



**UNIVERSIDADE FEDERAL DO CEARÁ**  
**FACULDADE FARMÁCIA ODONTOLOGIA E ENFERMAGEM**  
**PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA**

**MAUDIELA ISABEL ARITA TORRES**

**EFEITO DE UM PRIMER CERÂMICO AUTOCONDICIONANTE NAS  
PROPRIEDADES DE SUPERFÍCIE, MECÂNICAS E ADESIVAS DE CERÂMICA À  
BASE DE DISSILICATO DE LÍTIO CIMENTADAS EM DIFERENTES  
SUBSTRATOS**

**FORTALEZA**

**2025**

MAUDIELA ISABEL ARITA TORRES

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DIFERENTES SUBSTRATOS

Dissertação apresentada ao Programa de Pós-Graduação em Odontologia da Universidade Federal do Ceará, como requisito à obtenção do título de Mestre em Odontologia, área de concentração Clínica Odontológica.

Orientador: Prof. Dr. Raniel Fernandes Peixoto.

FORTALEZA

2025

Dados Internacionais de Catalogação na Publicação  
Universidade Federal do Ceará  
Sistema de Bibliotecas  
Gerada automaticamente pelo módulo Catalog, mediante os dados fornecidos pelo(a) autor(a)

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T646e Torres, Maudiela Isabel.

Efeito de um primer cerâmico autocondicionante nas propriedades De superfície, mecânicas e adesivas de cerâmica à base de dissilicato de lítio cimentadas em diferentes substratos : Efeito de um primer cerâmico autocondicionante nas propriedades de superfície, mecânicas e adesivas de cerâmica à base de dissilicato de lítio cimentadas em diferentes substratos / Maudiela Isabel Torres. – 2023.  
53 f. : il.

Dissertação (mestrado) – Universidade Federal do Ceará, Faculdade de Farmácia, Odontologia e Enfermagem, Programa de Pós-Graduação em Enfermagem, Fortaleza, 2023.  
Orientação: Prof. Dr. Raniel Fernandes Peixoto .

1. Cerâmicas vítreas odontológicas. 2. Adesão. 3. Primer cerâmico autocondicionante. I. Título.  
CDD 610.73

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## AGRADECIMENTOS

Agradeço, primeiramente, a Deus, pela dádiva da vida, pela saúde e pela força concedida em todos os momentos desta caminhada.

À minha mãe, **Sonia Esperanza Torres**, exemplo de coragem, resiliência e amor incondicional, meu eterno reconhecimento e gratidão. Amo você!

Aos meus irmãos **Gabriel Jusán Arita Torres, Julio Octavio Arita Torres, Pablo César Arita Torres, David Roberto Arita Torres e José Juan Arita Torres**, por cada palavra de incentivo e apoio silencioso ao longo do percurso. Amo vocês!

Às minhas cunhadas **Carolina Guedes de Arita e Nicole Ramos de Arita**, pelo carinho constante e apoio sincero ao longo desta jornada, sou grata por cada momento de incentivo e ajuda dedicados. Admiro e tenho profundo afeto por vocês!

Às parceiras **Sussan Daniela Salazar e Verônica Cecilia Mejía**, cuja amizade e presença foram fundamentais nos momentos mais desafiadores.

Aos técnicos **Karine Barros Nojosa e Manoel Florindo Junior**, pelo suporte técnico cuidadoso, pelas trocas valiosas, choradas e risadas e pela disposição constante em contribuir com esta pesquisa. Karine, meu sincero agradecimento por toda a ajuda e apoio que você me dedicou.

À **Universidade Federal do Ceará**, por ter me acolhido e formado com excelência,

À **CAPES**, pelo apoio financeiro essencial à realização deste trabalho,

À **EMBRAPA**, por sua contribuição científica e institucional.

Agradeço às amigas da pós-graduação, **Francisbênia Alves Silvestre e Nicole Escórcio de Meneses**, pela parceria, incentivo mútuo e tantas trocas valiosas ao longo dessa jornada, que tornaram o caminho mais leve! Admiro imensamente vocês!

Aos dedicados alunos **Gabriel Freitas de Souza, Isaac Augusto Dantas Nogueira, Lorena Raquel Matias Xavier e Luna Gabriela Reis Merise**, que me inspiraram com sua curiosidade e vontade de aprender academicamente mais também pelas enriquecedoras trocas culturais que compartilhamos

Estendo meu reconhecimento a cada pessoa do Brasil e meu país de origem, cujo esforço diário, muitas vezes invisível, também constrói os caminhos para fazer parte dessa conquista.

E, de modo muito especial, ao meu orientador, **Professor Raniel Fernandes Peixoto**, não conseguirei colocar as palavras de gratidão e admiração por o senhor, por ter paciência, pela orientação generosa, pela confiança depositada em meu trabalho e por ser um verdadeiro exemplo de dedicação acadêmica e excelente qualidade humana. Poucas coisas são tão especiais quanto ter ao lado um orientador, que também é um verdadeiro amigo, em quem confiar e acreditar. Muito obrigada por ser sempre tão gentil e tirar todas as dúvidas que tive. Sou imensamente grata por todo o apoio e dedicação. Obrigada por tudo sempre!

## RESUMO

**Introdução:** As cerâmicas odontológicas são amplamente utilizadas devido à sua estética, biocompatibilidade e desempenho mecânico. O protocolo padrão de ácido fluorídrico (HF) + silano melhora a adesão, mas apresenta desvantagens como toxicidade e sensibilidade técnica. Primers cerâmicos autocondicionantes, como o Monobond Etch & Prime (MEP), oferecem uma alternativa simplificada. No entanto, sua efetividade a longo prazo ainda é incerta, especialmente em diferentes substratos.

**Objetivo:** Este estudo avaliou o efeito do MEP, com ou sem jateamento com óxido de alumínio, sobre as propriedades de superfície, mecânicas e adesivas de cerâmicas à base de dissilicato de lítio (DL), cimentadas em diferentes substratos (dentina e resina composta), em comparação com o tratamento com ácido fluorídrico e silano (HF+S).

**Materiais e Métodos:** Espécimes de DL foram preparados em dois formatos: quadrados (10×10×4 mm, N=134) para análise de rugosidade de superfície ( $\Delta Sa$ ), ângulo de contato (CA), resistência adesiva à microtração ( $\mu RAT$ ) e microscopia eletrônica de varredura (MEV); e retangulares (15×2.5×1.5 mm, N=48) para resistência à flexão em três pontos ( $\sigma_{f3P}$ ). Quatro grupos foram testados de acordo com o tratamento de superfície: HF+S jateado, HF+S polido, MEP jateado e MEP polido. Após o tratamento, foram medidos  $\Delta Sa$ , CA e  $\sigma_{f3P}$ . Blocos de DL foram cimentados sobre resina composta ou dentina para avaliação da  $\mu RAT$  antes e após termociclagem (TC, 10.000 ciclos, 5 °C/55 °C, 30 s de permanência).  $\Delta Sa$  e CA foram analisados por *one-way* ANOVA/Tukey;  $\sigma_{f3P}$ , por *two-way* ANOVA medidas repetidas/Tukey; e  $\mu RAT$ , por ANOVA mista/Tukey.

**Resultados:** O jateamento aumentou significativamente ( $p < 0,05$ ) o  $\Delta Sa$  (HF+S:  $1,16 \pm 0,12$   $\mu m$ ; MEP:  $0,85 \pm 0,02$   $\mu m$ ) em comparação aos espécimes polidos (HF+S:  $0,47 \pm 0,04$   $\mu m$ ; MEP:  $0,38 \pm 0,04$   $\mu m$ ). O menor CA foi observado no grupo MEP jateado ( $27,50 \pm 4,09^\circ$ ). Ambos os grupos tratados com MEP apresentaram valor de  $\sigma_{f3P}$  semelhantes ao controle ( $p > 0,05$ ) e superiores ao HF+S ( $p < 0,05$ ). A termociclagem reduziu a  $\mu RAT$  em todos os grupos, mas os espécimes tratados com MEP mostraram maior estabilidade da resistência adesiva ( $\Delta \mu RAT \leq 2,0$  MPa). Superfícies jateadas apresentaram consistentemente maior  $\mu RAT$  do que as polidas. HF+S e MEP apresentaram desempenho semelhante, exceto em resina composta antes do envelhecimento, onde HF+S foi superior.

**Conclusão:** MEP com jateamento apresentou  $\mu$ RAT semelhante ao HF+S e maior estabilidade adesiva após TC. Além disso, MEP melhorou a molhabilidade, preservou a  $\sigma_{f3P}$  em comparação ao HF, reduziu a  $\Delta Sa$  e demonstrou desempenho consistente em dentina e resina composta.

**Palavras-chave:** Primer cerâmico autocondicionante. Monobond Etch & Prime. Ácido fluorídrico. Dissilicato de lítio. Cerâmicas vítreas odontológicas. Adesão.



## ABSTRACT

**Introduction:** Dental ceramics are widely used due to their esthetics, biocompatibility, and mechanical performance. The standard HF + silane protocol improves adhesion but has drawbacks like toxicity and technique sensitivity. Self-etching ceramic primers (SeCPs), such as Monobond Etch & Prime (MEP), offer a simplified alternative. However, their long-term effectiveness remains uncertain, especially on different substrates.

**Objective:** This study evaluated the effect of the MEP, with or without aluminum oxide air abrasion, on the surface, mechanical, and adhesive properties of lithium disilicate-based ceramics (LD), luted to different substrates (dentin and composite resin), compared to conventional hydrofluoric acid and silane treatment (HF+S).

**Material and Methods:** LD specimens were prepared in two formats: square-shaped (10×10×4 mm, N=134) for surface roughness ( $\Delta Sa$ ), contact angle (CA), microtensile bond strength ( $\mu TBS$ ), and scanning electron microscopy (SEM); and rectangular-shaped (15×2.5×1.5 mm, N=48) for three-point flexural strength ( $\sigma f3P$ ). Four groups were tested according to surface treatment: HF+S sandblasted, HF+S polished, MEP sandblasted, and MEP polished. After treatment,  $\Delta Sa$ , CA, and  $\sigma f3P$  were measured. LD blocks were cemented to either composite resin or dentin to evaluate  $\mu TBS$  before and after thermocycling (TC, 10,000 cycles, 5 °C/55 °C, 30 s dwell time).  $\Delta Sa$  and CA were analyzed using one-way ANOVA/Tukey;  $\sigma f3P$ , using two-way repeated-measures ANOVA/Tukey; and  $\mu TBS$ , using mixed ANOVA/Tukey.

**Results:** Air abrasion significantly increased ( $p < 0.05$ )  $\Delta Sa$  (HF+S:  $1.16 \pm 0.12 \mu m$ ; MEP:  $0.85 \pm 0.02 \mu m$ ) compared to polished specimens (HF+S:  $0.47 \pm 0.04 \mu m$ ; MEP:  $0.38 \pm 0.04 \mu m$ ). The lowest CA was observed in the MEP sandblasted group ( $27.50 \pm 4.09^\circ$ ). Both MEP-treated groups showed  $\sigma f3P$  values similar to the control ( $p > 0.05$ ) and higher than HF ( $p < 0.05$ ). Thermocycling reduced  $\mu TBS$  in all groups, but MEP-treated specimens showed greater bond strength stability ( $\Delta \mu TBS \leq 2.0 MPa$ ). Sandblasted surfaces consistently exhibited higher  $\mu TBS$  than polished ones. HF+S and MEP performed similarly, except on composite resin before aging, where HF+S was superior.

**Conclusion:** MEP with airborne-particle abrasion showed  $\mu TBS$  values similar to HF+S and greater adhesive stability after TC. Additionally, MEP improved wettability, preserved  $\sigma f3P$  compared to HF, reduced  $\Delta Sa$ , and demonstrated consistent performance on dentin and composite resin.

**Key-words:** Self-etching ceramic primer. Monobond Etch & Primer. Hydrofluoric acid. Lithium disilicate. Glass dental-ceramics. Adhesion.

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## 1 INTRODUÇÃO GERAL

As cerâmicas odontológicas tornaram-se essenciais na odontologia restauradora contemporânea, impulsionadas pela busca por sorrisos mais harmônicos e por estruturas dentárias com aparência natural (MOURA et al., 2020). Além de atenderem à crescente demanda estética, essas reabilitações ganham destaque na prática clínica atual, favorecidas pelos avanços na ciência dos materiais e pela valorização de tratamentos minimamente invasivos e altamente estéticos. Devido à excelente estética, biocompatibilidade e propriedades mecânicas, esses materiais são amplamente utilizados em coroas, facetas e restaurações parciais (DE SIQUEIRA et al., 2019; LOPES et al., 2019; LYANN et al., 2018; MOURA et al., 2020; SOUZA et al., 2020). Entre as cerâmicas vítreas, as formuladas com dissilicato de lítio se destacam pela combinação superior de propriedades ópticas, mecânicas e adesivas, sendo indicadas para restaurações livres de metal, como inlays, onlays, facetas e coroas (AL-HARTHI et al., 2018).

Diversos estudos vêm sendo conduzidos com o objetivo de aperfeiçoar a durabilidade das restaurações cerâmicas, por meio de pré-tratamentos de superfície e da aplicação de agentes de união (MALYSA *et al.*, 2021). O avanço no conhecimento sobre as características das cerâmicas e dos sistemas adesivos ampliou as possibilidades clínicas, permitindo tratamentos mais personalizados, com melhor custo-benefício, menor tempo clínico e maior longevidade restauradora (OLIVEIRA *et al.*, 2023; THIESEN *et al.*, 2019).

Para as cerâmicas vítreas à base de dissilicato de lítio, o protocolo convencional consiste na aplicação de ácido fluorídrico (HF) seguida da silanização (HF+S). O HF atua dissolvendo a matriz vítrea, expondo a sílica para promover a ligação química (MURILLO-GÓMEZ; WANDERLEY; DE GOES, 2019; TURUNÇ OĞUZMAN; ŞİŞMANOĞLU, 2024). O silano, por sua vez, eleva a energia superficial, melhora a molhabilidade e favorece a adesão entre a cerâmica hidrofílica e as resinas hidrofóbicas (MURILLO-GÓMEZ; DE GOES, 2017; SUNDFELD NETO *et al.*, 2015; WILLE; LEHMANN; KERN, 2022). No entanto, o uso inadequado do HF pode comprometer a integridade microestrutural da cerâmica e afetar negativamente a adesão (AWAD *et al.*, 2022; COLOMBO; MURILLO-GÓMEZ; DE GOES, 2019; FONZAR *et al.*, 2020). Além disso, o uso do HF exige precauções rigorosas devido à sua toxicidade e ao risco de danos aos tecidos moles, sendo inclusive proibido em alguns países (SUNDFELD NETO *et al.*, 2015; VICHI *et al.*, 2021).

Estudos recentes indicam que superfícies excessivamente porosas geradas pelo ataque com HF nem sempre promovem melhor adesão, podendo inclusive prejudicar a molhabilidade,

conforme descrito pelo modelo de Cassie–Baxter (DEL BIANCO et al., 2024). Esse modelo explica que, ao entrar em contato com uma superfície rugosa que apresenta bolsões de ar aprisionados, o líquido repousa parcialmente sobre o ar em vez de aderir completamente ao sólido. Essa interface composta entre sólido e ar reduz a área de contato líquido–sólido, aumentando o ângulo de contato e, conseqüentemente, diminuindo a molhabilidade, mesmo diante de uma rugosidade superficial elevada (MARMUR, 2003; BHUSHAN; JUNG, 2011; CASSIE; BAXTER, 1944). Assim, a topografia irregular criada pelo HF, especialmente quando combinado ao silano (HF+S), pode reduzir paradoxalmente a molhabilidade devido à penetração incompleta do líquido nas microcavidades. Além disso, a combinação de técnicas agressivas, como o HF e o jateamento com óxido de alumínio, pode induzir defeitos estruturais na cerâmica e comprometer sua resistência à flexão (ALRAHLAH et al., 2017).

Como alternativa ao protocolo convencional, surgem os primers cerâmicos autocondicionantes (PCAs), como o Monobond Etch & Prime (MEP), que associam em um único frasco um agente de ataque ácido mais brando e o silano, simplificando o protocolo clínico e reduzindo os riscos relacionados ao manuseio de substâncias agressivas (WILLE; LEHMANN; KERN, 2022). O MEP (Ivoclar Vivadent), único PCA disponível comercialmente, realiza simultaneamente o condicionamento da superfície cerâmica e a silanização, além de remover contaminantes salivares (ROMÁN-RODRÍGUEZ et al., 2017; WILLE; LEHMANN; KERN, 2022). Sua composição inclui fluoreto de amônio como agente de condicionamento e metacrilato de silano como agente de união, sendo indicado para o tratamento de cerâmicas vítreas à base de dissilicato de lítio cimentadas com cimentos resinosos (ROMÁN-RODRÍGUEZ et al., 2017; WILLE; LEHMANN; KERN, 2022). Embora apresentem desempenho inferior em alguns parâmetros quando utilizados isoladamente, esses sistemas demonstram potencial, especialmente quando associados a técnicas como o jateamento com óxido de alumínio (ALMIRO et al., 2022).

Apesar de o MEP proporcionar um protocolo clínico mais simples, sua eficácia adesiva em médio e longo prazo ainda está em avaliação (DAPIEVE et al., 2021). Estudos laboratoriais têm apresentado resultados divergentes: alguns indicam que o ácido HF proporciona maiores valores de resistência de união (ALAYAD, 2023; ALKHUDHAIRY, 2018; ÇINAR et al., 2023; DIMITRIADI et al., 2020; DONMEZ; OKUTAN; YUCEL, 2020; EL-DAMANHOURY et al., 2020; EL-DAMANHOURY; GAINANTZOPOULOU, 2018; GUIMARÃES et al., 2018; LOPES et al., 2019; MOUSLY et al., 2020; PRADO et al., 2018; SOUZA et al., 2020), enquanto outros apontam desempenho semelhante entre o MEP e o protocolo convencional com HF+S (ALKHUDHAIRY et al., 2019; ALRAHLAH et al., 2017; ALSHIHRI, 2019;

CARDENAS *et al.*, 2019; COLOMBO; MURILLO-GÓMEZ; DE GOES, 2019; DAPIEVE *et al.*, 2023; DE SIQUEIRA *et al.*, 2019; KLIPPEL *et al.*, 2021; LIEBERMANN; DETZER; STAWARCZYK, 2019; LU *et al.*, 2024; LYANN *et al.*, 2018; MAIER *et al.*, 2019; MURILLO-GÓMEZ; DE GOES, 2019; ROMÁN-RODRÍGUEZ *et al.*, 2017; SIQUEIRA *et al.*, 2016; TRIBST *et al.*, 2018; TURUNÇ OĞUZMAN; ŞİŞMANOĞLU, 2024; VICHİ *et al.*, 2021; VILA-NOVA *et al.*, 2022; WILLE; LEHMANN; KERN, 2017). Há ainda relatos que indicam desempenho superior do MEP ao método tradicional em determinadas condições (LEVARTOVSKY *et al.*, 2021; TURUNÇ-OĞUZMAN; ŞİŞMANOĞLU, 2023). Embora muitos trabalhos tenham investigado os protocolos de adesão em cerâmicas, ainda existem incertezas quanto à efetividade dos PCAs.

Diante desse contexto, a escolha do protocolo ideal de tratamento de superfície deve levar em conta não apenas os efeitos imediatos sobre a adesão, mas também sua durabilidade frente ao envelhecimento hidrotérmico e às tensões funcionais (DIMITRIADI *et al.*, 2020; KOMOTO; MASEKI; NARA, 2021). A constante evolução dos sistemas adesivos e dos métodos de preparo superficial amplia as possibilidades clínicas e contribui para tratamentos restauradores mais personalizados, seguros e duradouros (OLIVEIRA *et al.*, 2023; THIESEN *et al.*, 2019).

## **2 OBJETIVOS**

### **2.1 Geral**

Avaliar o efeito do tratamento com o primer cerâmico autocondicionante Monobond Etch & Prime (MEP) nas propriedades de superfície, mecânicas e adesivas da cerâmica à base de dissilicato de lítio (DL) obtida por sistema CAD/CAM e cimentada sobre diferentes substratos, em comparação ao protocolo convencional com ácido hidrofluorídrico e silano (HF+S).

### **2.2 Específicos**

- Caracterizar a superfície da cerâmica à base de DL submetida a diferentes agentes de condicionamento (HF+S e MEP) e texturas superficiais (polida e jateada com óxido de alumínio), por meio da análise de rugosidade de superfície, ângulo de contato e microscopia eletrônica de varredura.
- Avaliar a resistência à fratura da cerâmica à base de DL por meio do teste de flexão em três pontos, em função do tipo de agente de condicionamento e textura superficial.
- Avaliar a resistência adesiva da cerâmica aos diferentes substratos (dentina e resina composta), por meio do teste de microtração, considerando o tratamento de superfície e a termociclagem.

### 3 HIPÓTESES

#### 3.1 Propriedades de superfície e mecânicas

- **H<sub>0</sub> (Hipótese nula):** O tratamento com Monobond Etch & Prime (MEP), com ou sem jateamento prévio com óxido de alumínio, não promove diferenças significativas nas propriedades de superfície (rugosidade, molhabilidade) e na resistência à flexão da cerâmica de dissilicato de lítio (DL) em comparação ao protocolo convencional com ácido hidrofluorídrico e silano (HF+S).
- **H<sub>1</sub> (Hipótese alternativa):** O tratamento com MEP, com ou sem jateamento prévio com óxido de alumínio, promove diferenças significativas nas propriedades de superfície (rugosidade, molhabilidade) e/ou na resistência à flexão da cerâmica de DL em comparação ao protocolo convencional com HF+S.

#### 3.2 Propriedades adesivas – substrato dentina

- **H<sub>0</sub> (Hipótese nula):** O tipo de tratamento de superfície da cerâmica (MEP jateado, MEP polido, HF+S jateado e HF+S polido) e a realização ou não da termociclagem não influenciam significativamente a resistência adesiva da cerâmica de DL à dentina.
- **H<sub>1</sub> (Hipótese alternativa):** O tipo de tratamento de superfície da cerâmica (MEP jateado, MEP polido, HF+S jateado e HF+S polido) e/ou a realização da termociclagem influenciam significativamente a resistência adesiva da cerâmica de DL à dentina.

#### 3.3 Propriedades adesivas – substrato resina composta

- **H<sub>0</sub> (Hipótese nula):** O tipo de tratamento de superfície da cerâmica (MEP jateado, MEP polido, HF+S jateado e HF+S polido) e a realização ou não da termociclagem não influenciam significativamente a resistência adesiva da cerâmica de DL à resina composta.
- **H<sub>1</sub> (Hipótese alternativa):** O tipo de tratamento de superfície da cerâmica (MEP jateado, MEP polido, HF+S jateado e HF+S polido) e/ou a realização da termociclagem influenciam significativamente a resistência adesiva da cerâmica de DL à resina composta.



#### 4 CAPÍTULO

Esta dissertação está baseada no Artigo 46 do Regimento Interno do Programa de Pós-graduação em Odontologia da Universidade Federal do Ceará, que regulamenta o formato alternativo para dissertações de Mestrado e teses de Doutorado, e permite a inserção de artigos científicos de autoria ou coautoria do candidato. Por se tratar de pesquisa envolvendo seres humanos, o projeto de pesquisa deste trabalho foi submetido à apreciação do Comitê de Ética em Pesquisa da Universidade Federal do Ceará, com o protocolo **#7.574.147**. Assim sendo, esta dissertação é composta de um capítulo que contém um artigo científico que será submetido para publicação no periódico **“The International Journal of Adhesion and Adhesives”** (Anexo A), sob o título **“Effect of a self-etching ceramic primer on the surface, mechanical, and adhesive properties of lithium disilicate-based glass-ceramics bonded to different substrates”**.

**Effect of a self-etching ceramic primer on the surface, mechanical, and adhesive properties of lithium disilicate-based glass-ceramics bonded to different substrates**

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## Effect of a self-etching ceramic primer on the surface, mechanical, and adhesive properties of lithium disilicate-based glass-ceramics bonded to different substrates

### Abstract

This study evaluated the effect of a self-etching ceramic primer (Monobond Etch & Prime – MEP), with or without aluminum oxide air abrasion (sandblasting), on the surface, mechanical, and adhesive properties of lithium disilicate-based glass-ceramics (LD), compared to conventional hydrofluoric acid and silane treatment (HF+S). LD specimens were prepared in two formats: square-shaped (10×10×4 mm, N=134) for surface roughness ( $\Delta Sa$ ), contact angle (CA), microtensile bond strength ( $\mu TBS$ ), and scanning electron microscopy (SEM); and rectangular-shaped (15×2.5×1.5 mm, N=48) for three-point flexural strength ( $\sigma_{f3P}$ ). Four groups were tested according to surface treatment: HF+S sandblasted (HF+S[s]), HF+S polished (HF+S[p]), MEP sandblasted (MEP[s]), and MEP polished (MEP[p]). After treatment,  $\Delta Sa$ , CA, and  $\sigma_{f3P}$  were measured. LD blocks were cemented to either composite resin or dentin to evaluate  $\mu TBS$  before and after thermocycling (TC, 10,000 cycles, 5 °C/55 °C, 30 s dwell time). Sandblasting significantly increased  $\Delta Sa$  (HF+S[s]:  $1.16 \pm 0.12 \mu m$ ; MEP[s]:  $0.85 \pm 0.02 \mu m$ ) compared to polished specimens (HF+S[p]:  $0.47 \pm 0.04 \mu m$ ; MEP[p]:  $0.38 \pm 0.04 \mu m$ ). The lowest CA was observed in the MEP[s] group ( $27.50 \pm 4.09^\circ$ ). Both HF+S-treated groups (HF+S[s] and HF+S[p]) showed the lowest  $\sigma_{f3P}$  values. TC reduced  $\mu TBS$  in all groups, but MEP-treated specimens (MEP[s] and MEP[p]) showed greater bond strength stability ( $\Delta \mu TBS \leq 2.0 MPa$ ). Sandblasted surfaces consistently exhibited higher  $\mu TBS$  than polished ones. HF+S and MEP performed similarly, except on composite resin before TC, where HF+S was superior. In conclusion, MEP[s] demonstrated  $\mu TBS$  values comparable to HF+S and provided greater adhesive stability after TC. Furthermore, MEP enhanced wettability, better preserved  $\sigma_{f3P}$  compared to HF, reduced  $\Delta Sa$ , and showed consistent performance on both dentin and composite resin, supporting its effectiveness as a reliable surface treatment for lithium disilicate ceramics.

**Key-words:** Self-etching ceramic primer. Monobond Etch & Primer. Hydrofluoric acid. Lithium disilicate. Glass dental-ceramics. Adhesion.

### 1. Introduction

Dental ceramics are essential in modern restorative dentistry, driven by the demand for smile enhancement and natural-looking dental structures [1]. Their superior esthetics,

biocompatibility, and mechanical properties make them ideal for crowns, veneers, and partial restorations [1–5].

Extensive research has aimed to optimize the long-term success of ceramic restorations through surface pretreatment and bonding agent application [6]. Advances in understanding ceramic properties and adhesive systems have expanded clinical options, enabling tailored treatments that improve cost-effectiveness, reduce chair time, and enhance restoration longevity [7,8].

The standard protocol for lithium disilicate-based glass-ceramics involves hydrofluoric acid etching followed by silane (HF+S) application. HF dissolves the glassy matrix, exposing silica for chemical bonding [9,10]. Silane increases surface energy, improves wettability, and promotes adhesion between hydrophilic ceramics and hydrophobic resins [11–13]. However, improper HF use can cause microstructural damage, reduce microhardness, and impair bonding [14–16]. Additionally, due to its high toxicity and risk of chemical burns, HF use demands caution and is restricted in some countries [13,17].

Self-etching ceramic primers (SeCPs) have emerged as potential alternatives to the conventional HF+S protocol by simplifying clinical procedures and reducing technique sensitivity. Among them, Monobond Etch & Prime (MEP, Ivoclar Vivadent) is the only commercially available product, combining etching and silanization in a single step while also removing salivary contaminants [12,18]. MEP contains ammonium polyfluoride as the etching agent and silane methacrylate as the coupling agent, making it suitable for lithium disilicate-based glass-ceramics bonded with resin cements [12,18].

Although Monobond Etch & Prime (MEP) offers a simplified clinical protocol, its medium- and long-term effectiveness in ceramic bonding remains under investigation [19]. In vitro studies have shown conflicting results: some indicate that HF acid achieves higher bond strengths [3,5,20–29], while others report similar results between MEP and the conventional HF+S protocol [2,4,9,15,17,18,30–43]. Additionally, some studies have even suggested that MEP may outperform the traditional method under specific conditions [44,45]. Although many studies have investigated ceramic bonding protocols, there is still uncertainty regarding the effectiveness of SeCP. While numerous studies have explored ceramic bonding protocols, uncertainties persist regarding the overall effectiveness of self-etching ceramic primers (SeCPs). Although MEP simplifies the clinical procedure, its long-term performance, particularly under varied clinical conditions, warrants further investigation. Additionally, while many studies focus on individual aspects of bonding, such as bond strength or surface treatments, fewer examine the combined effect of sandblasting and conditioning agents across

substrates like composite resin and dentin, which are commonly encountered in clinical practice, where composite resins are used for core build-ups and dentin remains a difficult bonding surface.

Therefore, this study aimed to evaluate the impact of SeCP on the surface, mechanical, and adhesive properties of lithium disilicate-based glass-ceramics, with or without sandblasting, on composite resin and dentin substrates, using prolonged use simulations. The hypotheses tested were that SeCP, with or without sandblasting, would: (1) improve the mechanical and surface properties of lithium disilicate ceramics; (2) enhance  $\mu$ TBS for composite resin substrates both before and after thermocycling (TC); and (3) improve  $\mu$ TBS for dentin substrates before and after TC, with SeCP performing similarly to or better than HF.

## 2. Materials and methods

### 2.1. Experimental design

The study design followed the guidelines of the modified CONSORT for in vitro studies on dental materials [46]. This blinded in vitro study was conducted with four parallel experimental groups, categorized based on the surface texture of the lithium disilicate-based glass-ceramic (sandblasted or polished as control) and the conditioning agent (HF+S or MEP). The groups were as follows: HF+S sandblasted (HF+S[s]), HF+S polished (HF+S[p]), MEP sandblasted (MEP[s]), and MEP polished (MEP[p]). Detailed information on material composition and treatment protocols is provided in Table 1. Both the examiner responsible for the analyses and the statistician were blinded to group allocation.

The evaluated outcomes included surface roughness ( $S_a$ ,  $\mu\text{m}$ ), contact angle (CA, in degrees), three-point flexural strength ( $\sigma_{f3P}$ , MPa), microtensile bond strength ( $\mu$ TBS, MPa), and failure modes. The  $\mu$ TBS test was conducted on dentin and composite resin substrates, following TC, to simulate different clinical scenarios of metal-free restorations. The study protocol, as well as the informed consent for the use of extracted human teeth, was reviewed and approved by the Research Ethics Committee involving human participants (approval number #7.574.147).

### 2.2. Sample size estimation

A preliminary pilot study was carried out to calculate the appropriate sample size, using  $\mu$ TBS as the primary outcome variable. The experimental design included two independent factors, with time considered as a repeated factor (within-subject) and group as a between-subject factor, requiring a multifactorial repeated-measures ANOVA (F-tests). The calculation

was based on the following parameters: a medium effect size ( $f = 0.41$ ), significance level ( $\alpha$ ) of 0.05, statistical power ( $1-\beta$ ) of 0.80, four groups, and three degrees of freedom. Under these conditions, the analysis indicated that a minimum of eight specimens per group would be sufficient, totaling 32 samples. The sample size estimation was performed using the G\*Power software, version 3.1.9.7 (Heinrich Heine University, Düsseldorf, Germany).

### *2.3. Preparation and surface treatment of lithium disilicate-based glass-ceramic specimens*

Blocks of lithium disilicate-based glass-ceramic (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were sectioned to obtain two types of specimens: square-shaped ( $10 \times 10 \times 4$  mm,  $N = 134$ ), used for Sa, CA,  $\mu$ TBS, and scanning electron microscopy (SEM) analyses; and rectangular-shaped ( $15 \times 2.5 \times 1.5$  mm,  $N = 48$ ), used for  $\sigma_f^{3P}$ . Sectioning was performed using a low-speed precision cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) equipped with a diamond wafering blade under continuous distilled water irrigation. All specimens were subsequently crystallized in a ceramic furnace (Alumini Sinter Press, EDG Equipamentos, São Carlos, SP, Brazil) following the manufacturer's recommended protocol. Surface polishing was carried out by a single trained operator using a manual polishing unit with a multi-specimen holder (Arotec, Cotia, SP, Brazil). Each specimen was polished for 30 seconds using silicon carbide abrasive papers (Solventum, St. Paul, MN, USA) in 400- and 600-grit sequences under water irrigation [7]. After polishing, specimens were ultrasonically cleaned in distilled water for 10 minutes.

Surface treatments were performed by a second researcher (G. F. P.) to ensure blinding of the primary investigator. Specimens were divided into two main groups ( $n = 91$ ) based on surface texture: polished (control) or sandblasted with  $50 \mu\text{m}$  aluminum oxide particles ( $\text{Al}_2\text{O}_3$ , Bio-Art, São Carlos, SP, Brazil) for 10 s at 2.5 bar pressure and 10 mm distance. Each group was then subdivided ( $n = 41$ ) according to the conditioning protocol: HF+S or MEP.

In the HF+S groups, 10% HF acid (Condac Porcelana, FGM, Joinville, SC, Brazil) was applied for 20 s, followed by rinsing and air drying for 30 s. Then, Monobond N (Ivoclar Vivadent, Schaan, Liechtenstein) was applied for 60 s and air-dried. In the MEP groups, MEP (Ivoclar Vivadent, Schaan, Liechtenstein) was rubbed onto the surface with a microbrush for 20 s, left undisturbed for 40 s, and then rinsed and dried for 30 s. All treatments were performed under standardized conditions.

Table 1. Types, materials (manufacturers), main compositions, and batch numbers of the ceramic and its respective surface treatment materials used in this study.

Type	Material (Manufacturer)	Main composition	Batch #
Lithium disilicate-based glass-ceramic	IPS e.max CAD (Ivoclar Vivadent)	(wt%): SiO <sub>2</sub> (57-80%), Li <sub>2</sub> O (11-19%), K <sub>2</sub> O (0-13%), P <sub>2</sub> O <sub>5</sub> (0–11%), ZrO <sub>2</sub> (0–8%), ZnO (0–8%), Al <sub>2</sub> O <sub>3</sub> (0–5%) MgO (0–5%), coloring oxides (0–8%)	YB93XH
Self-etching ceramic primer	Monobond Etch & Prime (Ivoclar Vivadent)	(wt%): TDT (≤10%), trimethoxypropyl methacrylate (1-<2.5%, silane), methacrylate phosphoric ester (3-<10%), butanol and water (75-85%), food coloring (<1%, fast Green)	Z02K70
10% hydrofluoric acid	Condac Porcelain (FGM)	(wt%): Hydrofluoric acid (10%), water (75-85%), thickener, surfactant, and coloring (5-15%)	270324
Silane	Monobond N (Ivoclar Vivadent)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Z07R1B
Etch-and-rinse resin cement	RelyX ARC (Solventum)	(wt%): Inorganic phase – ZrO <sub>2</sub> + SiO <sub>2</sub> (67.5%, average particle size: 1.5 µm); Organic phase – BIS-GMA, TEGDMA, tertiary amine, benzoyl peroxide, pigments (32.5%)	2308600321

Abreviações: TDT, Tetrabutylammonium dihydrogen trifluoride. BIS-GMA, bisfenol A-glicidil metacrilato. TEGDMA, dimetacrilato de trietilenglicol.

## 2.4. Surface and mechanical characterization of lithium disilicate-based glass-ceramic

### 2.4.1. Laser Confocal Microscopy (Surface roughness [Sa])

The Sa was measured in 32 specimens ( $n = 8$ ), both before and after surface treatments, to determine roughness change ( $\Delta Sa$ ) for each sample. The analysis was conducted by a third researcher (B. S. H. T.). The Sa was evaluated using a laser confocal microscope (OLS5000, Olympus, Tokyo, Japan) with a 10x magnification. The Sa parameter represents the arithmetic mean of the surface height deviations relative to a baseline and is a 3D evaluation method that considers an area of the surface.

### 2.4.2. Contact angle (CA)

The CA was evaluated on 32 square-shaped ceramic specimens ( $n = 8$ ) using the sessile drop method [47]. Prior to testing, specimens were stored dry in individual plastic tubes. Measurements were performed using a goniometer (Digidrop, GBX Scientific, Bourg-de-Péage, Drôme, France) equipped with analysis software (Visiodrop, version 13.05.2.10, GBX Scientific, Bourg-de-Péage, Drôme, France) and a camera (Pixelink, Nikon, Sendai, Japan). Each specimen was fixed on an adjustable stage with double-sided tape and positioned directly beneath a vertically oriented 21-gauge needle (outer diameter  $\approx 0.815$  mm), connected to a 1 mL syringe. To ensure consistency in the volume of adhesive applied, a calibrated rotation-controlled dispensing system, coupled to the goniometer, was used to hold the syringe. Manual rotation of the device allowed the release of a single drop of adhesive – approximately 0.05 mL – uniformly onto the dentin surface of all samples. This method guaranteed standardization of both drop size and application conditions among the groups. CA measurements were recorded 5 seconds after drop deposition via a high-resolution camera integrated into the goniometer system. The CA was calculated as the mean of the left and right angles, automatically determined by the analysis software.

### 2.4.3. Three-point flexural strength ( $\sigma_f^{3P}$ )

The  $\sigma_f^{3P}$  was evaluated in 48 rectangular-shaped ceramic specimens ( $n = 8$ ) using a universal testing machine (model Instron, Instron Corporation, Norwood, MA, USA) equipped with a 500 N load cell and operated via Bluehill Universal software (Instron Corporation, Norwood, MA, USA). Six experimental groups were tested: polished and sandblasted specimens treated with HF+S or MEP, as well as two additional control groups composed of specimens subjected only to polishing or only to sandblasting, without further surface



conditioning. The test followed the ISO 6872:2015 guidelines for  $\sigma_f^{3P}$  of dental ceramics, with modifications to the specimen dimensions and support span. Due to the size limitations of the lithium disilicate blocks, the specimens were fabricated with dimensions of  $15 \times 2.5 \times 1.5$  mm and tested with a support span of 12 mm, maintaining proportionality to the ISO standard recommendations.

Each specimen was positioned with its center aligned under a metallic loading piston, and the force was applied at a crosshead speed of 1.0 mm/min until failure. The maximum load at fracture was recorded in Newtons (N) and used to calculate the flexural strength.

#### 2.4.4. Scanning electron microscopy (SEM)

Square-shaped ceramic specimens ( $n = 1$ ) from six groups (four experimental groups: HF+S[p], HF+S[s], MEP[p], MEP[s], and two additional control groups with specimens that were either only polished or only airborne-particle sandblasted) were affixed to aluminum stubs using carbon adhesive tape and coated with a thin layer of gold using a sputter coater (SCD 050, Bal-tec, Balzers, Liechtenstein) to ensure adequate electrical conductivity. Surface characterization was then performed using a SEM (SS550, Shimadzu Corp., Kyoto, Japan) under high vacuum conditions. Representative images were captured at  $10,000\times$  magnifications to analyze the surface texture and microstructural features.

#### 2.5. Microtensile bond strength ( $\mu$ TBS)

Thirty-two human molars were sectioned using a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, IL, USA) to obtain standardized coronal dentin blocks ( $10 \times 10 \times 4$  mm). The surfaces were flattened and polished with silicon carbide papers (#200, #400, and #600) under water cooling, each for 30 seconds, to ensure uniform surface roughness and smear layer formation [48]. Composite resin specimens simulating core build-ups were fabricated using the same dimensions and mold ( $10 \times 10 \times 4$  mm). The composite resin (Opallis DA3.5, FGM Produtos Odontológicos, Joinville, SC, Brazil) was incrementally inserted and light-cured for 40 seconds per layer with an LED unit (Valo, 1200 mW/cm<sup>2</sup>, Ultradent, South Jordan, UT, USA), following the manufacturer's instructions. Composite resin blocks were polished using the same abrasive sequence to standardize surface conditions. Dentin and composite resin specimens were randomly allocated into four experimental groups. All procedures were performed by a single operator (M. I. A. T.), blinded to the ceramic surface treatments to ensure methodological consistency.

Dentin blocks were etched with 37% phosphoric acid gel (Ultradent, South Jordan, UT, USA) for 15 seconds, rinsed for 30 seconds, and gently dried with cotton pellets. Two coats of adhesive (Adper Single Bond 2, Solventum, St. Paul, MN, USA) were actively applied for 20 seconds each, with a 5-second air-drying interval, followed by light-curing for 40 seconds using a Valo LED unit (Ultradent, South Jordan, UT, USA). Composite resin blocks underwent a similar protocol: etched with 37% phosphoric acid for 40 seconds, rinsed and dried for 20 seconds, followed by the application of two coats of silane (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein), then the same adhesive and light-curing steps used for dentin.

Ceramic specimens were bonded to dentin and composite resin blocks using an etch-and-rinse resin cement (RelyX™ ARC, Solventum, St. Paul, MN, USA). Light activation was applied to all surfaces for a total of 150 seconds using a Valo LED unit (Ultradent, South Jordan, UT, USA). The irradiance was checked using a radiometer (Light Meter, Delta OHM, Padova, Veneto, Italy) immediately before initiating light-curing for each experimental group to ensure consistent light output throughout the specimen preparation. The assembled specimens (dentin/composite resin – resin cement – ceramic) were stored in distilled water at 37°C for 24 hours.

Each specimen was then sectioned longitudinally; one half was immediately prepared for  $\mu$ TBS testing, while the other half underwent TC (OMC 250L, Odeme Dental Research, Luzerna, SC, Brazil) for 10,000 cycles, with 30-second immersions at 5°C and 55°C to simulate one year of clinical use [49]. After TC, the remaining halves were also prepared for delayed  $\mu$ TBS testing. Specimens were sectioned into beams (microtensile sticks) with a cross-sectional area of approximately  $1.0 \pm 0.1 \text{ mm}^2$  for testing for each specimen, the mean  $\mu$ TBS value of all sticks obtained under the same condition was calculated and considered as a single statistical unit (experimental unit) in the analysis.

The  $\mu$ TBS test was performed using a Universal Testing Machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil) with a 100 Kgf load cell, at a crosshead speed of 0.5 mm/min. The bond failure force was recorded in Kgf, and  $\mu$ TBS values were calculated in MPa.

## 2.6. Failure modes

The failure modes of specimens tested for  $\mu$ TBS were examined using a digital optical microscope (S8APO Leica, Leica Microsystems, Wetzlar, Germany) and categorized into six types: (1) adhesive failure between cement and ceramic (ADH-C); (2) mixed failure involving both adhesive and cohesive failure within the ceramic (MIX-C); (3) adhesive failure between cement and substrate (ADH-S); (4) mixed failure involving both adhesive and cohesive failure

within the substrate (MIX-S); (5) cohesive failure within the ceramic (COH-C); and (6) cohesive failure within the substrate (COH-S).

## 2.7. Statistical Analysis

The Shapiro-Wilk test was applied to assess data distribution, confirming normality for all variables. Sa and CA were analyzed using one-way ANOVA, as they involved a single independent factor.  $\sigma_f^{3P}$  and  $\mu\text{TBS}$ , which included two factors of variation, a two-way repeated-measures ANOVA and two-way mixed ANOVA were performed, respectively. Tukey's post hoc test was applied for all multiple comparisons. Statistical analyses were conducted using IBM SPSS Statistics (version 20.0; IBM Corp., Armonk, NY, USA), with the significance level set at 5% ( $\alpha < 0.05$ ).

## 3. Results

### 3.1. Surface and mechanical properties of lithium disilicate-based glass-ceramic

Table 2 presents the data for Sa and CA. One-way ANOVA revealed statistically significant differences among the groups for Sa ( $F_{(3)} = 13.235$ ;  $p < 0.001$ ) and CA ( $F_{(3)} = 43.100$ ;  $p < 0.001$ ). Table 3 displays the  $\sigma_f^{3P}$ . A two-way repeated-measures ANOVA indicated significant effects for surface texture ( $F_{(1)} = 96.0$ ;  $p < 0.001$ ), conditioning agent ( $F_{(2)} = 23.2$ ;  $p < 0.001$ ), and their interaction ( $F_{(2)} = 13.9$ ;  $p < 0.001$ ).

Sandblasting significantly increased Sa compared with polished condition ( $p < 0.05$ ), regardless of the conditioning agent. In the polished state, HF+S[p] ( $0.47 \pm 0.04 \mu\text{m}$ ) and MEP[p] ( $0.38 \pm 0.04 \mu\text{m}$ ) exhibited statistically similar Sa values ( $p > 0.05$ ). Among the sandblasted groups, HF+S[s] specimens ( $1.16 \pm 0.12 \mu\text{m}$ ) showed significantly higher Sa values than MEP[s] specimens ( $0.85 \pm 0.02 \mu\text{m}$ ) ( $p < 0.05$ ).

Specimens subjected to sandblasting exhibited significantly lower CA values than their polished counterparts, indicating enhanced surface wettability after surface abrasion ( $p < 0.05$ ). Among all groups, MEP[s] showed the lowest CA ( $27.50 \pm 4.09^\circ$ ), which differed significantly from all others ( $p < 0.05$ ). In contrast, HF+S[p] displayed the highest CA ( $47.84 \pm 2.91^\circ$ ), also significantly different from the remaining groups ( $p < 0.05$ ). HF+S[s] ( $35.76 \pm 4.63^\circ$ ) and MEP[p] ( $37.76 \pm 5.45^\circ$ ) exhibited statistically similar CA values ( $p > 0.05$ ).

Table 2. Mean and standard deviation of surface roughness change ( $\Delta Sa$ ,  $\mu m$ ) and contact angle (CA, degrees) of lithium disilicate ceramic specimens, according to the surface texture (airborne-particle sandblasted and polished) and the conditioning agent (HF+S and MEP).

Group	$\Delta Sa$	CA
HF+S polished	$0.47 \pm 0.04$ C	$47.84 \pm 2.91$ C
MEP polished	$0.38 \pm 0.04$ C	$37.76 \pm 5.45$ B
HF+S sandblasted	$1.16 \pm 0.12$ A	$35.76 \pm 4.63$ B
MEP sandblasted	$0.85 \pm 0.02$ B	$27.50 \pm 4.09$ A
$p^a$	<0.001*	<0.001*

HF, hydrofluoric acid. S, silane. MEP, monobond etch & primer. <sup>a</sup>ANOVA/Tukey's test. Different uppercase letters within columns indicate statistically significant differences.

The  $\sigma_{f3P}$  results showed that sandblasting significantly decreased fracture resistance across all groups (control, HF+S[s], and MEP[s]) ( $p < 0.05$ ). Notably, MEP treatment maintained  $\sigma_{f3P}$  at similar levels on both polished and sandblasted surfaces. In contrast, HF+S produced the lowest  $\sigma_{f3P}$  values for both polished ( $237.04 \pm 9.95$  MPa) and sandblasted ( $201.36 \pm 16.88$  MPa) conditions.

Table 3. Mean and standard deviation of three-point flexural strength ( $\sigma_{f3P}$ , MPa) of lithium disilicate ceramic specimens, according to the surface texture (airborne-particle sandblasted and polished) and the conditioning agent (HF+S and MEP).

Conditioning agent	Surface texture		$p^a$
	Polished	Sandblasted	
Control	$263.51 \pm 10.26$ Aa	$238.84 \pm 13.51$ Ab	<0.001*
HF+S	$237.04 \pm 9.95$ Ba	$201.36 \pm 16.88$ Bb	<0.001*
MEP	$253.90 \pm 9.00$ Aa	$223.24 \pm 14.78$ Ab	<0.001*
$p^b$	0,023*	0,012*	

HF, hydrofluoric acid. S, silane. MEP, monobond etch & primer. <sup>a</sup>Student T test. <sup>b</sup>ANOVA/Tukey's test. Different uppercase (within columns) and lowercase (within lines) letters indicate statistically significant differences.

SEM images of treated surfaces are shown in Figure 1. Sandblasting induced significant surface modifications (Figure 1.A2), with HF treatment further enhancing these changes (Figure 1.B2). HF not only altered the surface topography but also increased micro-retention

formation on sandblasted surfaces, creating larger micro-retentions (yellow arrows, Figure 1.B2) compared to polished surfaces (Figure 1.B1). In contrast, MEP treatment caused more subtle surface alterations (Figure 1.C1 and 1.C2).

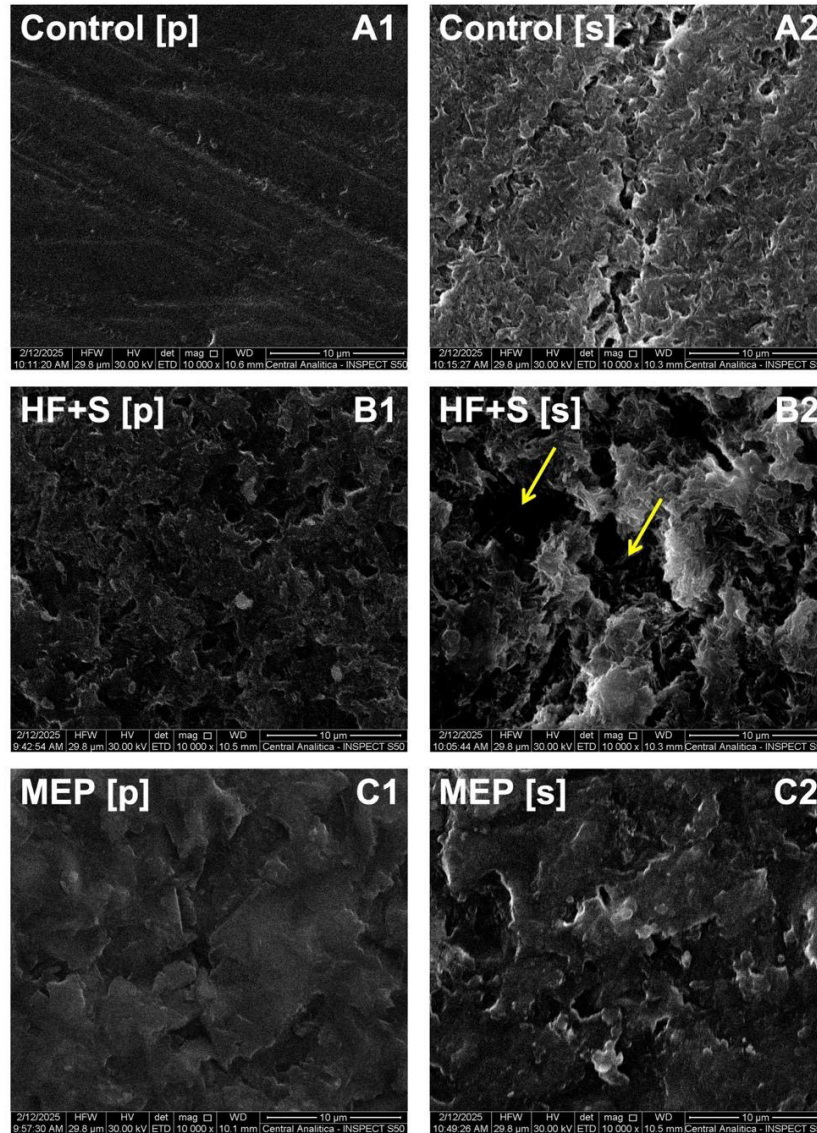


Figure 1. Scanning electron microscopy (SEM) images of lithium disilicate-based glass-ceramic surfaces after different surface treatments, captured at 10,000× magnification. Images in (1) show polished surfaces, while those in (2) show sandblasted surfaces. (A) represents untreated controls, (B) surfaces treated with hydrofluoric acid, and (C) surfaces treated with a self-etching ceramic primer (MEP).

### 3.2. $\mu$ TBS

The results of the 2-way mixed ANOVA, examining the effects of surface treatment, time, and their interaction on composite resin and dentin substrates, are presented in Table 4.

The  $\mu$ TBS (MPa) values for both substrates are shown in Table 5. TC significantly reduced the  $\mu$ TBS across all groups and substrates (before vs. after,  $p < 0.05$ ). Despite the TC effect, sandblasted surfaces consistently exhibited higher  $\mu$ TBS than polished ones ( $p < 0.05$ ), regardless of the conditioning agent (HF+S or MEP) or substrate (composite resin or dentin). HF+S and MEP treatments resulted in statistically similar  $\mu$ TBS values ( $p > 0.05$ ) for both polished and sandblasted specimens, independent of the substrate, except for composite resin before TC, where HF+S[s] specimens ( $13.99 \pm 3.11$  MPa) exhibited higher  $\mu$ TBS than MEP[s] specimens ( $10.94 \pm 2.51$  MPa). The greatest reductions in  $\mu$ TBS ( $\Delta\mu$ TBS) were observed in the HF+S[p] groups (Composite:  $4.67 \pm 1.34$  MPa; Dentin:  $3.03 \pm 0.94$  MPa) and HF+S[s] groups (Composite:  $4.45 \pm 1.65$  MPa; Dentin:  $3.27 \pm 1.08$  MPa).

Table 4. Two-way mixed ANOVA of microtensile bond strength ( $\mu$ TBS, MPa) for the composite resin and dentin substrates.

Substrate	Source	Sun of squares	df	Mean square	F	p
Composite resin	Surface treatment (ST)	370.166	3	123.389	13.339	<0.001*
	Time (T)	27.125	1	27.125	410.290	<0.001*
	ST * T	85.943	3	28.648	42.413	<0.001*
Dentin	Surface treatment (ST)	429.527	3	143.176	11.307	<0.001*
	Time (T)	103.327	1	103.327	320.110	<0.001*
	ST * T	6.083	3	2.028	6.282	0.002*

\* Statistically significant difference.

Table 5. Mean and standard deviation of microtensile bond strength ( $\mu$ TBS, MPa) of lithium disilicate ceramic specimens, according to the surface treatment and thermocycling time.

Substrate	Thermocycling		p <sup>a</sup>	ΔμTSB
	Before	After		
Composite resin				
HF+S sandblasted	13.99 ± 3.11 Aa	9.53 ± 2.43 Ab	<0.001*	-4.45 ± 1.65 B
MEP sandblasted	10.94 ± 2.51 Ba	9.29 ± 2.92 Ab	0.002*	-1.65 ± 0.94 A
HF+S polished	11.79 ± 1.39 Ba	7.12 ± 2.53 Bb	<0.001*	-4.67 ± 1.34 B
MEP polished	7.53 ± 1.21 Ca	6.53 ± 1.52 Bb	0.003*	-1.00 ± 0.64 A
p <sup>b</sup>	<0.001*	0.001*		<0.001*

Dentin				
HF+S sandblasted	12.84 ± 3.72 Aa	9.57 ± 3.37 Ab	<0.001*	-3.27 ± 1.08 C
MEP sandblasted	11.79 ± 2.30 Aa	9.75 ± 2.52 Ab	<0.001*	-2.03 ± 0.53 AB
HF+S polished	7.57 ± 2.37 Ba	4.54 ± 1.54 Bb	<0.001*	-3.03 ± 0.94 BC
MEP polished	6.51 ± 1.89 Ba	4.68 ± 1.89 Bb	<0.001*	-1.83 ± 0.50 A
p <sup>b</sup>	<0.001*	<0.001*		0.002*

HF, hydrofluoric acid. S, silane. MEP, monobond etch & primer. <sup>a</sup>Paired T test. <sup>b</sup>ANOVA/Tukey's test. Different uppercase (within columns) and lowercase (within lines) letters indicate statistically significant differences.

### 3.3. Failure modes

The distribution of failure modes is shown in Figure 2. For composite resin substrates, failures occurred predominantly on the ceramic side, mainly as ADH-C or MIX-C. In contrast, dentin substrates exhibited a higher incidence of failures on the substrate side, particularly after TC (ADH-S or MIX-S).

For composite resin substrates, before TC, sandblasted ceramics demonstrated a higher proportion of MIX-C failures (HF+S[s], 62.5%; MEP[s], 50.0%), whereas polished ceramics showed predominantly MIX-C failures for HF+S[p] (37.5%) and ADH-C failures for MEP[p] (62.5%). After TC, ADH-C failures became predominant across all composite resin groups, exceeding 75.0% in polished specimens and approaching 50.0% in sandblasted specimens, accompanied by a marked reduction in mixed failures. Cohesive failures (COH-C and COH-S) were rarely observed.

For dentin substrates, polished ceramics also exhibited a considerable proportion of failures on the ceramic side before TC, primarily MIX-C in HF+S[p] (50.0%) and ADH-C in MEP[p] (50.0%). Conversely, sandblasted ceramics showed fewer failures on the ceramic side (ADH-C, MIX-C, and COH-C), with most failures occurring on the dentin side: MIX-S (37.5%) or COH-S (37.5%) in HF+S[s] and MIX-S (37.5%) or ADH-S (25.0%) in MEP[s]. After TC, the failure pattern shifted: polished ceramics continued to show predominantly ADH-C failures (HF+S[p], 37.5%; MEP[p], 50.0%), whereas sandblasted groups exhibited a predominance of failures on the dentin side.

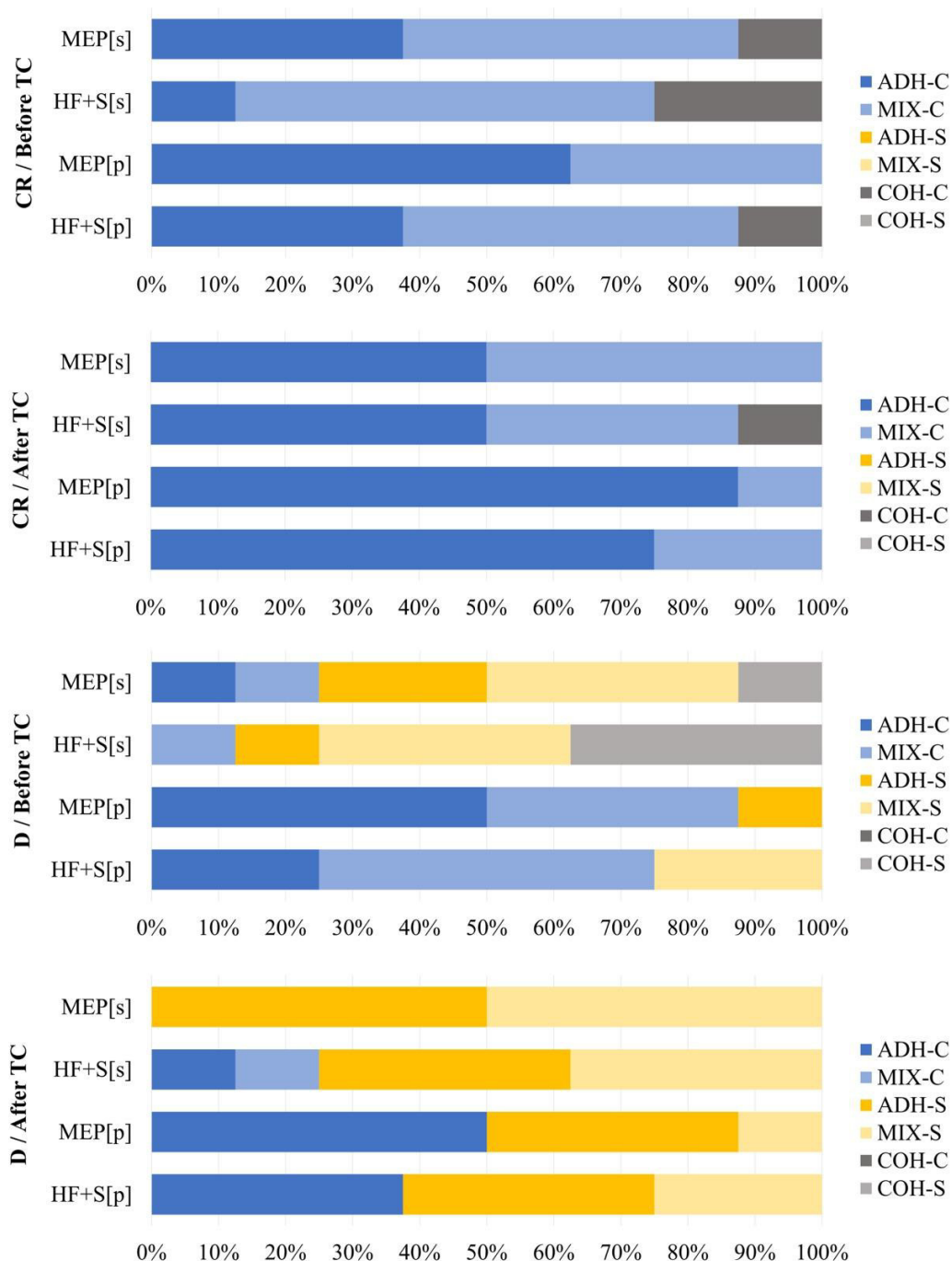


Figure 2. Failure modes observed in the experimental groups for microtensile bond strength to composite resin (RC) and dentin (D) substrates. ADH-C: adhesive failure between cement and ceramic; MIX-C: mixed failure involving adhesive and cohesive failure in ceramic; ADH-S: adhesive failure between cement and substrate; MIX-S: mixed failure involving adhesive and cohesive failure in substrate; COH-C: cohesive failure within ceramic; COH-S: cohesive failure within substrate. Blue tones represent adhesive and cohesive failures associated with the ceramic, yellow tones indicate those related to the substrates, and gray tones correspond to cohesive failures.



#### 4. Discussion

This study investigated the surface and mechanical properties of lithium disilicate-based glass-ceramic treated with two conditioning protocols – HF+S and a SeCP – under different surface conditions (polished and sandblasted). Additionally, the durability of the bond strength to composite resin and dentin substrates was assessed before and after TC. It was hypothesized that SeCP, with or without sandblasting, would (1) improve the mechanical and surface properties of lithium disilicate ceramics, (2) enhance the  $\mu$ TBS to composite resin both before and after TC, and (3) improve the  $\mu$ TBS to dentin under the same conditions, performing comparably to or better than HF+S.

Airborne-particle abrasion with  $\text{Al}_2\text{O}_3$  significantly increased Sa across all specimens, regardless of the conditioning agent, consistent with prior studies showing that sandblasting creates microporosities that enhance micromechanical retention [8,50]. This surface alteration was evident in SEM images (Figure 1). Among sandblasted groups, HF+S yielded the highest Sa values, likely due to the additive effect of HF acid, which further dissolves the glassy matrix and generates deeper micro-retentive features [7,51–55]. SEM analysis confirmed a more pronounced etching pattern in the HF+S group, with extensive microcavities and glassy phase dissolution. In contrast, no significant difference in Sa was observed between HF+S and MEP in polished specimens, indicating that sandblasting primarily drives surface roughness in this setting. Some studies reporting values comparable to those obtained with HF [26,38,56,57].

Wettability, measured by CA, was significantly improved by airborne-particle abrasion regardless of the conditioning agent. Sandblasted specimens showed lower CA values than polished ones, due to increased surface area and irregularities that facilitate adhesive spreading. Among all groups, sandblasted MEP-treated specimens had the lowest CA, indicating superior wettability. MEP's milder acidic component promotes controlled, uniform demineralization, as SEM images revealed a consistent surface without overetching or deep degradation. This helps preserve surface energy and enhances wettability without damaging the ceramic's structure [16,34,58]. These components are known to decrease CA and increase free surface energy [29,40,56,59], which explains the enhanced wettability observed. Together, these results suggest that combining MEP with sandblasting optimizes surface wetting while preserving ceramic integrity, favoring both chemical and micromechanical bonding, supported by the more uniform SEM topography in this group.

In contrast, although HF+S produced higher Sa, its aggressive and less selective etching created excessive porosity and irregularities, including microcavities. SEM images confirmed heterogeneous features and localized overetching, which may trap air within surface

depressions. According to the Cassie-Baxter model, when a liquid contacts a rough surface with trapped air pockets, the droplet rests partially on air rather than fully wetting the solid substrate. This composite solid–air interface reduces the liquid–solid contact area, thereby increasing the contact angle and impairing wettability, despite increased surface roughness [60–62]. Thus, the irregular topography created by HF+S may paradoxically reduce wettability due to incomplete liquid penetration into the microcavities, as supported by the SEM observations for this group.

Interestingly, airborne-particle abrasion significantly reduced flexural strength in all groups, regardless of the conditioning protocol. Among them, HF-treated specimens showed the lowest flexural strength values in both polished and sandblasted conditions, indicating that HF acid etching may compromise the mechanical integrity of lithium disilicate. This is likely due to the aggressive and non-selective dissolution of the glassy matrix by HF, which can lead to subsurface defects and microcracks that act as stress concentrators, especially when combined with prior abrasion [27,63,64]. By contrast, MEP-treated groups maintained flexural strength values statistically similar to the control group in both surface conditions, which may be attributed to the more conservative surface modification shown in SEM, characterized by a smoother and less damaged surface, even after airborne-particle abrasion. Thus, while sandblasting alone contributes to surface damage and loss of strength, the choice of conditioning agent modulates this effect – MEP being notably less harmful than HF, both from a mechanical and microstructural perspective.

Taken together, the results for surface roughness, wettability, and flexural strength suggest that the first hypothesis – stating that SeCP, with or without sandblasting, would improve the mechanical and surface properties of lithium disilicate ceramics – was partially accepted. Although MEP (SeCP) demonstrated significant advantages in terms of wettability and preservation of flexural strength when compared to HF, it did not improve surface roughness in the absence of sandblasting, nor did it fully prevent the strength reduction caused by abrasion. Therefore, while SeCP shows promise as a more conservative and effective alternative to HF – especially when combined with surface roughening – it did not lead to consistent improvements across all evaluated parameters. Nonetheless, its performance supports its use as a viable conditioning strategy that balances surface reactivity and structural preservation.

The long-term success of adhesive ceramic restorations relies heavily on the stability of the bond between the ceramic and the resin cement. Inadequate bonding at the interface can lead to failures that compromise the restoration, including microleakage, secondary caries, hypersensitivity, pulpal irritation, ceramic fracture, and even debonding [9,41]. As such,

identifying adhesive strategies that combine clinical practicality with durable bond strength remains a central goal in restorative dentistry.

In this study, TC significantly reduced  $\mu$ TBS across all groups, reinforcing the well-established understanding that hydrolytic degradation weakens the adhesive interface over time [44,65,66]. This trend is consistent with previous studies reporting substantial bond strength loss for both HF+S and MEP protocols after TC [2,4,22,23,38,40,43,67]. The degradation is attributed not only to hydrolytic breakdown of adhesive components but also to internal stresses from temperature fluctuations, which induce shear forces due to mismatched thermal expansion coefficients among materials – promoting crack formation and interfacial failure [5,11,40]. Simultaneously, water uptake accelerates the release of residual monomers and contributes to polymer matrix deterioration, further compromising the mechanical properties of polymer-based materials and increasing their likelihood of failure [68]. The shift in failure modes after TC corroborates this mechanism: mixed failures were largely replaced by adhesive ones on both substrates, indicating that thermal and hydrolytic stresses preferentially weakened the interfaces rather than the bulk of the materials. This transition suggests that degradation processes progressively reduce the capacity for stress dissipation across multiple interfaces, concentrating failures at the weakest adhesive bonds.

Despite the degradative effects of TC, sandblasted surfaces consistently demonstrated higher  $\mu$ TBS values than polished ones on both tested substrates – composite resin (core build-up simulation) and dentin – highlighting the importance of micromechanical retention via surface texturization. Although increasing ceramic surface roughness is a widely adopted strategy, it does not necessarily translate into improved bond strength, as no clear positive correlation between roughness and adhesion has been established [15]. The effectiveness of the resin-ceramic bond appears to depend more on the resin cement's ability to infiltrate and polymerize within the microtexture than on the size or depth of surface irregularities. Excessively aggressive treatments – such as prolonged HF acid application – can lead to excessive dissolution of the glassy matrix, creating deep and wide voids that may compromise long-term bonding [3,15,44]. This interpretation is consistent with the failure analysis: sandblasted groups showed a higher proportion of mixed failures before TC, indicating effective micromechanical interlocking, while polished groups exhibited mainly adhesive failures, reflecting weaker interaction with the cement. After TC, however, both conditions shifted toward adhesive failures, reinforcing that micromechanical retention delays but does not prevent interfacial degradation over time.

When evaluating the effect of conditioning agents, both HF+S and MEP yielded statistically similar  $\mu$ TBS values under most experimental conditions, indicating that MEP may serve as a viable alternative to the traditional HF+S protocol. This outcome is corroborated by several studies reporting no significant differences in bond strength between the two methods [2,4,9,15,17,18,30–43]. MEP acts through the interaction of ammonium polyfluoride and trimethoxypropyl methacrylate, requiring 20 seconds of active rubbing followed by a 40-second waiting period. While it promotes milder surface alterations compared to HF, its clinical effectiveness has been well documented [22,69]. Interestingly, two studies found MEP to outperform HF+S: one in specimens evaluated before TC [45], and another in comparison to 5% HF [44], underscoring the influence of specific bonding conditions and protocols. On the other hand, the superior performance of HF+S in certain situations can be attributed to its dual action: micromechanical interlocking provided by HF etching and chemical bonding achieved through silanization. HF stands out for its high capacity to dissolve the glassy matrix of ceramics, creating pronounced microporosities and microretentive features that improve surface roughness and facilitate stronger initial adhesion [26,29,40,43]. This initial advantage of HF+S was also reflected in the failure patterns: before TC, HF+S-treated ceramics bonded to composite resin showed a predominance of mixed failures at the ceramic side, consistent with stronger micromechanical retention. However, after TC, this benefit diminished, with adhesive failures becoming predominant, highlighting the susceptibility of the HF-derived surface to hydrolytic degradation.

The only exception observed in our findings occurred in the composite substrate prior to TC, where the HF+S protocol significantly outperformed MEP. This result may be explained by the more aggressive micromechanical retention produced by HF acid etching, which promotes pronounced microporosities and surface microretentions on the ceramic. Such features may have favored deeper penetration of the resin cement into the treated surface, resulting in stronger initial adhesion [26,29]. This effect was likely enhanced by the composite substrate, which, due to its rigid and hydrophobic nature, provides a more stable and less permeable bonding interface compared to dentin. Unlike dentin, which contains water and organic content that may interfere with resin infiltration and polymerization, the composite core may have facilitated more uniform stress distribution and more effective polymerization of the cement layer. Consequently, this condition maximized the adhesive benefit derived from the microtexture created by HF+S, emphasizing the importance of substrate characteristics in modulating the efficacy of surface treatments. Consistently, the failure mode analysis showed that composite resin substrates were associated with a higher proportion of failures at the

ceramic side, underscoring that when bonding to this substrate, the ceramic–cement interface is the most vulnerable link. Conversely, dentin groups revealed a greater incidence of failures at the dentin side, particularly after TC, highlighting the organic and hydrated nature of dentin as a critical factor compromising long-term stability.

Despite this favorable initial performance, the greatest reduction in  $\Delta\mu\text{TBS}$  after TC was observed in the HF+S-treated groups, particularly on polished surfaces. This finding suggests that interfaces exhibiting initially higher bond strengths may be more vulnerable to degradation under thermal stress. Although this reduction has often been attributed to the instability of silane bonding on smoother surfaces, it is more plausibly influenced by the characteristics of the micromechanical retention generated by HF etching. HF acid creates a complex and highly retentive surface topography – enhancing adhesion through increased surface area and mechanical interlocking – but such irregularities may also act as stress concentration zones if not adequately infiltrated by resin cement [2,18]. This effect was corroborated by SEM observations, which revealed more pronounced surface irregularities and deeper microporosities in the HF-treated specimens compared to those conditioned with MEP. Under repeated TC, these uneven regions may facilitate the initiation and propagation of microcracks, reducing bond stability over time. In contrast, the more uniform and moderately roughened surfaces produced by MEP likely favored more consistent resin infiltration and stress distribution, which may explain the smaller variation in bond strength observed after aging. This difference is again reflected in the failure patterns, where MEP-treated specimens showed more balanced distributions of mixed and adhesive failures before aging, with a less abrupt shift to adhesive failures after TC compared to HF+S, suggesting better interfacial stability.

The findings of this study hold significant clinical relevance for the selection of conditioning protocols in bonding to dental and restorative substrates. The comparable bond strength observed with SeCP relative to the conventional HF+S protocol – particularly after TC – supports SeCP as a promising, less aggressive alternative for ceramic surface treatment. This simplifies clinical procedures while reducing the risks associated with HF acid use. Moreover, SeCP demonstrated consistent and reliable performance on both dentin and composite resin substrates, highlighting its versatility across a variety of restorative scenarios and its potential to facilitate adhesion in complex restorations involving multiple substrates. Clinically, the failure mode results suggest that when bonding to composite resin cores, clinicians should be aware that the ceramic–cement interface is the critical site of degradation, while in dentin, the dentin–cement interface is more likely to compromise restoration longevity. Sandblasting improved initial interfacial performance in both cases by increasing the proportion of mixed

failures, although adhesive failures became dominant after aging. This highlights the transient nature of micromechanical benefits and the importance of chemical durability in long-term adhesion. Importantly, although all groups exhibited some reduction in bond strength after TC, this decline was also present in the HF+S groups. Notably, the magnitude of bond strength reduction ( $\Delta\mu\text{TBS}$ ) was statistically less pronounced for SeCP-treated surfaces, indicating better stability under TC. These results fully support hypotheses 2 and 3.

Nonetheless, some limitations should be considered. In vitro conditions cannot fully replicate the complex oral environment, where saliva, operator variability, and patient factors influence adhesive durability. While TC is a standard aging method, it does not encompass all clinical mechanical and chemical challenges. Thus, further studies—including extended aging protocols and clinical trials—are needed to confirm SeCP's long-term performance in vivo. Future research should also investigate different restorative materials, SeCP formulations, application techniques, and the chemical and morphological dynamics at the adhesive interface to better optimize bonding. Ultimately, developing safer, simpler conditioning methods that maintain or enhance bond strength remains essential for achieving more predictable and lasting dental restorations.

## 5. Conclusion

Within the limitations of this study, MEP combined with airborne-particle abrasion showed bond strength comparable to HF, with smaller  $\mu\text{TBS}$  reduction after TC. MEP resulted in improved wettability and higher flexural strength than HF, although lower than untreated controls, and produced lower surface roughness. Adhesive performance was consistent across dentin and composite substrates, supporting MEP as a viable surface treatment for lithium disilicate ceramics in medium-term adhesion.

## References

- [1] Moura DMD, de Araújo AMM, de Souza KB, Veríssimo AH, Tribst JPM, de Assunção e Souza RO. Hydrofluoric acid concentration, time and use of phosphoric acid on the bond strength of feldspathic ceramics. *Braz Oral Res* 2020;34:e018. <https://doi.org/10.1590/1807-3107BOR-2020.VOL34.0018>.
- [2] de Siqueira FSF, Campos VS, Wendlinger M, Muso RAC, Gomes JC, Reis A, et al. Effect of self-etching primer associated to hydrofluoric acid or silane on bonding to lithium disilicate. *Braz Dent J* 2019;30:171–8. <https://doi.org/10.1590/0103-6440201902366>.

- [3] Lopes GC, Perdigão J, Baptista D, Ballarin A. Does a self-etching ceramic primer improve bonding to lithium disilicate ceramics? Bond strengths and FeSEM analyses. *Oper Dent* 2019;44:210–8. <https://doi.org/10.2341/17-355-L>.
- [4] Lyann SK, Takagaki T, Nikaido T, Uo M, Ikeda M, Sadr A, et al. Effect of Different Surface Treatments on the Tensile Bond Strength to Lithium Disilicate Glass Ceramics. *J Adhes Dent* 2018;20:261-8. <https://doi.org/10.3290/j.jad.a40632>.
- [5] Souza KB, Moura DMD, Da Silva SEG, De Araújo GM, De Pinto RAS, Leite FPP, et al. Effect of different surface treatments and multimode adhesive application on the Weibull characteristics, wettability, surface topography and adhesion to CAD/CAM lithium disilicate ceramic. *J Appl Oral Sci* 2020;28:1–12. <https://doi.org/10.1590/1678-7757-2020-0122>.
- [6] Malysa A, Wezgowiec J, Orzeszek S, Florjanski W, Zietek M, Wieckiewicz M. Effect of Different Surface Treatment Methods on Bond Strength of Dental Ceramics to Dental Hard Tissues: A Systematic Review. *Molecules* 2021;26:1–14. <https://doi.org/https://doi.org/10.3390/molecules26051223>.
- [7] Oliveira LT, de Castro EF, Azevedo V, de Andrade OS, Faraoni JJ, Palma-Dibb RG, et al. Effect of Ceramic Conditioners on Surface Morphology, Roughness, Contact Angle, Adhesion, Microstructure, and Composition of CAD/CAM Ceramics. *Oper Dent* 2023;48:277–93. <https://doi.org/10.2341/21-078-L>.
- [8] Thiesen KPPR, de Souza MDB, Schmitt VL, Nahsan FPS, Naufel FS. Bond strength to ZLS ceramics at different etching times and cementation protocols after aging. *Braz Dent Sci* 2019;22:488–96. <https://doi.org/10.14295/bds.2019.v22i4.1816>.
- [9] Turunç Oğuzman R, Şişmanoğlu S. Adhesive Performance of Resin Cement to Glass-Ceramic and Polymer-Based Ceramic CAD/CAM Materials after Applying Self-Etching Ceramic Primer or Different Surface Treatments. *Materials* 2024;17:2. <https://doi.org/10.3390/ma17010002>.
- [10] Murillo-Gómez F, Wanderley RB, De Goes MF. Impact of silane-containing universal adhesive on the biaxial flexural strength of a resin cement/glass-ceramic system. *Oper Dent* 2019;44:200–9. <https://doi.org/10.2341/17-356-L>.
- [11] Murillo-Gómez F, De Goes MF. Effect of Different Silane Treatments on Long-Term Bonding Between Non-Etched Glass-Ceramic and Resin Cement. *Odovtos-Int J Dent Sc* 2017;19:33–46. <https://doi.org/10.15517/ijds.v0i0.27950>.

- [12] Wille S, Lehmann F, Kern M. Durability of resin bonding to lithium disilicate using different self-etching and conventional ceramic primers after long-term aging. *Dent Mater* 2022;38:444–50. <https://doi.org/10.1016/j.dental.2021.12.027>.
- [13] Sundfeld Neto D, Naves LZ, Costa AR, Correr AB, Consani S, Borges GA, et al. The effect of hydrofluoric acid concentration on the bond strength and morphology of the surface and interface of glass ceramics to a resin cement. *Oper Dent* 2015;40:470–9. <https://doi.org/10.2341/14-133-L>.
- [14] Awad MM, Alhalabi F, Alotaibi N, Alzamil F, Binalrimal S, Alrahlah A, et al. A systematic review and meta-analysis of bond strength studies associated with self-etching primer and HF acid etching of dental glass-ceramics. *Int J Adhes Adhes* 2022;118:103216. <https://doi.org/10.1016/j.ijadhadh.2022.103216>.
- [15] Colombo L do A, Murillo-Gómez F, De Goes MF. Bond Strength of CAD/CAM Restorative Materials Treated with Different Surface Etching Protocols. *J Adhes Dent* 2019;21:307–17. <https://doi.org/10.3290/j.jad.a42931>.
- [16] Fonzar RF, Goracci C, Carrabba M, Louca C, Ferrari M, Vichi A. Influence of acid concentration and etching time on composite cement adhesion to lithium-silicate glass ceramics. *J Adhes Dent* 2020;22:175–82. <https://doi.org/10.3290/j.jad.a44282>.
- [17] Vichi A, Fabian Fonzar R, Carrabba M, Louca C, Scotti N, Mazzitelli C, et al. Comparison between hydrofluoric acid and single-component primer as conditioners on resin cement adhesion to lithium silicate and lithium disilicate glass ceramics. *Materials* 2021;14:6776. <https://doi.org/10.3390/ma14226776>.
- [18] Román-Rodríguez J-L, Perez-Barquero J-A, Gonzalez-Angulo E, Fons-Font A, Bustos-Salvador J-L. Bonding to silicate ceramics: Conventional technique compared with a simplified technique. *J Clin Exp Dent* 2017;9:e384–6. <https://doi.org/10.4317/jced.53570>.
- [19] Dapieve KS, Aragonéz GC, Prochnow C, de Lima Burgo TA, Rippe MP, Pereira GKR, et al. Different Etching Times Of A One-Step Ceramic Primer: Effect On The Resin Bond Strength Durability To A Cad/Cam Lithium-Disilicate Glass-Ceramic. *J Adhes Dent* 2021;23:133–43. <https://doi.org/10.3290/j.jad.b1079573>.
- [20] Alayad A. Influence of lithium disilicate pretreatment using photodynamic therapy, non-thermal plasma, and laser treatment on surface roughness and bond strength. *Photodiagnosis Photodyn Ther* 2023;42:103512. <https://doi.org/10.1016/j.pdpdt.2023.103512>.



- [21] Alkhudhairy F. Microtensile and Microshear Bond Strength of Cemented Ceramics Using Self-Etching Primer versus Conventional Surface Treatment. *J Biomater Tissue Eng* 2018;8:698–703. <https://doi.org/10.1166/jbt.2018.1789>.
- [22] Çinar Ş, Fildişi MA, Altan B, Ozkan D. Effects of Two Different Acid Etching and Surface Washing Methods on Bond Strength on Different CAD-CAM Blocks under Aging Protocols. *Biomed Res Int* 2023;2023: 7982813. <https://doi.org/10.1155/2023/7982813>.
- [23] Dimitriadi M, Zinelis S, Zafiropoulou M, Silikas N, Eliades G. Self-etch silane primer: Reactivity and bonding with a lithium disilicate ceramic. *Materials* 2020;13:641. <https://doi.org/10.3390/ma13030641>.
- [24] Donmez MB, Okutan Y, Yucel MT. Effect of prolonged application of single-step self-etching primer and hydrofluoric acid on the surface roughness and shear bond strength of CAD/CAM materials. *Eur J Oral Sci* 2020;128:542–9. <https://doi.org/10.1111/eos.12747>.
- [25] El-Damanhoury HM, Elsahn NA, Sheela S, Gaintantzopoulou MD. Adhesive luting to hybrid ceramic and resin composite CAD/CAM Blocks: Er:YAG Laser versus chemical etching and micro-abrasion pretreatment. *J Prosthodont Res.* 2020;65:225-34. [https://doi.org/10.2186/jpor.JPOR\\_2020\\_50](https://doi.org/10.2186/jpor.JPOR_2020_50).
- [26] El-Damanhoury HM, Gaintantzopoulou MD. Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance. *J Prosthodont Res* 2018;62:75–83. <https://doi.org/10.1016/j.jpor.2017.06.002>.
- [27] Guimarães HAB, Cardoso PC, Decurcio RA, Monteiro LJE, De Almeida LN, Martins WF, et al. Simplified Surface Treatments for Ceramic Cementation: Use of Universal Adhesive and Self-Etching Ceramic Primer. *Int J Biomater* 2018;2018: 2598073. <https://doi.org/10.1155/2018/2598073>.
- [28] Mously HA, Kazim SA, Nageeb RN, Naguib GH, Hamed MT. Influence of Shear Bonding Strength of Lithium Disilicate to Enamel under Different Surface Treatments. *J Evol Med Dent Sci* 2020;9:705–10. <https://doi.org/10.14260/jemds/2020/154>.
- [29] Prado M, Prochnow C, Maria A, Marchionatti E, Baldissara P, Valandro LF, et al. Ceramic Surface Treatment with a Single-component Primer: Resin Adhesion to Glass Ceramics. *J Adhes Dent* 2018;20:99–105. <https://doi.org/10.3290/j.jad.a40303>.
- [30] Alkhudhairy F, Naseem M, Ahmad ZH, Alnooh AN, Vohra F. Efficacy of phototherapy with different conventional surface treatments on adhesive quality of lithium disilicate ceramics. *Photodiagnosis Photodyn Ther* 2019;25:292–5. <https://doi.org/10.1016/j.pdpdt.2019.01.015>.

- [31] Alrahlah A, Awad MM, Vohra F, Al-Mudahhi A, Al jeaidi ZA, Elsharawy M. Effect of self etching ceramic primer and universal adhesive on bond strength of lithium disilicate ceramic. *J Adhes Sci Technol* 2017;31:2611–9. <https://doi.org/10.1080/01694243.2017.1312079>.
- [32] Alshihri A. Etching efficacy and bonding performance of resin to lithium disilicate ceramic using self-etching primer with different reaction times. *J Adhes Sci Technol* 2019;33:1215–25. <https://doi.org/10.1080/01694243.2019.1585028>.
- [33] Cardenas AFM, Quintero-Calderon AS, Siqueira FSF de, Campos VS, Wendlinger M, Pulido-Mora CA, et al. Do Different Application Modes Improve the Bonding Performance of Self-etching Ceramic Primer to Lithium Disilicate and Feldspathic Ceramics? *J Adhes Dent* 2019;21:319–27. <https://doi.org/10.3290/j.jad.a42929>.
- [34] Dapieve KS, Pilecco RO, Temp RW, Villetti MA, Pereira GKR, Valandro LF. Adhesion to lithium disilicate glass-ceramics after aging: Resin viscosity and ceramic surface treatment effects. *J Mech Behav Biomed Mater* 2023;142: 105819. <https://doi.org/10.1016/j.jmbbm.2023.105819>.
- [35] Klippel GGP, Melo-Silva CL, Melo-Silva TCF, Elias CN, Biasic RS, Dos Santos C. Shear bond strength of lithium disilicate to resin cement after treatment with hydrofluoric acid and a self-etching ceramic primer. *Mater Res* 2021;24:e20210079. <https://doi.org/10.1590/1980-5373-MR-2021-0079>.
- [36] Liebermann A, Detzer J, Stawarczyk B. Impact of recently developed universal adhesives on tensile bond strength to computer-aided design/manufacturing ceramics. *Oper Dent* 2019;44:386–95. <https://doi.org/10.2341/18-017-L>.
- [37] Maier E, Bordihn V, Belli R, Taschner M, Petschelt A, Lohbauer U, et al. New Approaches in Bonding to Glass-Ceramic: Self-Etch Glass-Ceramic Primer and Universal Adhesives. *J Adhes Dent* 2019;21:209-17. <https://doi.org/10.3290/j.jad.a42546>.
- [38] Murillo-Gómez F, De Goes MF. Bonding effectiveness of tooth-colored materials to resin cement provided by self-etching silane primer after short- and long-term storage. *J Prosthet Dent* 2019;121:713.e1-713.e8. <https://doi.org/10.1016/j.prosdent.2018.12.018>.
- [39] Siqueira FSF, Alessi RS, Cardenas AFM, Kose C, Pinto SCS, Bandeca MC, et al. New single-bottle ceramic primer: 6-month case report and laboratory performance. *J Contemp Dent Pract* 2016;17:1033–9. <https://doi.org/10.5005/jp-journals-10024-1977>.

- [40] Tribst JPM, Anami LC, Özcan M, Bottino MA, Melo RM, Saavedra GSFA. Self-etching primers vs acid conditioning: Impact on bond strength between ceramics and resin cement. *Oper Dent* 2018;43:372–9. <https://doi.org/10.2341/16-348-L>.
- [41] Lu Y, Bierman TE, De Oliveira Dal Piva AM, Tribst JPM, Feilzer AJ, Kleverlaan CJ. Effect of Surface Treatment and Resin Cement on the Bond Strength of an Advanced Lithium Disilicate. *Eur J Dent* 2024;18:869-76. <https://doi.org/10.1055/s-0043-1776358>.
- [42] Vila-Nova TEL, Moura DMD, Araújo GM de, Pinto R de AS, Leite FPP, Melo RM, et al. Effect of Adhesive Resin Application on the Durability of Adhesion to CAD/CAM Glass-Ceramics after either Hydrofluoric Acid Etching or Self-etch Primer Application. *J Adhes Dent* 2022;24:279–89. <https://doi.org/10.3290/j.jad.b3240691>.
- [43] Wille S, Lehmann F, Kern M. Durability of Resin Bonding to Lithium Disilicate and Zirconia Ceramic using a Self-etching Primer. *J Adhes Dent* 2017;19:491–6. <https://doi.org/10.3290/j.jad.a39545>.
- [44] Levartovsky S, Bohbot H, Shem-Tov K, Brosh T, Pilo R. Effect of different surface treatments of lithium disilicate on the adhesive properties of resin cements. *Materials* 2021;14:3302. <https://doi.org/10.3390/ma14123302>.
- [45] Turunç-Oğuzman R, Şişmanoğlu S. Influence of surface treatments and adhesive protocols on repair bond strength of glass-matrix and resin-matrix CAD/CAM ceramics. *J Esthet Restor Dent* 2023;35:1322–31. <https://doi.org/10.1111/jerd.13131>.
- [46] Faggion CM. Guidelines for reporting pre-clinical in vitro studies on dental materials. *J Evid-Based Dent Pract* 2012;12:182–9. <https://doi.org/10.1016/j.jebdp.2012.10.001>.
- [47] Huhtamäki T, Tian X, Korhonen JT, Ras RHA. Surface-wetting characterization using contact-angle measurements. *Nat Protoc* 2018;13:1521–38. <https://doi.org/10.1038/s41596-018-0003-z>.
- [48] Meharry MR, Moazzami SM, Li Y. Comparison of enamel and dentin shear bond strengths of current dental bonding adhesives from three bond generations. *Oper Dent* 2013;38:e237-45. <https://doi.org/10.2341/12-521-L>.
- [49] Paloco EAC, Berger SB, Lopes MB, Favaro JC, Gonini-Júnior A, Piauilino AIF, et al. Influence of resin cement and thermocycling on milled lithium disilicate ceramic microshear bond strength. *Acta Odontol Latinoam* 2021;34:226-32. <https://doi.org/10.54589/aol.34/3/226>
- [50] Borges GA, Sophr AM, Fernando De Goes M, Sobrinho LC, Chan DCN. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 2003;89:479-88. [https://doi.org/10.1016/s0022-3913\(02\)52704-9](https://doi.org/10.1016/s0022-3913(02)52704-9).

- [51] Demir N, Bozkurt M, Temizci T. Comparison of optical properties and bond strength of new CAD/CAM ceramic materials. *Mater Res Express* 2024;11:095202. <https://doi.org/10.1088/2053-1591/ad524e>.
- [52] Zogheib L V, Della Bona A, Kimpara ET, McCabe JF. Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. *Braz Dent J* 2011;22:45–50. <https://doi.org/10.1590/s0103-64402011000100008>.
- [53] Sudré JP, Salvio LA, Baroudi K, Sotto-Maior BS, Melo-Silva CL, Picorelli NMS. Influence of surface treatment of lithium disilicate on roughness and bond strength. *Int J Prosthodont* 2020;33:212–6. <https://doi.org/10.11607/ijp.6453>.
- [54] Venturini AB, Prochnow C, May LG, Bottino MC, Felipe Valandro L. Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic. *J Mech Behav Biomed Mater* 2015;48:241–8. <https://doi.org/10.1016/j.jmbbm.2015.03.025>.
- [55] Ramakrishnaiah R, Alkheraif AA, Divakar DD, Matinlinna JP, Vallittu PK. The effect of hydrofluoric acid etching duration on the surface micromorphology, roughness, and wettability of dental ceramics. *Int J Mol Sci* 2016;17:822. <https://doi.org/10.3390/ijms17060822>.
- [56] Strasser T, Preis V, Behr M, Rosentritt M. Roughness, surface energy, and superficial damages of CAD/CAM materials after surface treatment. *Clin Oral Investig* 2018;22:2787–97. <https://doi.org/10.1007/s00784-018-2365-6>.
- [57] Murillo-Gómez F, Palma-Dibb RG, De Goes MF. Effect of acid etching on tridimensional microstructure of etchable CAD/CAM materials. *Dent Mater* 2018;34:944–55. <https://doi.org/10.1016/j.dental.2018.03.013>.
- [58] Del Bianco F, Mazzitelli C, Maravic T, Josic U, Florenzano F, Baldissara P, et al. Bond Strength to Lithium-Disilicate Ceramic after Different Surface Cleaning Approaches. *J Adhes Dent* 2024;26:11–8. <https://doi.org/10.3290/j.jad.b4874329>.
- [59] Moreno MBP, Murillo-Gómez F, de Goes MF. Physicochemical and morphological characterization of a glass ceramic treated with different ceramic primers and post-silanization protocols. *Dent Mater* 2019;35:1073–81. <https://doi.org/10.1016/j.dental.2019.05.003>.
- [60] Marmur A. Wetting on hydrophobic rough surfaces: To be heterogeneous or not to be? *Langmuir* 2003;19:8343–8. <https://doi.org/10.1021/la0344682>.
- [61] Bhushan B, Jung YC. Natural and biomimetic artificial surfaces for superhydrophobicity, self-cleaning, low adhesion, and drag reduction. *Prog Mater Sci* 2011;56:1–108. <https://doi.org/10.1016/j.pmatsci.2010.04.003>.

- [62] Cassie ABD, Baxter S. Wettability of porous surfaces. *Trans Faraday Soc* 1944;40:546–51. <https://doi.org/10.1039/tf9444000546>.
- [63] Freitas JS, Souza LFB, Pereira GKR, May LG. Surface properties and flexural fatigue strength of an advanced lithium disilicate. *J Mech Behav Biomed Mater* 2023;147:106154. <https://doi.org/10.1016/j.jmbbm.2023.106154>.
- [64] Porto TS, Roperto RC, Teich ST, Faddoul FF, Rizzante FAP, Porto-Neto ST, et al. Brittleness index and its relationship with materials mechanical properties: Influence on the machinability of CAD/CAM materials. *Braz Oral Res* 2019;33:e026. <https://doi.org/10.1590/1807-3107bor-2019.vol33.0026>.
- [65] Brentel AS, Özcan M, Valandro LF, Alarça LG, Amaral R, Bottino MA. Microtensile bond strength of a resin cement to feldspathic ceramic after different etching and silanization regimens in dry and aged conditions. *Dent Mater* 2007;23:1323–31. <https://doi.org/10.1016/j.dental.2006.11.011>.
- [66] Höller B, Belli R, Petschelt A, Lohbauer U, Zorzin JI. Influence of Simulated Oral Conditions on Different Pretreatment Methods for the Repair of Glass-Ceramic Restorations. *J Adhes Dent* 2022;24:57–66. <https://doi.org/10.3290/j.jad.b2701717>.
- [67] Zorzin J, Beck S, Belli R, Petschelt A, Boccaccini AR, Lohbauer U. Adhesion and interfacial characterization of biomimetically texturized lithium disilicate. *Int J Adhes Adhes* 2019;91:131–41. <https://doi.org/10.1016/j.ijadhadh.2019.03.012>.
- [68] Scherer MM, Prochnow C, Venturini AB, Pereira GKR, Burgo TA de L, Rippe MP, et al. Fatigue failure load of an adhesively-cemented lithium disilicate glass-ceramic: Conventional ceramic etching vs etch & prime one-step primer. *Dent Mater* 2018;34:1134–43. <https://doi.org/10.1016/j.dental.2018.04.012>.
- [69] Dapieve KS, Machry R V, Pilecco RO, Kleverlaan CJ, Rocha Pereira GK, Venturini AB, et al. One-step ceramic primer as surface conditioner: Effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations. *J Mech Behav Biomed Mater* 2020;104:103686. <https://doi.org/10.1016/j.jmbbm.2020.103686>.

## 6 CONCLUSÃO GERAL

Dentro das limitações deste estudo, o primer autocondicionante Monobond Etch & Prime (MEP), especialmente quando associado ao jateamento com óxido de alumínio, apresentou desempenho adesivo comparável ao protocolo convencional com ácido fluorídrico e silano (HF+S), com menor variação na resistência adesiva após termociclagem, sugerindo maior estabilidade em médio prazo.

O MEP resultou em melhor molhabilidade e maior resistência à flexão do que o HF+S, embora inferior ao grupo controle. Apesar de apresentar menor rugosidade superficial, ainda foi eficaz na adesão quando associado ao jateamento, demonstrando que a combinação promove suficiente embricamento mecânico sem comprometer a integridade da cerâmica.

Assim, o MEP associado ao jateamento se mostra uma alternativa promissora ao HF+S, oferecendo equilíbrio entre desempenho adesivo, preservação mecânica e viabilidade clínica.

## REFERÊNCIAS

ALAYAD, A. Influence of lithium disilicate pretreatment using photodynamic therapy, non-thermal plasma, and laser treatment on surface roughness and bond strength. **Photodiagnosis and Photodynamic Therapy**, v. 42, p. 103512, 2023.

AL-HARTHI, A. A. *et al.* Laboratory study of micro-shear bond strength of two resin cements to leucite ceramics using different ceramic primers. **Journal of Contemporary Dental Practice**, v. 19, n. 8, p. 918–924, 2018.

ALKHUDHAIRY, F. Microtensile and microshear bond strength of cemented ceramics using self-etching primer versus conventional surface treatment. **Journal of Biomaterials and Tissue Engineering**, v. 8, n. 5, p. 698–703, 2018.

ALKHUDHAIRY, F. *et al.* Efficacy of phototherapy with different conventional surface treatments on adhesive quality of lithium disilicate ceramics. **Photodiagnosis and Photodynamic Therapy**, v. 25, p. 292–295, 2019.

ALMIRO, M. *et al.* Increasing acid concentration, time and using a two-part silane potentiates bond strength of lithium disilicate–reinforced glass ceramic to resin composite: an exploratory laboratory study. **Materials**, v. 15, n. 6, p. 2045, 2022.

ALRAHLAH, A. *et al.* Effect of self-etching ceramic primer and universal adhesive on bond strength of lithium disilicate ceramic. **Journal of Adhesion Science and Technology**, v. 31, n. 23, p. 2611–2619, 2017.

ALSHIHRI, A. Etching efficacy and bonding performance of resin to lithium disilicate ceramic using self-etching primer with different reaction times. **Journal of Adhesion Science and Technology**, v. 33, n. 11, p. 1215–1225, 2019.

AWAD, M. M. *et al.* A systematic review and meta-analysis of bond strength studies associated with self-etching primer and HF acid etching of dental glass-ceramics. **International Journal of Adhesion and Adhesives**, v. 118, p. 103216, 2022.

BHUSHAN, B.; JUNG, Y. C. Natural and biomimetic artificial surfaces for superhydrophobicity, self-cleaning, low adhesion, and drag reduction. **Progress in Materials Science**, v. 56, n.1, p. 1–108, 2011.

BORGES, G. A. *et al.* Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. **The Journal of Prosthetic Dentistry**, v. 8, n. 5, p. 479–488, 2003.

BRENTEL, A. S. *et al.* Microtensile bond strength of a resin cement to feldspathic ceramic after different etching and silanization regimens in dry and aged conditions. **Dental Materials**, v. 23, n. 11, p. 1323–1331, 2007.

CARDENAS, A. F. M. *et al.* Do different application modes improve the bonding performance of self-etching ceramic primer to lithium disilicate and feldspathic ceramics? **The Journal of Adhesive Dentistry**, v. 21, n. 4, p. 319–327, 2019.

CASSIE, A. B. D.; BAXTER, S. Wettability of porous surfaces. **Transactions of the Faraday Society**, v. 40, p. 546–551, 1944.

ÇINAR, Ş. *et al.* Effects of two different acid etching and surface washing methods on bond strength on different CAD-CAM blocks under aging protocols. **BioMed Research International**, v. 2023, p. 7982813, 2023.

COLOMBO, L. A.; MURILLO-GÓMEZ, F.; DE GOES, M. F. Bond strength of CAD/CAM restorative materials treated with different surface etching protocols. **The Journal of Adhesive Dentistry**, v. 21, n. 4, p. 307–317, 2019.

DAPIEVE, K. S. *et al.* One-step ceramic primer as surface conditioner: Effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 104, p. 103686, 2020.

DAPIEVE, K. S. *et al.* Different etching times of a one-step ceramic primer: Effect on the resin bond strength durability to a CAD/CAM lithium-disilicate glass-ceramic. **The Journal of Adhesive Dentistry**, v. 23, n. 2, p. 133–143, 2021.

DAPIEVE, K. S. *et al.* Adhesion to lithium disilicate glass-ceramics after aging: Resin viscosity and ceramic surface treatment effects. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 142, p. 105819, 2023.

DE SIQUEIRA, F. S. F. *et al.* Effect of self-etching primer associated to hydrofluoric acid or silane on bonding to lithium disilicate. **Brazilian Dental Journal**, v. 30, n. 2, p. 171–178, 2019.

DEL BIANCO, F. *et al.* Bond strength to lithium-disilicate ceramic after different surface cleaning approaches. **The Journal of Adhesive Dentistry**, v. 26, p. 11–18, 2024.

DEMIR, N.; BOZKURT, M.; TEMIZCI, T. Comparison of optical properties and bond strength of new CAD/CAM ceramic materials. **Materials Research Express**, v. 11, n. 9, p. 095202, 2024.

DIMITRIADI, M. *et al.* Self-etch silane primer: Reactivity and bonding with a lithium disilicate ceramic. **Materials**, v. 13, n. 3, p. 641, 2020.

DONMEZ, M. B.; OKUTAN, Y.; YUCEL, M. T. Effect of prolonged application of single-step self-etching primer and hydrofluoric acid on the surface roughness and shear bond strength of CAD/CAM materials. **European Journal of Oral Sciences**, v. 128, n. 6, p. 542–549, 2020.

EL-DAMANHOURY, H. M. *et al.* Adhesive luting to hybrid ceramic and resin composite CAD/CAM Blocks: Er:YAG Laser versus chemical etching and micro-abrasion pretreatment. **Journal of Prosthodontic Research**, v. 65, n. 2, p. 225–234, 2020.

EL-DAMANHOURY, H. M.; GAINANTZOPOULOU, M. D. Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance. **Journal of Prosthodontic Research**, v. 62, n. 1, p. 75–83, 2018.

FAGGION, C. M. Guidelines for reporting pre-clinical in vitro studies on dental materials. **Journal of Evidence-Based Dental Practice**, v. 12, n. 4, p. 182–189, 2012.



FONZAR, R. F. *et al.* Influence of acid concentration and etching time on composite cement adhesion to lithium-silicate glass ceramics. **The Journal of Adhesive Dentistry**, v. 22, n. 2, p. 175–182, 2020.

FREITAS, J. S. *et al.* Surface properties and flexural fatigue strength of an advanced lithium disilicate. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 147, p. 106154, 2023.

GUIMARÃES, H. A. B. *et al.* Simplified surface treatments for ceramic cementation: Use of universal adhesive and self-etching ceramic primer. **International Journal of Biomaterials**, v. 2018, p. 2598073, 2018.

HÖLLER, B. *et al.* Influence of simulated oral conditions on different pretreatment methods for the repair of glass-ceramic restorations. **The Journal of Adhesive Dentistry**, v. 24, n. 1, p. 57–66, 2022.

HUHTAMÄKI, T. *et al.* Surface-wetting characterization using contact-angle measurements. **Nature Protocols**, v. 13, n. 7, p. 1521–1538, 2018.

KLIPPEL, G. G. P. *et al.* Shear bond strength of lithium disilicate to resin cement after treatment with hydrofluoric acid and a self-etching ceramic primer. **Materials Research**, v. 24, n. 4, p. e20210079, 2021.

KOMOTO, M.; MASEKI, T.; NARA, Y. The effect of additional photochemical treatments on the bonding of silanized CAD/CAM ceramic restorations after water-storage. **Odontology**, v. 109, n. 3, p. 585–595, 2021.

LEVARTOVSKY, S. *et al.* Effect of different surface treatments of lithium disilicate on the adhesive properties of resin cements. **Materials**, v. 14, n. 12, p. 3302, 2021.

LIEBERMANN, A.; DETZER, J.; STAWARCZYK, B. Impact of recently developed universal adhesives on tensile bond strength to computer-aided design/manufacturing ceramics. **Operative Dentistry**, v. 44, n. 4, p. 386–395, 2019.

LOPES, G. C. *et al.* Does a self-etching ceramic primer improve bonding to lithium disilicate ceramics? Bond strengths and FeSEM analyses. **Operative Dentistry**, v. 44, n. 2, p. 210–218, 2019.

LU, Y. *et al.* Effect of surface treatment and resin cement on the bond strength of an advanced lithium disilicate. **European Journal of Dentistry**, v. 18, n. 13, p. 869–876, 2024.

LYANN, S. K. *et al.* Effect of different surface treatments on the tensile bond strength to lithium disilicate glass ceramics. **The Journal of Adhesive Dentistry**, v. 20, n. 3, p. 261–268, 2018.

MAIER, E. *et al.* New approaches in bonding to glass-ceramic: self-etch glass-ceramic primer and universal adhesives. **The Journal of Adhesive Dentistry**, v. 21, n. 3, p. 209–217, 2019.

MALYSA, A. *et al.* Effect of different surface treatment methods on bond strength of dental ceramics to dental hard tissues: A systematic review. **Molecules**, v. 26, n. 26, p. 1–14, 2021.

MARMUR, A. Wetting on hydrophobic rough surfaces: To be heterogeneous or not to be? **Langmuir**, v. 19, n. 20, p. 8343–8348, 2003.

MEHARRY, M. R.; MOAZZAMI, S. M.; LI, Y. Comparison of enamel and dentin shear bond strengths of current dental bonding adhesives from three bond generations. **Operative Dentistry**, v. 38, n. 6, p. e237–e245, 2013.

MORENO, M. B. P.; MURILLO-GÓMEZ, F.; DE GOES, M. F. Physicochemical and morphological characterization of a glass ceramic treated with different ceramic primers and post-silanization protocols. **Dental Materials**, v. 35, n. 8, p. 1073–1081, 2019.

MOURA, D. M. D. *et al.* Hydrofluoric acid concentration, time and use of phosphoric acid on the bond strength of feldspathic ceramics. **Brazilian Oral Research**, v. 34, p. e018, 2020.

MOUSLY, H. A. *et al.* Influence of shear bonding strength of lithium disilicate to enamel under different surface treatments. **Journal of Evolution of Medical and Dental Sciences**, v. 9, n. 10, p. 705–710, 2020.

MURILLO-GÓMEZ, F.; PALMA-DIBB, R. G.; DE GOES, M. F. Effect of acid etching on tridimensional microstructure of etchable CAD/CAM materials. **Dental Materials**, v. 34, n. 6, p. 944–955, 2018.

MURILLO-GÓMEZ, F.; DE GOES, M. F. Effect of different silane treatments on long-term bonding between non-etched glass-ceramic and resin cement. **Odovtos International Journal of Dental Sciences**, v. 19, n. 2, p. 33–46, 2017.

MURILLO-GÓMEZ, F.; DE GOES, M. F.. Bonding effectiveness of tooth-colored materials to resin cement provided by self-etching silane primer after short- and long-term storage. **Journal of Prosthetic Dentistry**, v. 121, n. 4, p. 713.e1-713.e8, 2019.

MURILLO-GÓMEZ, F.; WANDERLEY, R. B.; DE GOES, M. F. Impact of silane-containing universal adhesive on the biaxial flexural strength of a resin cement/glass-ceramic system. **Operative Dentistry**, v. 44, n. 2, p. 200–209, 2019.

OLIVEIRA, L. T. *et al.* Effect of ceramic conditioners on surface morphology, roughness, contact angle, adhesion, microstructure, and composition of CAD/CAM ceramics. **Operative Dentistry**, v. 48, n. 3, p. 277–293, 2023.

PALOCO, E. A. C. *et al.* Influence of resin cement and thermocycling on milled lithium disilicate ceramic microshear bond strength. **Acta Odontologica Latinoamericana**, v. 34, n. 3, p. 226–232, 2021.

PORTO, T. S. *et al.* Brittleness index and its relationship with materials mechanical properties: Influence on the machinability of CAD/CAM materials. **Brazilian Oral Research**, v. 33, p. e026, 2019.

PRADO, M. *et al.* Ceramic surface treatment with a single-component primer: Resin adhesion to glass ceramics. **The Journal of Adhesive Dentistry**, v. 20, n. 2, p. 99–105, 2018.

RAMAKRISHNAIAH, R. *et al.* The effect of hydrofluoric acid etching duration on the surface micromorphology, roughness, and wettability of dental ceramics. **International Journal of Molecular Sciences**, v. 17, n. 6, p. 822, 2016.

ROMÁN-RODRÍGUEZ, J. L. *et al.* Bonding to silicate ceramics: Conventional technique compared with a simplified technique. **Journal of Clinical and Experimental Dentistry**, v. 9, n. 3, p. e384–e386, 2017.

SCHERER, M. M. *et al.* Fatigue failure load of an adhesively-cemented lithium disilicate glass-ceramic: Conventional ceramic etching vs etch & prime one-step primer. **Dental Materials**, v. 34, n. 8, p. 1134–1143, 2018.

SIQUEIRA, F. S. F. *et al.* New single-bottle ceramic primer: 6-month case report and laboratory performance. **Journal of Contemporary Dental Practice**, v. 17, n. 12, p. 1033–1039, 2016.

SOUZA, K. B. *et al.* Effect of different surface treatments and multimode adhesive application on the Weibull characteristics, wettability, surface topography and adhesion to CAD/CAM lithium disilicate ceramic. **Journal of Applied Oral Science**, v. 28, p. 1–12, 2020.

STRASSER, T. *et al.* Roughness, surface energy, and superficial damages of CAD/CAM materials after surface treatment. **Clinical Oral Investigations**, v. 22, n. 8, p. 2787–2797, 2018.

SUDRÉ, J. P. *et al.* Influence of surface treatment of lithium disilicate on roughness and bond strength. **International Journal of Prosthodontics**, v. 33, n. 2, p. 212–216, 2020.

SUNDFELD NETO, D. *et al.* The effect of hydrofluoric acid concentration on the bond strength and morphology of the surface and interface of glass ceramics to a resin cement. **Operative Dentistry**, v. 40, n. 5, p. 470–479, 2015.

THIESEN, K. P. P. R. *et al.* Bond strength to ZLS ceramics at different etching times and cementation protocols after aging. **Brazilian Dental Science**, v. 22, n. 4, p. 488–496, 2019.

TRIBST, J. P. M. *et al.* Self-etching primers vs acid conditioning: Impact on bond strength between ceramics and resin cement. **Operative Dentistry**, v. 43, n. 4, p. 372–379, 2018.

TURUNÇ OĞUZMAN, R.; ŞİŞMANOĞLU, S. Adhesive performance of resin cement to glass-ceramic and polymer-based ceramic CAD/CAM materials after applying self-etching ceramic primer or different surface treatments. **Materials**, v. 17, n. 1, p. 2, 2024.

TURUNÇ-OĞUZMAN, R.; ŞİŞMANOĞLU, S. Influence of surface treatments and adhesive protocols on repair bond strength of glass-matrix and resin-matrix CAD/CAM ceramics. **Journal of Esthetic and Restorative Dentistry**, v. 35, n. 8, p. 1322–1331, 2023.

VENTURINI, A. B. *et al.* Influence of hydrofluoric acid concentration on the flexural strength of a feldspathic ceramic. **Journal of the Mechanical Behavior of Biomedical Materials**, v. 48, p. 241–248, 2015.

VICHI, A. *et al.* Comparison between hydrofluoric acid and single-component primer as conditioners on resin cement adhesion to lithium silicate and lithium disilicate glass ceramics. **Materials**, v. 14, n. 22, p. 6776, 2021.

VILA-NOVA, T. E. L. *et al.* Effect of adhesive resin application on the durability of adhesion to CAD/CAM glass-ceramics after either hydrofluoric acid etching or self-etch primer application. **The Journal of Adhesive Dentistry**, v. 24, n. 1, p. 279–289, 2022.

WILLE, S.; LEHMANN, F.; KERN, M. Durability of resin bonding to lithium disilicate and zirconia ceramic using a self-etching primer. **The Journal of Adhesive Dentistry**, v. 19, n. 6, p. 491–496, 2017.

WILLE, S.; LEHMANN, F.; KERN, M. Durability of resin bonding to lithium disilicate using different self-etching and conventional ceramic primers after long-term aging. **Dental Materials**, v. 38, n. 2, p. 444–450, 2022.

ZOGHEIB, L. V *et al.* Effect of hydrofluoric acid etching duration on the roughness and flexural strength of a lithium disilicate-based glass ceramic. **Brazilian Dental Journal**, v. 22, n. 1, p. 45–50, 2011.

ZORZIN, J. *et al.* Adhesion and interfacial characterization of biomimetically texturized lithium disilicate. **International Journal of Adhesion and Adhesives**, v. 91, p. 131–141, 2019.

**ANEXO A****NORMAS DO *INTERNATIONAL JOURNAL OF ADHESION AND ADHESIVES***

<https://www.sciencedirect.com/journal/international-journal-of-adhesion-and-adhesives/publish/guide-for-authors>

## ANEXO B

### PARECER DO COMITÊ DE ÉTICA

UNIVERSIDADE FEDERAL DO  
CEARÁ - UFC



#### PARECER CONSUBSTANCIADO DO CEP

##### DADOS DO PROJETO DE PESQUISA

**Título da Pesquisa:** Efeito do tratamento superficial com primer autocondicionante na resistência adesiva de cerâmica à base de dissilicato de lítio em diferentes substratos: estudo comparativo *In vitro*

**Pesquisador:** RANIEL FERNANDES PEIXOTO

**Área Temática:**

**Versão:** 2

**CAAE:** 75438023.9.0000.5054

**Instituição Proponente:** Programa de Pós-Graduação em Odontologia

**Patrocinador Principal:** Financiamento Próprio

##### DADOS DO PARECER

**Número do Parecer:** 7.574.147

##### Apresentação do Projeto:

O objetivo da presente pesquisa é caracterizar a superfície de cerâmicas à base de dissilicato de lítio após tratamento superficial, bem como avaliar o efeito do tratamento de superfície com primer cerâmico autocondicionante (Monobond Etch e Prime e MEP) na resistência adesiva da cerâmica cimentada sobre diferentes substratos (esmalte, dentina e resina composta) com simulação de uso em longo prazo (antes e após termociclagem). Quarenta tercelros molares humanos íntegros, com ausência de manchas brancas, sulcos ou fissuras na superfície do esmalte serão selecionados para esta pesquisa. Os dentes serão obtidos de cirurgias de remoção de tercelros molares nas clínicas de Cirurgia e Clínica Integrada do curso de Odontologia da UFC. Desses dentes extraídos serão confeccionado blocos dentes. termociclagem). A pesquisa será desenvolvida em 2 etapas. Na etapa 1, vinte e dois blocos (10 x 10 x 4 mm) de cerâmica à base de dissilicato de lítio serão submetidos a 2 tratamentos (n=11; Grupo HF [ácido fluorídrico + silano] e Grupo MEP [primer autocondicionante]) para caracterização superficial, por meio dos ensaios de molhabilidade e energia de superfície (n=5), rugosidade (n=5) e microscopia eletrônica de varredura (MEV, n=1). Na etapa 2 realizar-se-á a avaliação da resistência adesiva. Para tanto, quarenta tercelros molares humanos recém extraídos serão selecionados para obtenção de blocos (10 x 10 x 4 mm) com superfície plana em esmalte (n=20, Grupo E) e dentina (n=20, Grupo D). Adicionalmente, 20

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