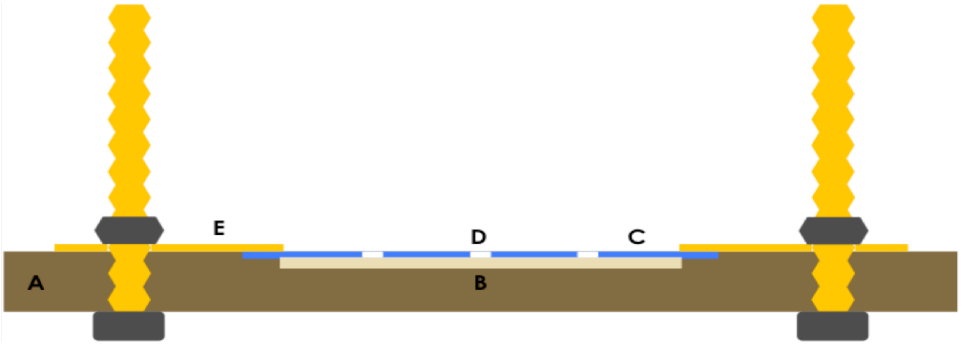
CEREC® CAD-CAM (Sirona Dental, Bernsheim, Germany) pre-crystallized blocks of zirconia-reinforced lithium silicate (ZLS) (VITA Suprinity, T A3, VITA Zahnfabrik, Bad Sackingen, Germany) and lithium disilicate (LD) (IPS e.max CAD, LT A3, Ivoclar Vivadent AG, Schaan, Liechtenstein) were selected for this study. Twelve different blocks were used, respectively, for each of the two tested materials.

From each block, 4 bars with dimension of 4.0 ± 0.2 mm in width, 1.2 ± 0.2 mm in thickness, and 15 ± 0.2 mm in length (ISO 6872:2015) were milled using a CEREC inLab MC-XL milling machine (Sirona Dental, Bernsheim, Germany). Milling burs were replaced before start. Milled bars were separated from the block's base by means of a low-speed-water-cooled diamond disc. Bars were then placed in the proprietary firing tray and isolated with firing paste (VITA firing paste, VITA Zahnfabrik, Bad Sackingen, Germany for VITA Suprinity; IPS Object Fix Putty, Ivoclar Vivadent, Schaan, Liechtenstein for e.max CAD). Final crystallization was performed following the manufacturer's instructions (VITA Vacumat 6000, VITA Zahnfabrik, Bad Sackingen, Germany).

After crystallization, bars were randomly divided into 18 groups according to hydrofluoric acid (HF) etching treatment (n=5). For VITA Suprinity, concentration of 4.9% (VITA Ceramics Etch, VITA Zahnfabrik, Bad Sackingen, Germany) and 9.5% (Ultradent Porcelain Etch Gel, Ultradent Products Inc., South Jordan, Utah) were used both at 0 (no etching), 20, 40, 60, 120 seconds (Xiaoping et al., 2014). For IPS e.max CAD, the same procedure was used but the 4.9% etching was performed with IPS Ceramic Etching Gel (Ivoclar Vivadent AG, Schaan, Liechtenstein). Fifteen specimens (*n*=15) were obtained for each group.

A dedicated device was created for the test. Three holes for the calibrated specimens preparation with a mean diameter of 0.8 mm, were equidistantly drilled in a blue plastic-wax sheet. An aluminum base was machined at the center of the upper surface, creating an empty space for maintaining in contact the underlying bar and the plastic-wax mold. An extra piece was centrally milled along all the thickness, letting a window in correspondence to the plastic mold, and fixed to the aluminum base by means of two bolts for the stabilisation of the plastic mold (Figure 1).

Figure 1. Schematic overview of the device dedicated to the specimen preparation. (A) Alluminium base; (B) Bar; (C) Blue plastic wax mold; (D) Specimen space (⌀=0.8 mm; h=0.5 mm); (E) Screwed alluminium extra-piece



Prior to starting, each group was vibrated in demineralized water for 3 minutes and oil-free-air dried (CP104, CEIA, Italy). Once the bar was in place into the mold, the hydrofluoric acid application was carried out by means of a syringe. Brushes were employed to scrub gently the acid through the holes (Bisco brush applicators, Bisco Inc., Schaumburg, IL, U.S.A.). For the removal, the hole was rinsed out with running water and spurted water for 15 seconds, respectively.

Each hole was completely dried with oil-free air stream. For each group, an ethanol-based silane coupling solution (Ultradent Silane, Ultradent Products Inc., South Jordan, Utah) was applied with a syringe, scrubbed with a brush and allowed to react for 60 seconds. Each hole was

abundantly dried with oil-free air stream until the solvent was completely evaporated.

A mixing tip was used to mix the base and catalyst of self-adhesive, auto-mixed, dual-cure resin cement (RelyX Uni-cem 2, 3M ESPE, Minnesota, U.S.A.). The thin tip was held close to the hole and the resin cement was injected into.

All the excesses were removed from the mold surface and then the resin cement was cured for 40 seconds using an halogen light-curing unit (Astralis 7, Ivoclar Vivadent AG, Schaan, Liechtenstein) with an output of 400-750 mW/cm^2. After 24 hours of storage in distilled water at 37º C, bar and plastic-wax mold were carefully separated.

Each specimen was examined using magnifying loupes (4.5 X) to identify any possible defects in the resin cement cylinder, such as bubbles and/or flow of resin cement beyond the limits of the bonding area. Defective or flawed specimens were discarded and re-prepared.

Afterwards, specimens were secured in a vise and attached to a shear-testing jig. A thin wire (0.2-mm diameter) was looped around the base of each resin cement cylinder, in contact with half of its circumference, keeping the setup aligned to ensure the correct orientation of the shear forces. The cement/ceramic interface was then tested under shear mode in a universal testing machine (Triax 50, Controls, Milano, Italy) at a

crosshead speed of 1 mm/min until failure. The μSBS was calculated in MPa by dividing the load at failure by the surface area (mm^2) of each specimen.

The results were statistically analyzed with three-way analysis of variance (ANOVA) and Tukey test for post hoc (p=0.05). The failure mode of the debonded specimens was determined using 120X stereomicroscope magnification and was classified as adhesive (A), mixed (M), cohesive in resin cement (CR), or ceramic (CC) (Lise et al., 2015).

SEM observation

For each tested group, an extra specimen was prepared for SEM observation (JSM-6060LV, JEOL, Tokyo, Japan). Prior to conditioning with HF at 4.9% and 9.5% following the application time of 0 (no etching), 20, 40, 60, 120 seconds, specimens were vibrated in demineralized water for 3 minutes and oil-free-air. For the acid removal, specimens were rinsed out with running and spurted water for 15 seconds, respectively, and vibrated in a 95% alcohol solution for 3 minutes.

Specimens were secured to SEM tabs with gold conducting tape, and gold coated in a vacuum sputter coater (SC7620 Sputter Coater, Polaron

Range, Quorum Technologies, Newhaven, UK). Surfaces were observed at x5000 magnification.

Results

The mean and standard deviations values of the μSBS are reported in Table 1.

According to the Tukey test for multi-comparison, significant differences were found between unetched and etched surfaces for both materials. Indeed, VITA Surpinity and IPS e.max CAD showed statistically higher bond strength after etching procedures compared with control groups. No significant differences were found with regard to the conditioning time. Significant higher bond strength values were recoreded for VITA Suprinity than IPS e.max CAD at 4.9% etching concentration, while differences were not statistically significant when 9.5% HF etch was applied.

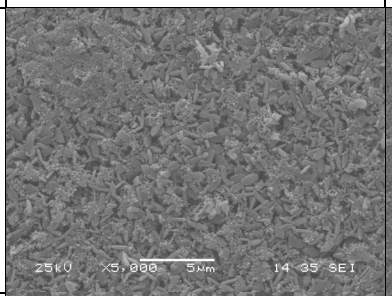
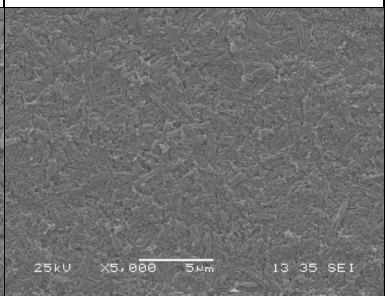
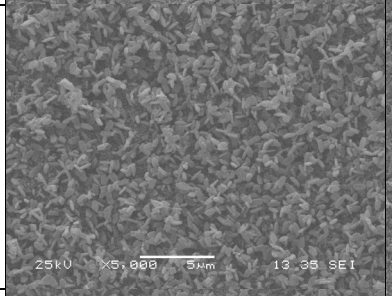
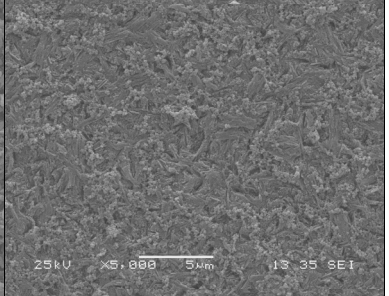
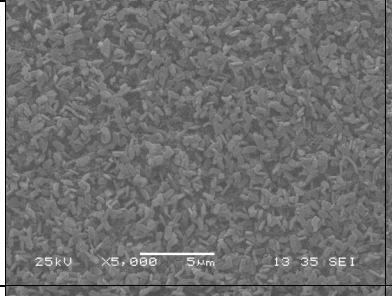
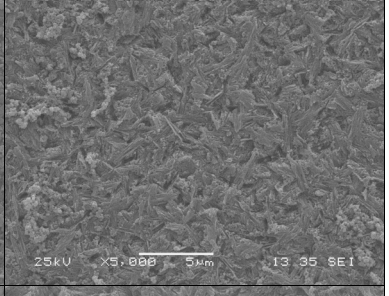
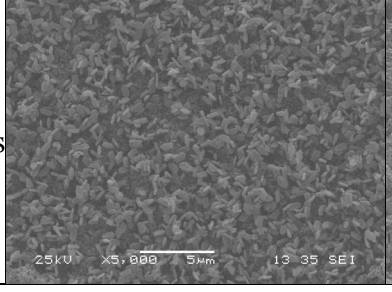
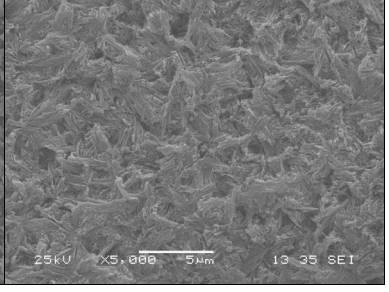
Table 1. μSBS of VITA Suprinity and IPS e.max CAD to RelyX Unicem 2. Different letters label statistically significant differences in bond strength with regard to the application time and concentration of hydrofluoric acid.

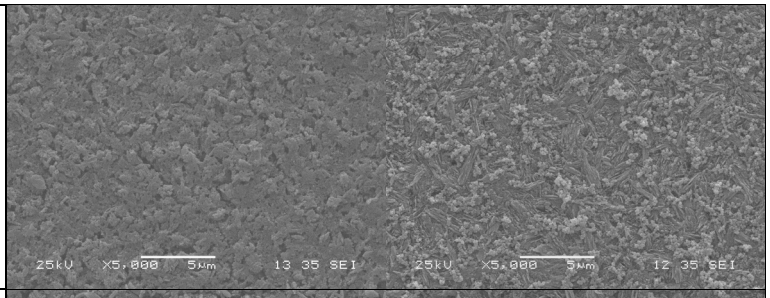
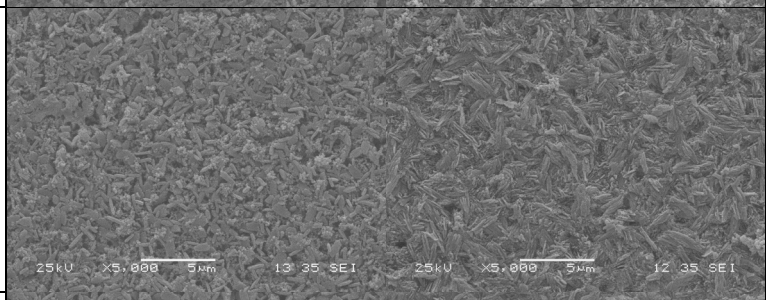
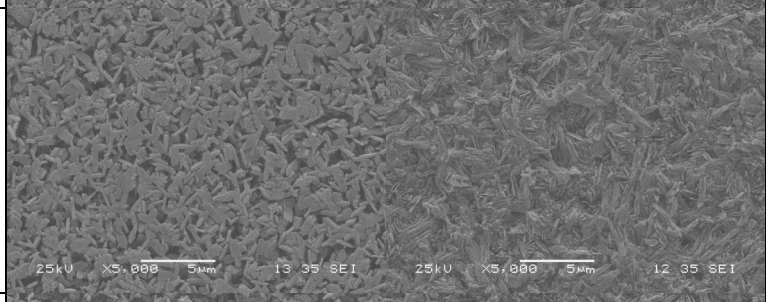
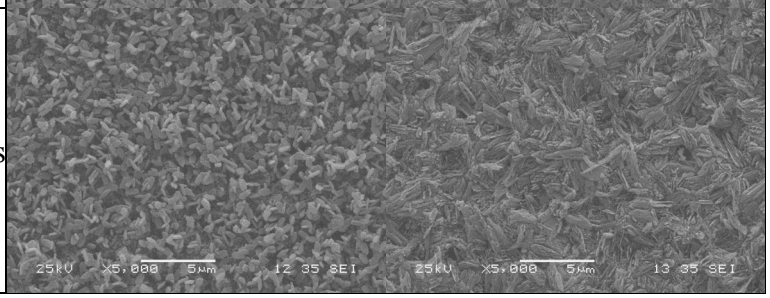
Time	4.9%^{A}				9.5%^{A}			
	VITA Suprinity^{A,a}		IPS e.max CAD^{B,b}		VITA Suprinity^{A,a}		IPS e.max CAD^{B,a}	
0^{B}	6.15	4.78	7.98	4.79	6.15	4.78	7.98	4.79
20^{A}	21.07	4.34	14.29	7.13	16.45	7.71	11.54	5.30
40^{A}	20.85	3.76	16.84	7.73	13.77	3.42	15.26	5.26
60^{A}	19.68	6.32	15.85	5.52	20.60	6.41	18.19	4.63
120^{A}	20.10	6.50	14.53	4.59	14.18	7.38	17.34	4.82
HF%	**VITA Suprinity^{a}**		**IPS e.max CAD^{b}**		**VITA Suprinity^{a}**		**IPS e.max CAD^{a}**	

SEM observation

The dissolution of the glassy phase together with the exposure of the crystalline matrix, were proportional to the hydrofluoric acid application times for both the tested glass ceramics (Figure 2). VITA Suprinity crystals became completely visible after HF etching at 4.9% and 9.5% for 40 and 120 seconds, respectively. On the contrary, IPS e.max CAD yielded the greater crystalline exposure by HF conditioning at 4.9% and 9.5% for 120 and 40 seconds, respectively.

Figure 2. Effect of 4.9% and 9.5% hydrofluoric acid etching for 20, 40, 60 and 120 seconds on VITA Suprinity and IPS e.max CAD. SEM analysis at 5000x.

Treatment		VITA Suprinity	IPS e.max CAD
HF 4.9%	20s		
	40s		
	60s		
	120s		

HF 9.5%	20s	
	40s	
	60s	
	120s	

Discussion

The purpose of the present study was to test the effect of different etching protocols on the adhesion to VITA Suprinity (ZLS) and IPS e.max CAD (LD). Higher μSBS values were observed for both materials after etching with hydrofluoric acid. However, etching time did not influence the bond strength to ZLS and LD, thus the first null hypothesis was accepted. By varying the concentration of the etchant, different behavior was observed for ZLS and LD. In fact, ZLS yielded higher μSBS values after etching with HF at 4.9% instead of at 9.5%. This difference was not detectable for LD, therefore the second null hypothesis was rejected for ZLS and accepted for LD. Furthermore, the bond strengths of the two tested ceramics differed on equal etching protocol. Indeed, ZLS showed better bonding ability than LD, thus the third null hypothesis was rejected.

The bond strengths of VITA Suprinity and IPS e.max CAD were seen to be independent from the etching time. By HF conditioning, the glassy and second crystalline phases dissolve and crystals network exposes (Borges et al., 2003; Chen et al., 1998a; Chen et al., 1998b; Phoenix & Shen, 1995; Thurmond et al., 1994). The contact area increases (Della Bona et al., 2004), as well as the roughness and the wettability of the ceramic surface (Della Bona et al., 2004; Ozcan & Vallittu, 2003; Salvio

et al., 2007; Spohr et al., 2003). Hence, resin cement can penetrate into the crystalline microstructure and, once polymerized, can be mechanically retained (Roulet et al., 1995; Sundfeld Neto et al., 2015).

Despite the retention into the etched surface of the resin cement is considered to be more important than the chemical bonds to the inorganic phase of glass ceramic (Hu et al., 2016; Yavuz et al., 2015), a methacrylate propyl trimethoxysilyl silane was used after conditioning since hydrofluoric acid etching in combination with silane primer is considered as the gold standard procedure for bonding to lithia silica-based ceramics (Aboushelib & Sleem, 2014; Della Bona et al., 2000; Della Bona et al., 2003; Della Bona et al., 2004; Filho et al., 2004; Jardel et al., 1999; Kalavacharla et al., 2015; Lise et al., 2015; Ozcan & Vallittu, 2003; Panah et al., 2008; Pisani-Proenca et al., 2006; Stewart et al., 2002). After silanization, the superficial energy improves, as well as the chemical interaction between the inorganic and organic matrix of the glass ceramic and the resin, respectively (Della Bona et al., 2004; Matsumura et al., 1987; Nakabayashi, 1997; Phoeniz & Schen, 1995; Söderholm & Shang, 1993).

Unlike most of the studies have measured the efficacy of etching and silane protocols on smoothed lithium disilicate surfaces, VITA Suprinity and IPS e.max CAD underwent to conditioning just after milling. The

milled surface might have limited the etching gel penetration and its spread into the crannies (Hay et al., 2008), thus a more outward etching pattern might have been obtained, which would explain the lower bond strength values obtained for the present test compared to those available in the literature (Brum et al., 2011). In fact, the standardization of the surfaces with silicon carbide paper (SiC) and diamond pastes prior to conditioning (Aboushelib et al., 2005; Aboushelib et al., 2006; Ayad et al., 2008; Clelland et al., 2007; Della Bona et al., 2000; Della Bona et al., 2003; Nagai et al., 2005; Nagayassu et al., 2006; Panah et al., 2008; Pekkan & Hekimoglu, 2009; Sato et al., 1999) removes any preexisting mechanical retention. This really allows to measure the effect of the tested etching protocols on the bond strength to lithia silica-based glass ceramics, but it does not faithfully replicate the in vivo situation where the hydrofluoric acid is applied on the coarse surface (Brum et al., 2011).

ZLS and LD showed different bonding ability after 4.9% and 9.5% HF etching. Unlike LD, ZLS yielded higher μSBS values after conditioning with hydrofluoric acid at 4.9% rather than 9.5%. The effectiveness of HF concentration on bonding to lithia silica-based ceramics is controversial. Depending on the test design, glass ceramics exhibited different bonding ability and the proper hydrofluoric acid concentration still remains unclear. In that respect, HF at 4.9% (Caparroso et al., 2014), 7.5%, 10%

or 15% (Sundfeld Neto et al., 2015) was seen to be equivalently efficient for the IPS e.max Press conditioning. Since the degree of glass dissolution might be considered proportional to the HF concentration (Roulet et al., 1995), it is reasonable to expect higher bond strength values at higher etchant acidity (Sundfeld Neto et al., 2015). However, the dissolution of the glassy phase does not rely on the acidic property of HF, but on the electronegativity of the fluoride in the glass to substitute oxygen and form SiF (Tian et al., 2014). This fact might explain the similar bond strengths recorded for IPS e.max CAD after the use of hydrofluoric acid at 4.9% and 9.5% (Kalavacharla et al., 2015).

To evaluate bonding between ceramic and resin cement, conventional shear and tensile bond tests are generally used (Heintze, 2010; Hu et al., 2016; Matsumura et al., 1997; Otani et al., 2015; Panah et al., 2008; Tzanakakis et al., 2016). These tests might lead to a non-uniform stress distribution along the bonded interface, thus fractures might initiate from flaws or high-stress concentration areas, and the overall bond strength can be wrongly estimated (Anusavice et al., 1980; Bottino et al., 2005; Chadwick et al., 1998; Della Bona & Van Noort, 1995; Della Bona et al., 2003; Leibrock et al., 1999; Pameijer et al., 1996; Van Noort et al., 1989; Versluis et al., 1997). To overcome these limitations, micro-scale tests are preferred to evaluate the properties of the adhesive interface (Sano et

al., 1994; Shimada et al., 2002). The use of specimens with a reduced cross-sectional area (1.0 ± 0.1 mm^2) results in a more uniform stress distribution along the interface, increasing the adhesive failure rate between the ceramic and the resin cements (Table 2) (Armstrong et al., 2010; Heintze et al., 2011; Hu et al., 2016; Phrukkanon et al., 1998; Pisani-Proenca et al., 2006).

Table 2. Failure mode expressed in %: A = adhesive, M = mixed, CR = cohesive in resin cement, CC = cohesive in ceramic.

Failure mode (%)		**HF**	
Etching time		**4.9%**	**9.5%**
VITA Suprinity	0	A = 100 M = 0 CR = 0 CC = 0	A = 100 M = 0 CR = 0 CC = 0
	20	A = 60 M = 40 CR = 0 CC = 0	A = 80 M = 20 CR = 0 CC = 0
	40	A = 57 M = 43 CR = 0 CC = 0	A = 87 M = 13 CR = 0 CC = 0
	60	A = 67 M = 33 CR = 0 CC = 0	A = 87 M = 13 CR = 0 CC = 0
	120	A = 47 M = 53 CR = 0 CC = 0	A = 100 M = 0 CR = 0 CC = 0
IPS e.max CAD	0	A = 100 M = 0 CR = 0 CC = 0	A = 100 M = 0 CR = 0 CC = 0
	20	A = 80 M = 20 CR = 0 CC = 0	A = 80 M = 20 CR = 0 CC = 0
	40	A = 60 M = 40 CR = 0 CC = 0	A = 53 M = 47 CR = 0 CC = 0
	60	A = 53 M = 40 CR = 7 CC = 0	A = 53 M = 47 CR = 0 CC = 0
	120	A = 53 M = 47 CR = 0 CC = 0	A = 47 M = 53 CR = 0 CC = 0

For the present study, μSBS was used since the stresses generated by shearing off the specimens parallel to the bonding interface, seem to replicate more truthfully those occurring in vivo when the luted crown undergoes to displacement forces (Cardoso et al., 1998). In addition, premature failures, especially for the un-treated surfaces, were avoided since the samples were not subjected to trimming after bonding as occur for μTBS (Brum et al., 2011; Lise et al., 2015; Shimada et al., 2002).

One limitation of experimental test design is represented by the lack of seating pressure during the cementation (Scrabeck et al., 1987). Because of the absence of the hydraulic pressure, the injected resin cement might have not deeply penetrated into the micromechanical undercuts, which would explain the similar results found among the groups.

For the specimens fabrication, Rely X Unicem 2 was used. The low post-operative sensitivity after the restoration placement, together with the minimized clinical steps, make this dual-curing self-adhesive cement widely spread among the clinicians (Magne et al., 2015; Sancakli et al., 2014; Yousaf et al., 2014). Through the functional phosphate monomers, Rely X Unicem 2 can chemically bond to the abutment (Gerth et al., 2006; Vrochari et al., 2009) without any pre-treatment of the dental structure, leading to a less time-consuming and sensitive luting procedure (Hu et al., 2016; Krämer et al., 2000). In addition, the phosphate ester

group of RelyX Unicem 2 can directly bond to zirconium oxides, creating chemical bonds between the resin cement and the ZrO_2-containing glass ceramics (Bottino et al., 2005; Hu et al., 2016; Sato et al., 2015; Wolfart et al., 2007). Since VITA Suprinity (8.0-12.0) has more zirconia content than IPS e.max CAD (0.0-8.0), it is reasonable to expect different bond strengths between these two materials (Della Bona et al., 2000; Ilie & Hickel, 2008; Meng et al., 2008; Pollington et al., 2010). Indeed, VITA Suprinity yielded higher μSBS values than IPS e.max CAD on equal pre-treatment protocol. Similarly, Aboushelib & Sleem found that the microtensile bond strength of Celtra Duo (30.4 ± 4.6 MPa) was higher than that of IPS e.max CAD (25.8 ± 4.8 MPa) (Aboushelib & Sleem, 2014). A possible explanation of this outcome might be found in the composition of either the resin cement (Diaz-Arnold et al., 1999; Hooshmand et al., 2012; Kumbuloglu et al., 2005; Marocho et al., 2013) and the lithia silica-based glass ceramic (Hu et al., 2016; Tian et al., 2014; Torres et al., 2009).

Since similar bond strengths were detected after different etching times (Zogheib et al., 2011), 20 seconds should be taken as a reference (Menees et al., 2014). Because of its higher toxicity (Tian et al., 2014), hydrofluoric acid at 9.5% should be avoided since no beneficial bonding effects were noted for neither VITA Suprinity nor IPS e.max CAD.

Conclusion

Based on the outcomes and within the limitations of this study, the following conclusions can be drawn:

- Hydrofluoric acid conditioning improved the adhesion of VITA Suprinity and IPS e.max CAD to resin cements.
- Hydrofluoric acid concentrations significantly influenced bond strength on VITA Suprinity while it has no influence on IPS e.max CAD.
- Hydrofluoric acid application time does not significantly influence the bond strength of VITA Suprinity and IPS e.max CAD.
- VITA Suprinity showed significantly higher bond strength compared with IPS e.max CAD.

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Summary, Conclusions and Future directions

Summary

In **Chapter 1** the physical, chemical and mechanical behaviour of glass ceramics was analyzed with regard to their composition. Different crystalline and glassy contents were seen to influence the resistance, the appearance, the polishability and the bonding ability of metal-free restorations. On the base of these considerations, dental ceramics were classified according to the fusion temperature, fabrication method, crystallinity and sensitivity to the hydrofluoric acid.

The history of lithia silica-based ceramics was briefly described starting from the precursor IPS Empress II, passing through the newer IPS e.max technology and getting to the newest zirconia-reinforced lithium-based ceramics. The developement of the materials followed the evolution of the digital technology, therefore lithia silica-based ceramics are currently available as pressable ingots or machinable blanks. After a brief summary of the history of dental CAD-CAM systems, some remarks were made about the advantages and disadvantages of the digital workflow compared to the traditional, with particular attention to the "in-office" CAD-CAM systems.

The present thesis aimed to evaluate the mechanical properties, the polishability and the bonding ability of lithia silica-based ceramics with different composition and micro-structure.

Prior to the abutment preparing, the restorative material has to be selected with regard to its mechanical characteristics, optical properties and processing methods. By choosing lithium disilicate ceramics, CAD-CAM and heat-pressing are available for the restoration fabrication. In **Chapter 2** the fracture resistance of IPS e.max CAD and IPS e.max Press was evaluated with regard to the opacity of the material. High-translucency, medium-translucency, low-translucency and medium-opacity bar-shaped specimens were fabricated according to the ISO 6872:2015 for both the tested ceramics. A three-point-bending test was performed and the flexural strength was calculated in MPa. IPS e.max CAD and IPS e.max Press showed similar flexural strengths conversely to what declared by the manufacturer. Within the CAD group, statistically significant difference emerged among the tested translucency, since medium-opacity yielded significantly lower values than medium-translucency and low-translucency. From the Scanning Electron Microscope (SEM) analysis, IPS e.max CAD MO showed smaller densely-distributed round-shaped crystals, which might explain

the lower mechanical performances obtained for this formulation. Since lithium disilicates showed similar flexural resistance regardless of the processing method, the selection of the formulation should involve other aspects than the mechanical properties, such as cost, workflow and accuracy.

After milling, lithia silica-based restorations are superficially rough in texture and opaque in appearance. Smooth and lustrous surfaces are desired since they prevent biological and technical complications from ocurring. In **Chapter 3** the efficacy of different finishing and polishing procedures was evaluated for VITA Suprinity and IPS e.max CAD after milling with CEREC In-Lab MC-XL. The surface roughness and gloss were quantitatively evaluated after munual finishing and polishing for 30 and 60 seconds, glazing paste and glazing spray, by using a Profilometer and a Glossmeter, respectively. The effect of the treatments was also qualitatively analysed by means of Scanning Electron Microscope (SEM). The results showed the tested systems were more effective on VITA Suprinity rather than on IPS e.max CAD. By comparing their efficacy, manual finishing and polishing for 60 seconds yielded the lowest roughness and the highest gloss. This indicates that manual finishing and polishing is as reliable as glazing, and this can depend on

either the application time or the characteristics of lithia silica-based glass ceramics.

The last step of the prosthetic workflow regards delivering. Once crystallized and finished, restorations are ready-to-cement with adhesive procedures. At the resin-ceramic interface, mechanical and physico-chemical interactions are necessary to obtain strong and long-lasting bonds. Since lithia silica-based ceramics become micro-retentive and resin cement-trapping after conditioning, the purpose of **Chapter 4** was to evaluate the effect of different hydrofluoric acid etching-times and concentrations on the bond strength of VITA Suprinity and IPS e.max CAD to a dual-curing self-adhesive resin cement.

The intaglio surface was replicated by milling forty-five bars for VITA Suprinity and IPS e.max CAD with CEREC In-Lab MC-XL. The bonding ability was assessed by μSBS test for the quantitative data, and SEM and failures mode analysis for the qualitative observations. Eighteen groups were identified according to the tested etching times of 0, 20, 40, 60 and 120 seconds, and HF concentrations of 4.9% and 9.5%. Once conditioned and silanated, specimens were fabricated with RelyX Unicem 2 and stored in distilled water at 37°C for 24 hours prior to testing. The results showed that significant differences were found

between un-etched and etched surfaces. Bonding to VITA Suprinity and IPS e.max CAD improved by etching, however no statistically significant differences were detected with regard to the conditioning time. Nevertheless, VITA Suprinity yielded better bonding ability than IPS e.max CAD, especially at HF concentration of 4.9%. This indicates that the application of HF at 4.9% for 20 seconds might be considered the most appropriate treatment for lithia silica-based ceramics.

Conclusions

Within the limitation of the present thesis, the following conclusions might be drown:

- The selection of the processing method for lithium disilicate ceramics should be based on cost, workflow and accuracy, since similar mechanical properties were found for IPS e.max Press and IPS e.max CAD.

- Translucency may influence the mechanical properties of lithium disilicate glass ceramics and this is particularly evident for CAD-CAM blanks.

- The high polishability of lithia silica-based blocks allowed manual polishing and finishing to yield statistically significant smoother and more glossy surfaces than the traditional spray or paste glazing and this was related to either the manual application time or the glass ceramic characteristics.

- To obtain surfaces compatibles with the oral environments at reduced working times and costs, manual finishing and polishing might effectively replace tradicional heat-mediated systems for chairside procedures.

- Hydrofluoric acid etching improves the bond strength of lithia silica-based restorations to resin cements. The application of HF at 4.9% for 20

seconds might be considered the most appropriate treatment for lithia silica-based ceramics.

Future directions

Althought lithia silica-based ceramics show long-term reliability as tooth-supported crowns, few data are available when its use involves implant-supported restorations. Even if the flexural strength might be improved by industrially modifing the microstructure and composition of the material, further investigations are necessary to evaluate the mechanical properties with regard to the supporting substrate. Special attention should be paid on the effect of bonding to dentine only, dentine and resin composite, or titanium, on the fracture resistance of lithia silica-based restorations. Additionally, further bonding protocols should be tested with regard to the resin cement and the superficial treatment of the material. Newer self-etching glass-ceramics primers are available for conditioning the intaglio surface, thus in vitro shear or tensile tests might be necessary to better understand their efficacy in promoting the adhesion to lithia silica-based glass ceramics. However, all these aspects should be deeply clarified by long-term randomized clinical trials.

Riassunto, Conclusioni e Direzioni future

Riassunto

Nel **Capitolo 1** il comportamento fisico, chimico e meccanico delle ceramiche vetrose è stato analizzato in funzione della loro composizione. Le diverse proporzioni tra matrice vetrosa e cristallina influenzano la resistenza, l'aspetto estetico e la capacità adesiva dei restauri metal-free. Sulla base di questa asserzione, le ceramiche sono state classificate secondo la temperatura di fusione, il metodo di fabbricazione, il contenuto cristallino e la sensibilità agli agenti mordenzanti, quali l'acido idrofluoridrico.

La storia dei silicati di litio è stata brevemente descritta iniziando dal precursore, IPS Empress II, passando attraverso la più nuova tecnologia IPS e.max e giungendo, infine, alle nuove ceramiche a base di silicati di litio, rinforzate con zirconia. Lo sviluppo dei materiali è andato di pari passo con l'evoluzione della tecnologia digitale, a tal punto che queste ceramiche sono attualmente disponibili sia come cialde pressabili a caldo, sia come blocchi fresabili. Dopo un breve riassunto sulla storia dei sistemi CAD-CAM ad uso dentale, i vantaggi e gli svantaggi del flusso di lavoro digitale sono stati confrontati con quelli del flusso tradizionale, ponendo una speciale attenzione sui sistemi "in-office".

Nella presente tesi, sono state analizzate le proprietà meccaniche, estetiche e adesive delle ceramiche a base di silicati di litio con una diversa micro-struttura e composizione.

Prima di procedere alla preparazione dell'elemento da protesizzare, il materiale restaurativo deve essere selezionato in funzione delle sue proprietà meccaniche, estetiche e di lavorazione. Per le ceramiche a base di disilicato di litio, i flussi di lavoro da poter seguire sono due: digitale attraverso la tecnologia CAD-CAM e tradizionale attraverso la tecnica di pressatura a caldo. Nel **Capitolo 2**, la resistenza alla frattura di due disilicati, l'IPS e.max CAD e l'IPS e.max Press, è stata valutata in funzione dell'opacità del materiale. Per entrambi i materiali testati, sono state prese in esame quattro traslucenze, nello specifico alta-traslucenza, media-traslucenza, bassa-traslucenza e media-opacità, e i campioni a forma di barra sono stati preparati seguento le normative ISO 6872:2008. È stato realizzato un three-point-bending test e la resistenza alla frattura è stata calcolata in MPa. La resistenza alla frattura è risultata essere simile per entrambi i materiali, nonostante il fabbricante dichiari valori leggermente più alti per l'IPS e.max Press. All'interno del gruppo CAD, differenze statisticamente significative sono emerse dal confronto tra le traslucenze testate. Infatti, il disilicato di litio a media-opacità ha ottenuto

valori statisticamente più bassi rispetto a quello a media e bassa traslucenza. Dalla Microscopia Elettronica a Scansione (SEM) emerge che i cristalli dell'IPS e.max CAD MO, morfologicamente sferici e densamente distribuiti, sono più piccoli rispetto a quelli delle altre formulazioni. Poichè la resistenza alla frattura del disilicato di litio non cambia in funzione del metodo di lavorazione, la selezione tra IPS e.max CAD e IPS e.max Press dovrebbe basarsi su altri aspetti quali i costi di produzione, il flusso di lavoro e/o la precisione dei manufatti.

Dopo il fresaggio, i restauri a base di silicati di litio presentano una superficie ruvida e opaca. Diviene fondamentale, quindi, rendere le superfici quanto più lisce e brillanti in modo da evitare l'insorgenza di complicazioni di tipo biologico e/o tecnico. Nel **Capitolo 3**, è stata valutata l'efficacia di diversi sistemi di rifinitura e lucidatura applicati su superfici fresate con il fresatore CEREC In-Lab MC-XL. La ruvidità e la lucentezza di VITA Suprinity e IPS e.max CAD, sono state misurate mediante un profilometro e un glossmetro, rispettivamente, dopo la rifinitura e lucidatura manuale per 30 e 60 secondi, e la glasure realizzata con pasta e spray. Qualitativamente, invece, i campioni sono stati analizzati mediante Microscopia Elettronica a Scansione (SEM). Dai risultati ottenuti si evince che la rifinitura di VITA Suprinity risulta

essere migliore di quella di IPS e.max CAD. Mettendo a confronto i quattro sistemi testati, il sistema manuale per 60 secondi ha permesso di ottenere le superfici più liscie e lucide. Pertanto, i sistemi manuali possono essere considerati una valida alternativa a quelli forno-dipendenti, e il risultato finale in termini di tessitura e lucentezza superficiale sarà legato tanto al tempo di applicazione quando alle caratteristiche intrinseche della ceramica vetrosa.

L'ultimo passaggio del flusso di lavoro protesico riguarda la consegna del manufatto. Una volta cristallizzato e rifinito, il restauro è pronto per essere cementato con tecniche adesive. Le interazioni meccaniche e fisico-chimiche assicurano un legame adesivo forte e durevole tra ceramica e resina composita. Poichè la componente vetrosa si dissolve dopo l'applicazione dell'acido idrofluoridrico e la superficie diventa, quindi, ruvida e micro-ritentiva per il cemento resinoso, lo scopo del **Capitolo 4** è stato quello di valutare l'effetto di diversi tempi di applicazione e di due concentrazioni di acido idrofluoridrico sulla forza di adesione di un cemento duale auto-adesivo a due ceramiche a base di silicati di litio.

La superficie interna del restauro è stata riprodotta fresando quarantacinque barrette, rispettivamente per VITA Suprinity e IPS e.max

CAD, con il fresatore CEREC In-Lab MC-XL. La capacità di adesione è stata valutata quantitativamente attraverso il test di μSBS, e qualitativamente attraverso la Microscopia Elettronica a Scansione (SEM) e l'analisi delle modalità di fallimento. Diciotto gruppi sono stati formati in funzione del tempo di applicazione dell'acido idrofluoridrico, rispettivamente di 0, 20, 40, 60 e 120 secondi, e della sua concentrazione, rispettivamente al 4.9% e 9.5%. In seguito alla mordenzatura e silanizzazione, i campioni sono stati preparati con il cemento RelyX Unicem 2 e conservati in acqua distillata a 37°C per 24 ore prima di essere testati. I risultati mostrano differenze statisticamente significative tra le superfici non mordenzate (0 secondi) e quelle mordenzate. La forza di adesione a VITA Suprinity e a IPS e.max CAD migliora dopo la mordenzatura, senza però variare in funzione del tempo di applicazione dell'acido idrofluoridrico. Inoltre, VITA Suprinity ha mostrato una maggior forza di adesione rispetto a IPS e.max CAD, e questo è specialmente evidente per la concentrazione al 4.9%. Sulla base dei risultati ottenuti, si evince che la forza di adesione varia in funzione della composizione della ceramica. Ciò nonostante, l'applicazione dell'acido idrofluoridrico al 4.9% per 20 secondi può essere considerata il trattamento più appropriato per le superfici dei restauri a base di silicati di litio.

Conclusioni

Nonostante i limiti della presente tesi, le seguenti conclusioni possono essere tratte:

- Dal momento che le proprietà meccaniche del disilicato di litio non variano in funzione del metodo di lavorazione, la scelta del flusso di lavoro si dovrebbe basare su altri aspetti quali i costi, i tempi di lavorazione e la precisione dei manufatti protesici.
- La traslucenza può influenzare le proprietà meccaniche del disilicato di litio e questo è particolarmente evidente per i blocchi ad uso CAD-CAM.
- La capacità delle ceramiche a base di silicati di litio, di essere rifinite e lucidate manualmente dipende dal tempo di applicazione e dalle caratteristiche intrinseche della ceramica stessa.
- Seguendo un flusso di lavoro digitale, i sistemi di rifinitura manuale possono efficacemente sostituire quelli tradizionali legati all'uso del forno, consentendo di ottenere superfici compatibili con il cavo orale in tempi e costi ridotti.
- La mordenzatura con acido idrofluoridrico migliora la forza di adesione dei cementi resinosi ai restauri a base di silicati di litio.
- L'applicazione dell'acido idrofluoridrico al 4.9% per 20 secondi può rappresentare il trattamento più appropriato per la cementazione adesiva delle ceramiche a base di silicati di litio.

Direzioni future

Sebbene le ceramiche a base di silicati di litio mostrino una buona affidabilità a lungo termine quando vengono impiegate per protesizzare singoli elementi dentali, pochi sono i dati disponibili nella letteratura per quanto riguarda il loro uso nei restauri a supporto implantare. Anche se la resistenza alla frattura può essere migliorata attraverso modifiche nella micro-struttura e nella composizione del materiale, sono necessarie ulteriori ricerche mirate a valutare il comportamento meccanico in funzione del sustrato di supporto. Una speciale attenzione andrebbe prestata all'effetto dell'adesione alla sola dentina, alla dentina e alla resina composita, come succede per i monconi ricostruiti, e al titanio, sulla resistenza alla frattura dei restauri a base di silicati di litio. Ulteriori protocolli di adesione andrebbero testati in funzione dei vari cementi disponibili sul mercato e del trattamento di superficie della ceramica. Nuovi primers auto-mordenzanti sono infatti disponibili sul mercato per il trattamento delle ceramiche vetrose, il che dimostra il sempre più crescente interesse verso questo argomento da parte delle industrie. Test di shear o di tensile saranno necessari per meglio comprendere l'efficacia di questi nuovi prodotti nel promuovere l'adesione alle vetro-ceramiche. Tuttavia, tutti questi aspetti dovrebbero essere maggiormente chiariti attravero studi clinici randomizzati a lungo termine.

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Curriculum Vitae

Riccardo Fabian Fonzar

Date of birth: August 9th, 1987

Place of birth: Portogruaro (Venice), Italy

Higher education: Degree in Dentistry (2012), University of Valencia, Valencia, Spain

Professional position

Private practice

Office: Via dei Tigli 54/56, Villa Primavera - Campoformido (Udine), 33030, Italy

Telephone: +39 0432 69 500

Fax: +39 0432 699 441

E-mail: riccardofabianfonzar@gmail.com

Publications

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