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Etching conditions and surface changes: a guideline for glass-ceramic materials

Possibilidades de condicionamento e alterações superficiais: uma diretriz para materiais vitrocerâmicos

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ABSTRACT

Statement of Problem: In terms of ceramic restorations, the achievement of a reliable adhesion involves following specific protocols according to the ceramic material's microstructure. **Purpose:** The aim of this comprehensive review is to enlighten concepts and characteristics regarding adhesion/luting procedures to glass-ceramic materials, guiding dental clinicians to ensure a long-term success of their glass-ceramic restorations. **Materials and Methods:** These protocols represent scientifically based treatments applied to establish micromechanical retention and chemical adhesion among the different interfaces involved in the adhesive procedure (which comprises ceramic restoration, resin cement and tooth substrate). **Results:** Regarding glass-ceramic restorations, the protocols carried out to produce topographical changes on the cementation surface normally improve bond ability, and consequently, lead to an increased load-bearing capacity under fatigue of the restorations. **Conclusions:** It is imperative to elucidate the concepts related to the distinct glass-ceramic materials available in the dental market and clarify the specifications for the correct surface treatment of each material, since each one presents different microstructure due to their singular chemical composition and arrangement.

KEYWORDS

Acid etching, Adhesive cementation, Silanization, Surface treatment, Vitreous ceramics.

RESUMO

Declaração do Problema: Em termos de restaurações cerâmicas, a obtenção de uma adesão confiável envolve seguir protocolos específicos de acordo com a microestrutura do material cerâmico. **Objetivo:** O objetivo desta revisão é esclarecer conceitos e características sobre procedimentos de adesão/cimentação para materiais vitrocerâmicos, orientando os clínicos a garantir o sucesso a longo prazo de suas restaurações vitrocerâmicas. **Materiais e Métodos:** Esses protocolos representam tratamentos com base científica aplicados para estabelecer retenção micromecânica e adesão química entre as diferentes interfaces envolvidas no procedimento adesivo (que compreende restauração cerâmica, cimento resinoso e substrato dentário). **Resultados:** Em relação às restaurações vitrocerâmicas, os protocolos realizados para produzir mudanças topográficas na superfície de cimentação normalmente melhoram a capacidade de adesão e, consequentemente, levam a um aumento da capacidade de suporte de carga sob fadiga das restaurações. **Conclusões:** É imperativo elucidar os conceitos relacionados aos distintos materiais vitrocerâmicos disponíveis no mercado odontológico e esclarecer as especificações para o tratamento de superfície correto de cada material, uma vez que cada um apresenta microestrutura diferente devido à sua composição química e arranjo singulares.

PALAVRAS-CHAVE

Condicionamento ácido, Cimentação adesiva, Silanização, Tratamento de superfície, Cerâmica vítrea.

CRITICAL REVIEW

Dental clinicians often deal with complex situations when replacing missing teeth or failed restorations. To meet these challenges, manufacturers have developed a wide variety of ceramic materials, each one with distinct chemical compositions and mechanical properties. These materials are no longer limited to traditional layering techniques; they now include heat-pressing and computer-aided design/computer-aided manufacturing (CAD/CAM) methods [1].

Among the available materials for indirect restorations, glass-ceramics (with its modifications) are a particularly appealing option due to their combination of aesthetic properties and moderate strength, derived from their vitreous phase reinforced by crystals [2]. For glass-ceramic restorations to achieve sufficient load-bearing capacity, adhesive cementation is essential, as it allows better load distribution throughout the restoration-cement-tooth interface [3]. Prior to cementation, the internal surface of the glass-ceramic restoration must be treated to create surface porosities or irregularities that facilitate the penetration of resin cement, thereby improving bond strength [1]. The surface characteristics rely on the microstructure and composition of the chosen ceramic [4].

Hydrofluoric acid (HF) etching is a well-established method for ensuring strong adhesion to glass-ceramics [5-7]. This technique selectively attacks the silica phase of the ceramic, creating a topographic pattern that enhances bonding by increasing surface roughness and free surface energy [4]. Furthermore, HF etching is preferred when compared to sandblasting, as it generates greater bond strength and prevents the formation of cracks on the ceramic surface, which can lead to premature bond failures [4]. Despite its proven effectiveness, HF is a hazardous substance that can cause severe burns and tissue damage upon contact [8], and for that, it is not recommended for intraoral use [9].

While creating a microrough surface is beneficial for bonding, overly aggressive pretreatment can degrade the glass-ceramic surface, potentially weakening the restoration [10,11]. Thompson and Anusavice [12] observed that glass-ceramic crown failures often originated at the cementation surface, where pretreatment procedures were applied, suggesting that HF over-etching can be a significant cause of failure in these materials [11].

Several commercial alternatives to HF etching have been introduced for glass-ceramics, including alumina particle abrasion, laser irradiation, nonthermal plasma treatment, silica vapor phase deposition, selective infiltration etching, ceramic primers, and combinations of these techniques [5,13-22]. One such alternative is a ceramic primer developed by Ivoclar Vivadent, designed to simplify the process by combining HF etching and silane application into a single bottle, achieving comparable adhesion and fatigue strength [21-23]. However, most of these alternative methods either lack substantial evidence in the literature or do not surpass the effectiveness of HF etching followed by silane application, highlighting the need for further research [20].

Given the complexity of the available surface treatment options and its impact on reliability/clinical longevity of glass-ceramic restorations, it is essential to provide dental clinicians with clear guidelines to assist in selecting the most appropriate pretreatment for each glass-ceramic material. Therefore, the aim of this study is to offer a comprehensive overview of the surface treatments for cementation of glass-ceramic restorations.

METHODS

Scientific papers in English and no period restriction, including systematic reviews, clinical trials, and in vitro studies, were manually selected from dental journals indexed in PubMed (via MEDLINE) and Google Scholar, without restrictions on publication date. The selected papers were in English and focused on glass-ceramic surface treatments and luting procedures. The information was then organized and presented in a simplified manner to ensure clarity and accessibility for dental clinicians.

Glass-ceramic characterization

Feldspathic ceramic

Feldspathic ceramics present noteworthy characteristics such as high aesthetics and strong bonding to resin cements, despite having a relatively low flexural strength of approximately 150 MPa [24]. Belli et al. [25] evaluated feldspathic ceramic microstructure and identified the presence of two distinct phases: a highly etchable glass phase and an acid-resistant crystalline phase. Consequently, after hydrofluoric acid etching, the high-fusion glass particles are dissolved meanwhile the low-fusion glassy matrix undergoes a less selective dissolution process [25]. This results in a highly porous material, well-documented in the literature as "honeycomb-like appearance" [26,27]. The recommendation for etching feldspathic ceramic restorations is described in Table I according to commercial brand and manufacturer.

Leucite-enhanced ceramic

Leucite-enhanced glass-ceramic exhibits a flexural strength ranging from 120 to 180 MPa, due to its finely dispersed leucite crystal reinforcement [28]. Leucite fillers are uniformly distributed throughout the glass, typically at concentrations between 35 to 45% by volume [28]. Belli et al. [25] examined the shape and size of the dissolved crystallites following etching, revealing well-dispersed, round and slightly elongated cavities ranging in size from 0.5 to 3μm.

Table I - Surface treatments suggested by the manufacturers according to the glass-ceramic composition

Ceramic	Surface treatment	Commercial Name (Brand)
Feldspathic	5% HF* for 60 s + rinse with air/water spray for 30 seconds + silane	Vitablocs Mark II (VITA)
		Vita PM9 (VITA)
		CEREC Blocs
		GC initial LRF (GC)
		G Ceram
		IPS Classic (Ivoclar Vivadent)
		Super Porcelain NX3 (Noritake)
Leucite-enhanced	5% HF* for 60 s + rinse with air/water spray for 30 seconds + silane	IPS Empress Esthetic and IPS Empress CAD (Ivoclar Vivadent)
		GC Initial™ LRF BLOCK (GC Corporation)
		Optimum Pressable CeramicOPC® (Jeneric/Pentron)
		Finesse® (Dentsply)
Lithium disilicate	5% HF* for 20 s + rinse with air/water spray for 30 seconds + silane or ME&P**	IPS e.max Press and IPS e.max CAD (Ivoclar Vivadent)
	5% HF* for 20 s + rinse with air/water spray for 30 seconds + silane	Rosetta SM (HAASBio)
		n!ce (Straumann)
		Vintage LD Press (Shofu)
		Vita Ambria (VITA)
	4% HF* for 30 s + rinse with air/water spray for 30 seconds + silane	Amber Mill/Press/Press Master (HASSBio)
	5% HF* for 30 s + rinse with air/water spray for 30 seconds + silane	CEREC Tessera (Dentsply)
	5-9% HF* for 20 s + rinse with air/water spray for 30 seconds + silane	LiSi Press/Block (GC)
		K2 LI (Yeti GmbH)
		Rainbow LS (GENOSS)
		Creation SL Press (Creation Willi Geller)
		Livento Press (Cendres + Metaux)
Zirconia-reinforced lithium silicate	5% HF* for 20 s + rinse with air/water spray for 30 seconds + silane	Vita Suprinity (VITA)
		Celtra DUO (Dentsply)
	5% HF* for 30 s + rinse with air/water spray for 30 seconds + silane	Celtra Press (Dentsply)
PICN	5% HF* for 60 s + rinse with air/water spray for 30 seconds + silane	Vita Enamic (VITA)

*HF: hydrofluoric acid; **ME&P: Monobond Etch&Prime (see manufacturer recommendation for using).

According to Apel et al. [29] the crystalline content in leucite-enhanced ceramics is not very effective in promoting crack deflection (toughening mechanism), commonly observed in lithium disilicate glass-ceramics [30]. However, the high glass phase content (approximately 60% by volume for Empress CAD) plays an essential role in aesthetics and in hydrofluoric acid sensitivity, enhancing adhesion [31]. Usually, the manufacturers recommend a 5% HF application for 1 minute to etch leucite-enhanced ceramics (Table I).

Lithium disilicate-based ceramic

Lithia-based glass ceramics, particularly lithium disilicate (LDS), have gained popularity since their introduction in dentistry due to their ability to chemically bond, reflect light similarly to natural enamel, demonstrate high long-term survival rates, exhibit enhanced mechanical properties, and provide precise fitting accuracy through the heat-press technique [32-34]. Consequently, lithium disilicate has proven to be suitable for various clinical applications, including inlays, onlays, veneers, crowns, three-unit bridges up to the premolars, and implant abutments and crowns [34].

These materials offer significantly higher flexural strength compared to conventional glass ceramics, with values ranging from 360 to 440 MPa, while maintaining excellent translucency due to a specialized firing process [24]. The mechanical properties are enhanced by lithium disilicate crystals (approximately 70% by volume), which are needle-shaped and measure about 0.5 to 4 μm, randomly embedded in the glassy matrix [35]. These randomly oriented crystals act as “crack stoppers,” contributing to increased flexural strength and improved fatigue resistance [36].

Lithium disilicate can be acid-etched and silanized, facilitating a straightforward and predictable adhesive cementation process that has been shown to provide long-lasting clinical results [34]. Various concentrations of hydrofluoric acid (HF) are available on the market, capable of modifying the microstructure through partial glass dissolution, exposing the crystalline phase, increasing surface energy, and enabling micromechanical interlocking with the resin cement via microporosities created on the ceramic surface [37,38]. Additionally, a chemical bond is formed through the interaction of hydrofluoric acid with the ceramic surface, resulting in hexafluorosilicate formation. However, it is crucial to follow the appropriate protocol for each material, as over-etching can weaken the ceramic by causing substantial surface disruption and the formation of flaws, thereby compromising its mechanical performance [11]. The recommended etching protocol for lithium disilicate ceramics is controversial among the manufacturers and recently includes the application of Monobond Etch&Prime or the use of HF from 4 to 9.5% for 20 up to 30 seconds, as outlined by the manufacturer’s guidelines (Table I).

Zirconia-reinforced lithium silicate

Zirconia-reinforced lithium silicate (ZLS) has been introduced to improve mechanical and aesthetic properties of lithium disilicate [39], achieved through the presence of metasilicate and zirconia crystals into the glass matrix [40]. This material consists of a glassy matrix composed of lithium metasilicates and lithium orthophosphates [2], that generate particles of lithium silicate after the crystallization process. This combination creates a material with a flexural strength between 550 and 674 MPa [41,42]. The reinforcement of the glassy phase makes the material useful for anterior and posterior full crowns, partial occlusal coverages, and veneer restorations [2].

When compared to a conventional lithium disilicate ceramic, ZLS presents a lower percentage of crystal phase content, ranging from 40 to 57% as opposed to 70% [43]. However, ZLS incorporates smaller crystals, and the glassy matrix is reinforced by the highly dispersed zirconium dioxide (approximately 10% by weight), which is assumed to enhance the strength of the glassy phase [2,42]. Notably, despite the presence of zirconium dioxide crystals, it is important to highlight that ZLS can be etched with HF, a feature not observed in conventional zirconia-based systems. The manufacturer recommendation for etching ZLS is displayed in Table I.

Polymer-infiltrated-ceramic-network (PICN)

Polymer-infiltrated ceramic network (PICN) materials were developed to combine the best features of all-ceramic materials with those of dental composites. These materials integrate the elastic modulus of polymers, which is more compatible with dentin, with the aesthetic qualities and chemical stability of ceramics [2].

PICNs consist of an inorganic component (a porous ceramic, similar to feldspathic ceramics) comprising approximately 86 wt%, and an organic polymeric component making up around 14 wt% (Vita Zahnfabrik). According to Coldea et al. [44], this combination offers several advantages, including reduced brittleness, improved fracture toughness, and increased rigidity and hardness, while enhancing flexibility and machinability compared to conventional glass-ceramics.

PICNs also exhibit higher flexural and fatigue strengths than feldspathic ceramics [25], with an elastic modulus closely matching that of natural tooth tissue [45]. However, they are more prone to slow crack growth [35]. An analysis of PICN microstructures by Belli et al. [25] revealed a crystalline structure similar to feldspathic ceramics, with a glassy phase predominantly composed of pure glass.

Due to the ease with which the polymer phase of PICNs can be repaired using composite resins [46], these materials offer a compelling combination of the properties of glass-ceramics and composites. As such, they are indicated for anterior and posterior crowns, partial occlusal coverage, and veneer restorations, and are commercially available in CAD/CAM blocks [2]. Table I demonstrates the manufacturer's recommendations for etching PICN materials.

CLINICAL CONSIDERATIONS

Ceramic etching and silane coupling agents

Bi-functional trialkoxy silanes contain two distinct functional groups: an alkoxy group and a carbon-carbon double bond (C=C). The alkoxy group can bond with inorganic materials, while the carbon-carbon double bond attaches to organic materials. In adhesive cementation, silane coupling agents are employed to enhance bonding between the luting agent and silica-based restorative materials [47]. This effectiveness is due to chemical bonding and improved surface wettability when a silane agent is applied to ceramic surfaces. Silane agents should be applied in thin films. If the siloxane film is too thick, it may lead to cohesive failure; if too thin, it may result in incomplete coverage of the ceramic surface, causing inadequate contact between the silanized substrate and the resin cement [48].

The low viscosity of silane agents is essential for proper wetting and close contact with the ceramic substrate [48], especially after ceramic etching. Thanks to its low viscosity, the silane is able to penetrate the irregularities produced by HF etching, promoting a large surface area with effective chemical bonding between the ceramic and the resin luting agent. During drying, silane forms a thin, branched siloxane film of approximately 20-100 nm thick on the etched surface [48]. However, studies have indicated that applying silane alone to ceramic surfaces does not improve bond strength because of insufficient mechanical retention [49-51]. So, the efficacy of the silanization processes is strongly dependent on a correct surface treatment of the ceramic. In this sense, errors in the conditioning of ceramic surfaces are very harmful. One of the errors that can occur is the over-etching of the surfaces, which generates deep defects that make it difficult for the infiltration of the luting agent. In these situations, the application of the unfilled resin, like the non-solvated bond of the adhesive system over silane can provide better infiltration to the irregularities created on etched surfaces, irrespective of the etching time, as shown in a previous study [52].

The benefits of using resin luting agents

Given the inherent brittleness and limited flexural strength of glass-ceramics, adhesive cementation with composite resin-based materials is recommended to enhance the fracture strength of the restoration [53-58]. Silica-based glass ceramic restorations luted with resin composites demonstrate superior clinical survival rates compared to those cemented with glass ionomer [59] or zinc phosphate cements [60].

An appropriate resin-bonding protocol can significantly increase the fracture strength of dental ceramics, thereby optimizing the performance of indirect esthetic restorations [61]. Resin cements also play a vital role in blocking crack propagation by filling and healing cracks and irregularities on the intaglio surface of the restoration [37,53,62], which improves the overall longevity of ceramic restorations by optimizing load transmission across the restoration assembly [63]. This behavior includes the irregularities produced by an ordinary HF etching. However, if the glass-ceramic is overetched, the use of an unfilled resin over silane also provides better infiltration to the irregularities created on etched surfaces, irrespective of the conditioning time, reinforcing the material [63].

Important aspects before cementing glass-ceramic restorations

After performing HF etching on glass-ceramic restorations (as outlined in Table I), precipitates and salts often accumulate on the surface due to the removal of the glassy matrix [64]. To effectively remove these residues from feldspathic ceramics, the most effective method appears to be the use of an ultrasonic bath with distilled water [64-66]. However, Belli et al. [67] demonstrated that for leucite and lithium disilicate ceramics, an ultrasonic bath is just as effective as air/water spray in producing good bond strength and removing the residue layer. Similarly, Magalhães et al. [68] found that a simple air/water spray for 30 seconds is sufficient to remove precipitates from lithium disilicate surfaces following 20 seconds of HF etching, thus negating the need for an ultrasonic bath and reducing chairside preparation time.

A universal cleaning paste (Ivoclean, Ivoclar Vivadent) is available for cleansing etched or contaminated surfaces, such as those exposed to saliva or blood, prior to cementation. This sodium hydroxide-based product effectively removes various contaminants from ceramic surfaces, ensuring a clean surface for resin bonding when used before or after etching [69-71]. Another product, ZirClean (Bisco), is designed to eliminate phosphate contamination on zirconia and glass-ceramic surfaces after intraoral try-ins, ensuring reliable cementation results.

CONCLUSION

The cementation of glass-ceramic restorations requires meticulous selection of materials and techniques. One of these techniques is the etching of ceramic surfaces. Therefore, the clinician must be aware of:

The glass-ceramics' microstructure influences the etching time, and according to the HF concentration, the etching time must be adjusted based on the ceramic manufacturer's instruction.

HF is toxic even at low concentrations and short exposure times. Therefore, HF must be carefully handled with adequate protraction to avoid severe tissue damage to the professional staff and the patients.

In case of over-etching of the ceramic surface, the use of an unfilled resin (non-solvated adhesive) may be necessary to correctly fill the porosity produced by the HF.

The application of a silane coupling agent is vital to maximize the glass-ceramic bonding, However, solely HF etching or the silanization is insufficient for ensuring robust and durable adhesion. The combination of the micromechanical and chemical bond is the key to the durability of adhesive cementation.

Author's Contributions

CP: Conceptualization, Writing – Review & Editing. JPP: Conceptualization, Data Curation, Formal Analysis, Supervision. MCGE: Methodology, Validation, Formal Analysis, Writing, Data Curation, Supervision. GKRP: Formal Analysis, Writing, Data Curation. LSG: Methodology, Validation, Formal Analysis, Writing, Data Curation, Supervision.

Conflict of Interest

The authors hereby declare that there are no conflicts of interest regarding the research, authorship, or publication of this article. All authors have approved the final version of the manuscript and agree with its submission.

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Regulatory Statement

As this manuscript is a literature review, it does not involve any original studies with human participants or animals conducted by the authors, and therefore did not require ethical approval.

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