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Calebe de Melo

**Efeito do tratamento de superfície do etileno acetato de vinila  
na delaminação de protetores bucais customizados**

*Effect of surface treatment of ethylene vinyl acetate on the delamination of  
custom-fitted mouthguards*

Dissertação apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia como requisito parcial para obtenção do Título de Mestre em Odontologia na área de Clínica Odontológica Integrada.

Uberlândia, 2023

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Orientador: Prof. Dr. Carlos José Soares

***Banca Examinadora:***

Prof. Dr. Carlos José Soares, UFU

Prof. Dr. Hugo Lemes Carlo, UFU

Prof. Dr. Paulo Nelson Filho, FORP-USP

***Suplentes:***

Prof. Dr. Luís Henrique Araújo Raposo, UFU

Prof. Dr. Júlio Cesar Franco Almeida, UNB



**UNIVERSIDADE FEDERAL DE UBERLÂNDIA**  
 Coordenação do Programa de Pós-Graduação em Odontologia  
 Av. Pará, 1720, Bloco 4L, Anexo B, Sala 35 - Bairro Umarama, Uberlândia-MG, CEP 38400-902  
 Telefone: (34) 3225-8115/8108 - www.ppgoufu.com - copod@umarama.ufu.br



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## DEDICATÓRIA

Dedico o meu mestrado,

A Deus, pela minha vida, pelo farto sustento, pela graça derramada e pelas incontáveis bênçãos.

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*“Deem graças em todas as circunstâncias, pois esta é a vontade de Deus para vocês  
em Cristo Jesus”*

*1 Tessalonicenses 5:18*

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# **R**ESUMO

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## RESUMO

O presente estudo investigou o efeito do tratamento de superfície do etileno acetato de vinila (EVA) na delaminação de protetores bucais personalizados. A contaminação do EVA durante a fabricação do protetor bucal pode causar delaminação. Nesse estudo foi avaliado o efeito de diferentes tratamentos de superfície do EVA no ângulo de contato, resistência da ligação laminada e capacidade de alongamento. Amostras de duas placas de EVA coladas foram preparadas (n = 30). A dureza Shore A das amostras de placas de EVA foi medida antes e após a termoplástificação. As placas de EVA foram aleatoriamente alocadas em um dos 5 grupos de tratamento de superfície: sem tratamento (controle); álcool isopropílico, 100%; clorofórmio, 99,8%; monômero de resina acrílica autopolimerizável (metacrilato, dimetacrilato de etileno glicol e iniciador químico - tipo amina); e álcool etílico, 70%. A força máxima de ruptura e o alongamento no momento da fratura foram calculados por meio de ensaio de tração axial em máquina de teste universal. O ângulo de contato na superfície do EVA foi medido usando o software ImageJ. Foi realizada microscopia eletrônica de varredura da superfície do EVA. A resistência à delaminação foi obtida dividindo a força máxima de ruptura pela área de união entre as placas de EVA. Os dados de resistência da união e alongamento máximo foram analisados por meio de ANOVA em um fator, seguido pelo teste de Tukey e o teste de Dunnett. Os dados do modo de falha foram analisados usando o teste do Qui-quadrado ( $\alpha = .05$ ). Os resultados mostraram que o tratamento da superfície do EVA influenciou significativamente a resistência da união e o alongamento máximo ( $p < .001$ ). O grupo controle apresentou maior ângulo de contato e resistência de união e alongamento máximo significativamente menores que os demais grupos ( $p < .001$ ). As amostras tratadas com monômero de resina acrílica e clorofórmio apresentaram resistência de ligação laminada e alongamento máximo semelhantes. O grupo do monômero de

resina acrílica apresentou um ângulo de contato significativamente menor ( $p < .001$ ). Todos os tratamentos apresentaram resistência de união e alongamento máximo significativamente maiores que o grupo controle. O tratamento com monômero de resina acrílica e clorofórmio apresentaram resistência de união e alongamento máximo significativamente maiores, e o grupo de monômero de resina acrílica apresentou um ângulo de contato menor que os outros grupos. Contudo, o clorofórmio deve ser evitado devido aos seus efeitos prejudiciais.

**PALAVRAS-CHAVE:** protetores bucais personalizados, etileno acetato de vinila, tratamento de superfície, delaminação, resistência da ligação laminada, capacidade de alongamento.

# **A**bstract

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## ABSTRACT

The present study investigated the effect of surface treatment of ethylene vinyl acetate (EVA) on the delamination of custom-fitted mouthguards. Contamination of EVA during mouthguard fabrication can cause delamination. We evaluated the effects of different EVA surface treatments on the contact angle, laminate bond strength, and elongation capacity. Specimens of two bonded EVA plates were prepared ( $n = 30$ ). The Shore A hardness of standardized EVA plate specimens was measured before and after thermoplasticization. The EVA plates were randomly allocated to one of five different surface treatment groups: no treatment (control); isopropyl alcohol, 100%; chloroform, 99.8%; self-cure acrylic resin monomer (methacrylate, ethylene glycol dimethacrylate, and chemical initiator—amine type); and ethyl alcohol, 70%. The maximum breaking force and elongation at the site of fracture were recorded using a universal testing machine. The contact angle surface was measured using ImageJ software. Scanning electron microscopy of the EVA surface was performed. The laminate bond strength was obtained by dividing the maximum breaking force by the bonding area between the two EVA plates. The laminate bond strength and maximum elongation data were analyzed by one-way ANOVA, followed by the Tukey's and the Dunnett test. The failure mode data was analyzed using the chi-square test ( $\alpha = .05$ ). Results showed that EVA surface treatment significantly influenced the laminate bond strength and maximum elongation ( $p < .001$ ). The control group had a higher contact angle and significantly lower laminate bond strength and maximum elongation than the other groups ( $p < .001$ ). The acrylic resin monomer and chloroform-treated specimens had similar laminate bond strength and maximum elongation. The acrylic resin monomer group had a significantly lower contact angle ( $p < .001$ ). All treatments had a significantly higher laminate bond strength and maximum elongation than

the control group. The acrylic resin monomer and chloroform groups had a significantly higher laminate bond strength and maximum elongation, and the acrylic resin monomer group had a lower contact angle than the other groups. Chloroform should be avoided due to its hazardous effects.

**KEYWORDS:** custom-fitted mouthguards, ethylene vinyl acetate, surface treatment, delamination, laminate bond strength, elongation capacity.



# **I**ntrodução e Referencial teórico

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## 1. INTRODUÇÃO E REFERENCIAL TEÓRICO

Os protetores bucais personalizados desempenham papel importante na prática de esportes de contato, atuando como barreira de proteção essencial contra lesões orofaciais (Sousa *et al.*, 2020). Esses dispositivos, fabricados a partir do material termoplástico etileno acetato de vinila (EVA), são projetados para equilibrar características como flexibilidade, resistência ao impacto e conforto, contribuindo para prática esportiva mais segura (Soares *et al.*, 2020; Sousa *et al.*, 2020). A espessura do protetor bucal desempenha papel crucial na prevenção de danos causados pelos dentes durante o impacto (Newsome *et al.*, 2001).

Um estudo recente avaliou a importância do efeito protetivo de protetores bucais à facetas em resina composta (de Bragança *et al.*, 2023). O estudo mostrou que protetores bucais reduzem tensões e proporcionam absorção de choque, independentemente do tipo de protetor testado se com inserção de ar ou de estrutura rígida. Isso demonstra que os protetores bucais desempenham papel importante na proteção de restaurações estéticas contra danos causados por impactos da mesma forma que proporcionam proteção à dentes hígidos e estruturas de suporte (Firmiano *et al.*, 2019). Pacientes que possuem implantes instalados na região anterior podem estar sujeitos a traumas severo durante a prática esportiva, indicando a indicação de protetores bucais personalizados (Carvalho *et al.*, 2018). Da mesma forma, pacientes que possuem aparelhos ortodônticos, muito frequente em praticantes de esportes, devem estar atentos ao uso de protetores bucais, sendo os customizados os mais indicados para prevenir danos aos dentes e tecidos moles (Alves *et al.*, 2020).

No entanto, um desafio significativo na fabricação dos protetores bucais personalizados é a ocorrência de delaminação precoce (Tanabe *et al.*, 2020). Este fenômeno

compromete a eficácia do dispositivo e pode levar a falhas prematuras, reduzindo a capacidade protetiva e levando ao abandono do uso dos mesmos (Takeda *et al.*, 2006). A delaminação ocorre quando as camadas de EVA se separam, resultando na perda da integridade estrutural e da capacidade de proteção do protetor bucal (Tanabe *et al.*, 2020). Quando plastificado o EVA reduz significativamente a espessura (Veríssimo *et al.*, 2016; ). Com a necessidade de confecção de protetores bucais customizados com espessuras de aproximadamente 4,0 mm de espessura (Veríssimo *et al.*, 2016), há a necessidade de plastificação de duas lâminas de EVA, devido a dificuldade de se encontrar placas de EVA de 5 mm de espessura. Com isso, após a plastificação da primeira lâmina de EVA são realizados recorte próximo ao limite do fundo de vestibulo e acabamento de bordas (Takahashi & Bando, 2023), gerando resíduos e necessidade de manipulação (Veríssimo *et al.*, 2016; Carvalho *et al.*, 2018; Bragança *et al.*, 2023). Diversos fatores podem contribuir para a delaminação, sendo a contaminação do EVA durante o processo de fabricação um dos principais. A presença de impurezas, partículas ou substâncias estranhas entre as camadas de EVA pode interferir na adesão adequada entre elas, levando à delaminação do dispositivo (Maroosis, 1999).

Para a adequada laminação de placas de EVA a temperatura (Tanabe *et al.*, 2020), e o tratamento de superfície podem interferir gerando interfaces estáveis e bem aderidas. Temperatura de plastificação não inferiores a 120 °C é indicada para definir coreta plastificação do EVA (Tanabe *et al.*, 2020). Para o tratamento de superfície, diversos protocolos têm sido indicados com uso de aquecimento, e de soluções detergentes ou solventes aplicados sobre a placa de EVA (Ihara *et al.*, 2009). Uma técnica comumente empregada para melhorar a adesão entre as camadas de EVA é o tratamento da superfície

com clorofórmio (Ihara *et al.*, 2009). Esta abordagem visa promover a interação molecular entre as camadas, aumentando a aderência e reduzindo a probabilidade de delaminação. No entanto, é importante notar que a aquisição de clorofórmio é estritamente regulamentada em muitos países devido aos seus efeitos nocivos à saúde humana e ao meio ambiente (Smith & Johnson, 2023). Portanto, a aplicação deste tratamento pode apresentar desafios logísticos e regulatórios.

A delaminação pode levar a outros aspectos podem afetar a qualidade e desempenho dos protetores bucais personalizados. A exposição a diferentes fluidos, como bebidas esportivas e saliva, pode causar alterações na cor e dureza do material (Mańka-Malara *et al.*, 2023). No entanto, estudos demonstram que essas mudanças não são estatisticamente significativas e geralmente não comprometem a funcionalidade do protetor bucal, quando comparadas aos problemas associados à delaminação e falta de higienização adequada, como o acúmulo de biofilme e mau odor (Mańka-Malara *et al.*, 2023; Ribeiro *et al.*, 2021). Porém a criação de nicho de acúmulo de bactérias nas interfaces geradas pela delaminação pode favorecer ao odor desagradável.

É fundamental destacar que a durabilidade e resistência ao desgaste dos protetores bucais personalizados, bem como sua higienização e manutenção adequadas, estão diretamente relacionadas ao uso feito pelo atleta (Tribst *et al.*, 2018). A fim de evitar problemas como o mau odor, é necessário considerar tanto o tratamento de superfície durante a fabricação para prevenir a delaminação precoce quanto a desinfecção regular realizada pelo paciente (Fukasawa *et al.*, 2016).

Além da proteção contra lesões orofaciais, os protetores bucais podem desempenhar

papel ergogênico na prática esportiva. A confiança na proteção contra lesões, que pode ser reforçada pelo uso de um protetor bucal, tem sido associada a melhorias no desempenho esportivo (Ageberg & Roos, 2016). Isso sugere que o uso de protetores bucais pode potencialmente melhorar a postura, relaxar os músculos e aumentar o desempenho geral, tornando o protetor bucal ainda mais funcional e recomendado para todos os esportes. No entanto, a pesquisa nesta área ainda é limitada e mais estudos são necessários para confirmar e entender melhor esses efeitos ergogênicos dos protetores bucais.

Nesse contexto, a análise da interface entre as camadas de EVA desempenha papel crucial na compreensão dos mecanismos envolvidos na delaminação, nos efeitos do tratamento de superfície nas propriedades mecânicas e adesivas das placas, bem como nas estratégias para prevenir a delaminação precoce. Portanto evidencia-se a relevância de se estudar protocolos de tratamento mais acessíveis, menos tóxicos aos profissionais e com consequente maior efetividade na performance dos protetores bucais cosntumizados. A realização de pesquisas contínuas nessas áreas é essencial para aprimorar a eficácia, durabilidade e segurança desses dispositivos, que são de grande importância na proteção da saúde bucal durante a prática esportiva.

# Capítulo

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## 2. CAPÍTULO 1

### ARTIGO 1





Effect of surface treatment of ethylene vinyl acetate on the delamination of custom-fitted mouthguards

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## ORIGINAL ARTICLE

# Effect of surface treatment of ethylene vinyl acetate on the delamination of custom-fitted mouthguards

Calebe de Melo<sup>1</sup>  | Júlia Borges Resende<sup>1</sup>  | Maribí Isomar Terán Lozada<sup>1</sup>  |  
Lilibeth Carola Leyton Mendoza<sup>1</sup>  | Maria Tereza Hordones Ribeiro<sup>1</sup>  |  
Priscila Barbosa Ferreira Soares<sup>2</sup>  | Carlos José Soares<sup>3</sup> 

<sup>1</sup>School of Dentistry, Federal University of Uberlândia, Uberlândia, Brazil

<sup>2</sup>Department of Periodontology and Implantology, School of Dentistry, Universidade de Uberlândia, Uberlândia, Brazil

<sup>3</sup>Department of Operative Dentistry and Dental Materials, School of Dentistry, Universidade de Uberlândia, Uberlândia, Brazil

## Correspondence

Carlos José Soares, School of Dentistry, Federal University of Uberlândia, Avenida Pará, 1720, Bloco 4L, Anexo A, sala 42, Campos Umuarama, Uberlândia, Minas Gerais, CEP. 38400-902, Brazil.  
Email: [carlosjsoares@ufu.br](mailto:carlosjsoares@ufu.br)

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## Abstract

**Background/Aim:** Contamination of ethylene vinyl acetate (EVA) during mouthguard fabrication can cause delamination. The study evaluated the effects of different EVA surface treatments on the contact angle, laminate bond strength, and elongation capacity.

**Materials and Methods:** Specimens of two bonded EVA plates were prepared ( $n = 30$ ). The Shore A hardness of standardized EVA plate specimens was measured before and after thermo-plasticization. The EVA plates were randomly allocated to one of five different surface treatment groups: no treatment (control); isopropyl alcohol, 100%; chloroform, 99.8%; self-cure acrylic resin monomer (methacrylate, ethylene glycol dimethacrylate, and chemical initiator—amine type); and ethyl alcohol, 70%. The maximum breaking force and elongation at the site of fracture were recorded using a universal testing machine. The contact angle surface was measured using ImageJ software. Scanning electron microscopy of the EVA surface was performed. The laminate bond strength was obtained by dividing the maximum breaking force by the bonding area between the two EVA plates. The laminate bond strength and maximum elongation data were analyzed by one-way ANOVA, followed by the Tukey's and the Dunnett test. The failure mode data was analyzed using the chi-square test ( $\alpha = .05$ ).

**Results:** EVA surface treatment significantly influenced the laminate bond strength and maximum elongation ( $p < .001$ ). The control group had a higher contact angle and significantly lower laminate bond strength and maximum elongation than the other groups ( $p < .001$ ). The acrylic resin monomer and chloroform-treated specimens had similar laminate bond strength and maximum elongation. The acrylic resin monomer group had a significantly lower contact angle ( $p < .001$ ).

**Conclusions:** All treatments had a significantly higher laminate bond strength and maximum elongation than the control group. The acrylic resin monomer and chloroform groups had a significantly higher laminate bond strength and maximum elongation and the acrylic resin monomer group had a lower contact angle than the other groups. The chloroform should be avoided due its hazardous effects.



KEYWORDS

delamination, dentoalveolar trauma, ethylene vinyl acetate, mouthguard, surface treatment

1 | INTRODUCTION

Contact sports carry an increased risk of dental trauma, accounting for 39% of all dental injuries in children.<sup>1</sup> Further, the incidence of dental trauma is up to 73.7% in boxing, 41.2% in jiu-jitsu, 37.1% in handball, 36.4% in basketball, and 22.3% in soccer.<sup>2</sup> Dental trauma is recognized as one of the most prevalent injuries/diseases in the world affecting more than one billion people.<sup>3</sup> Orofacial injuries may involve tooth fracture, avulsion, lateral luxation, soft tissue laceration, and temporomandibular joint damage.<sup>4,6</sup> This type of complication represents 18% of all injuries during sports practices, with 50% directly affecting the teeth.<sup>7</sup> Tooth protrusion and lack of lip sealing double the risk of dental trauma,<sup>8</sup> with the maxillary anterior teeth, especially the central incisors, being the most affected due to their position in the dental arch.<sup>5,9,10</sup>

The use of a custom-fit mouthguard made with ethylene vinyl acetate (EVA), which can absorb and dissipate the stress and deformation caused by dental trauma, is recommended to protect the teeth and periodontal tissues.<sup>5</sup> According to the American Society of Testing and Materials, a custom-fit mouthguard is the most recommended in comparison with other mouthguards due to its ability to provide greater impact protection and better adaptation. Additionally, custom-fit mouthguards of appropriate thickness do not significantly interfere with breathing, speech, and thereby the athletic performance.<sup>11-13</sup> The efficiency of mouthguards is also related to the type of EVA used and its physical and mechanical properties.<sup>13</sup> According to the American National Standards Institute,<sup>14</sup> EVA must have low water absorption, adequate hardness, impact resistance, and low incidence of delamination.<sup>12,15</sup> Therefore, to optimize impact absorption, the use of two thermoforming plates is recommended to obtain the ideal thickness of 4 mm.<sup>13,16</sup>

Mouthguards must be replaced periodically due to their deterioration or permanent deformation, which reduces their protection capacity and intraoral stability.<sup>17</sup> Extended use of mouthguards can cause eventual delamination of the EVA plates,<sup>18,19</sup> leading to contamination and increased water sorption.<sup>12,20</sup> This can decrease the performance, quality, and durability of the mouthguard and reduce its protection capacity.<sup>19</sup> Early delamination can occur due to errors in the mouthguard fabrication process.<sup>18,21</sup> The plasticization of the first EVA plate is followed by cutting and edge wear, which contaminate the EVA surface coming in contact with the second EVA plate.<sup>12</sup>

The EVA surface treatment before plasticization is not well established, and few recommendations are provided by EVA

manufacturers.<sup>12,22</sup> Studies have recommended heating of the first EVA plate,<sup>23</sup> application of chloroform, and even no surface treatment.<sup>12,23</sup> The surface treatment can improve the bond strength between EVA plates, which reduces contamination of the delamination and interface surfaces.<sup>24,25</sup> Measuring the bond strength between the double layers of EVA mouthguards has previously been performed using a standardized specimen shape following ISO 37-2017.<sup>24,25</sup>

There is therefore a lack of a standardized protocol for the treatment of the EVA surface. Some surface treatment agents for EVA are already available, such as Mouth Guard Fitter (YAMAHACHI DENTAL MFG., Japan) and ENTFETTER Dressing Agent (ERKODENT, Germany), but they have not been fully evaluated, nor are they available in all marketplaces. Considering that the leading manufacturers of EVA have provided limited specific surface treatment guidelines for their plates, studies on EVA surface treatment are necessary to improve the quality of the fabricated mouthguards. Therefore, the aim of this study was to evaluate the effects of different surface treatment protocols on the double-layer EVA mouthguard based on the contact angle, scanning electronic microscopy (SEM) analysis, and laminate bond strength and elongation between two EVA plates used for mouthguard fabrication. The null hypothesis was that the surface EVA treatment does not influence the biomechanical interaction of the thermos-formed EVA used for mouthguard fabrication.

2 | MATERIALS AND METHODS

Soft colored circular EVA plates (Bio-Art Dental Equipment) with a 15-mm diameter and 3-mm thickness (Table 1) were used in pairs to obtain specimens with a final thickness of 4 mm ( $n = 30$ ). The thickness of the EVA plates was measured using a digital caliper (Mitutoyo) at three sites in each quadrant before and after plasticization.

Shore A hardness (Model CV06-113, CV Instruments Europe BV) of the standard EVA plates ( $n = 30$ ) was measured before and after plasticization. The indenter was applied perpendicular at three sites of the plate without shock, and the hardness was recorded 10 s after applying the 10 N load.<sup>21,26</sup> The mean Shore A hardness value for each plate was considered an experimental unit.

The first EVA plate was heated in a vacuum plasticizer (PlastiVac P7, Bio-Art) for 150 s. Using an infrared thermometer (MT 395A, Minipa), the temperature of the plasticization was recorded at 150 s as  $130 \pm 2^\circ\text{C}$ . A rectangular metal model measuring  $75 \times 70$  mm with

TABLE 1 EVA plate materials used.

Shape	Type	Dimension	Batch numbers	Manufacture
Circular	Soft plate mixed colors	Diameter—130 mm, Thickness—3.0 mm	54,492/51,901	Bioart, São Carlos, SP, Brazil

two central holes of 5 mm diameter was used for EVA plasticization during the specimen's fabrication. All specimens were prepared by vacuum forming for 20 s in accordance with the manufacturer's recommendation. The first EVA plate was allowed to cool for 15 min at room temperature and was then separated from the metal plate. The standardized finishing time for the first EVA plate was 1 min. Edge-cutting was performed using a silicon carbide stone drill (DHPPro), and finishing and polishing were performed using rubber points (G2052F, DHPPro) with a low-speed handpiece. An air spray was applied on the plate surface for 10 s as the final treatment.

A bonding area of 15 mm × 70 mm was delimited using a digital caliper (Mitutoyo). One of the following five surface treatments was applied on the limited bonding area of the EVA plate ( $n = 15$ , Table 2):

1. no treatment (NoT): no treatment of the standardized specimens.
2. isopropyl alcohol (IsoAlc): active application of isopropyl alcohol 100% (Quality).
3. chloroform (Chlo): active application of chloroform 99.8% (Alphatec).
4. acrylic resin monomer (AcRm): active application of resin monomer VIPI Flash (VIPI Odonto Products, Pirassununga)—a solution of metal methacrylate, ethylene glycol dimethacrylate, and chemical initiator (amine type).
5. 70% alcohol (70Alc): active application of 70% ethyl alcohol and 30% distilled water (Ciclo Farma).

On the first EVA plate, two pieces of baking paper were placed to cover both sides of the centralized bonding treated area. No hand contact was performed on the treated bonding area. The second plate was plasticized for 150 s at  $130 \pm 2^\circ\text{C}$ , and bonded over the first EVA plate and stored at room temperature ( $23 \pm 2^\circ\text{C}$ ; Figure 1).

Measurement of the contact angle was performed on a standardized image obtained by a digital camera (DXM-I200; Nikon). The distilled water drop was deposited over the EVA specimen at a controlled temperature ( $23 \pm 2^\circ\text{C}$ ). The volume of the sessile drop was maintained at 5  $\mu\text{L}$  using a micro pipette (Labmate Soft; HTL Lab Solutions). The contact angle after 60 s of placing the water drop was recorded. Five samples were tested in each group. The images were used to calculate the water contact angle with the EVA plate

following different surface treatments using a public domain software (ImageJ, National Institutes of Health; Figure 2).

The treated surface EVA plate was fixed on to a metal stub and coated with a thin layer of gold (QR 150ES, Quorum). The surface was analyzed under an SEM (VEGA 3 LMU, Tescan) with 60× and 1000× magnification.

After 24 h of preparing the specimens with the two EVA plates, they were cut with an ISO 9001 certified hand pressure cutting machine (SOMEH Projects Products and Services) using a dumbbell knife producing specimens according to ISO 37-2017.<sup>25</sup> The bonding area was measured using a digital caliper (Mitutoyo) and cut in half with sharp scissors. The width and length of the bonding area in each specimen was measured with a digital caliper (Mitutoyo). The rectangular bonding area was standardized at  $7.5 \pm 0.7 \text{ mm} \times 4.0 \pm 0.1 \text{ mm}$ .<sup>21</sup>

The specimens were attached to two pneumatic clamps (2712 Series Pneumatic Action Grips, Instron Corporation, Norwood, MA, USA), and were subjected to a tensile strength test using 50 mm/min crosshead speed in a universal testing machine (ElectroPuls® E3000, Instron; Figure 3). The maximum displacement (mm) and maximum fracture force (N) were recorded using a dedicated software (Blue Hill 2, Instron). The laminate bonding strength (MPa) was calculated by dividing the fracture force (N) by the bonding area ( $\text{mm}^2$ ) for each specimen.<sup>24</sup> The failure mode was classified after the test by visual analysis according to the following six levels (Figure 4):

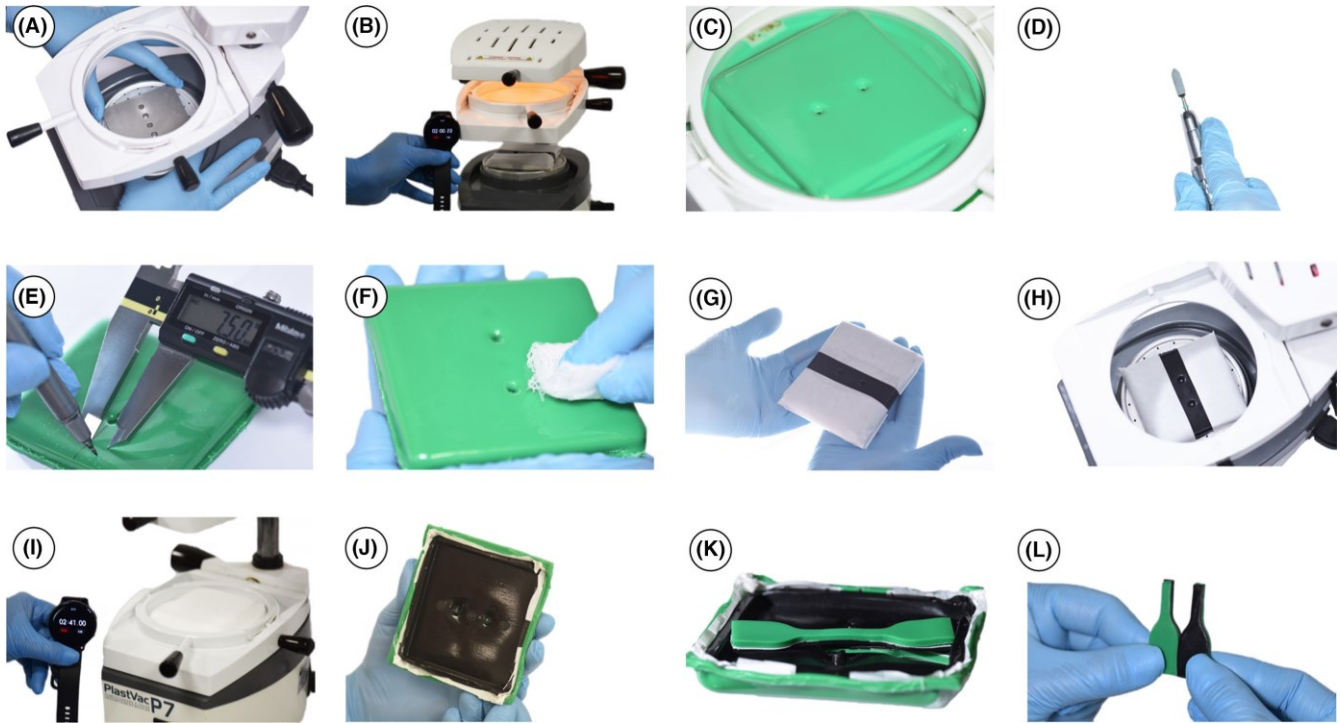
- I No fracture;
- II Adhesive fracture at the bonded area;
- III Cohesive fracture at the first plate close to the bonded area;
- IV Cohesive fracture at the second plate close to the bonded area;
- V Cohesive fracture at the first plate far from the bonded area;
- VI Cohesive fracture at the second plate far from the bonded area.

The laminated bond strength (MPa), maximum elongation (mm), contact angle, and Shore A hardness data were tested for normal distribution (Shapiro–Wilk) and equality of variances (Levene's test). They were then analyzed by one-way analysis of variance (ANOVA) followed by the Tukey's and the Dunnett tests. Failure modes were analyzed using the chi-square test. All tests employed an  $\alpha = .05$  significance level. All analyses were carried out with the

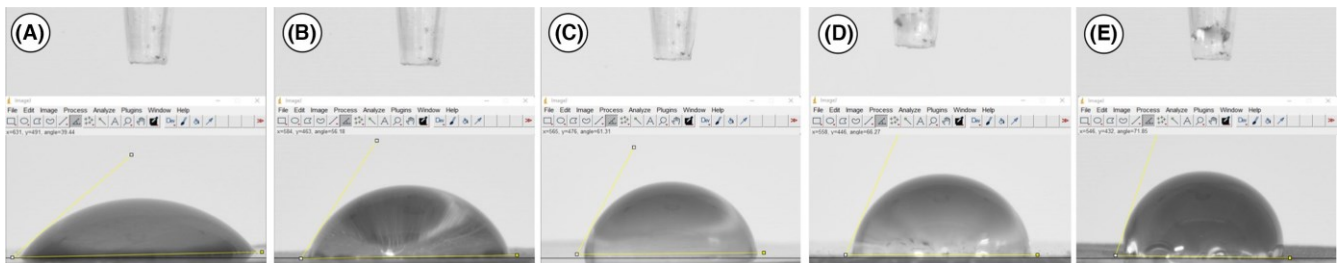
**TABLE 2** Materials used for surface EVA plates treatment materials.

Treatment	Composition	Batch number	Manufacture <sup>a</sup>
Self-curing acrylic resin monomer	Methylmethacrylate; EDMA; DMT; Inhibitor; Fluorescent	180,214	Vipiflash, Pirassununga, Brazil
100% Isopropyl alcohol	100% Isopropyl alcohol	15,748	Quality, Campo Grande, Brazil
70% alcohol	30% Distilled water; 70% Isopropyl alcohol	0036/012020	Ciclo Farma, Serrana, Brazil
99.8% Chloroform	99.8% Chloroform; 0.2% Distilled water	25,270	Alphatec, Cajuru, Brazil

<sup>a</sup>Information provided by manufacturers.



**FIGURE 1** Specimen fabrication: (A) Metal model positioned in vacuum plasticizer; (B) EVA plate heating for 2 min; (C) Metal plate covered by the first plasticized EVA plate; (D) Silicon carbide stone drill for EVA preparation; (E) Bonding area measurement; (F) Surface treatment; (G) First plate wrapped with baking paper delimiting adhesion area; (H) Wrapped plate positioned in vacuum laminator; (I) Heating and lamination of the second plate with a cooling time of 20 min; (J) Final aspect of the two laminated plates; (K) Making a dumbbell cut; (L) Final aspect of the specimen.



**FIGURE 2** Contact water angle ( $\theta$ ) analysis: (A) acrylic resin monomer; (B) chloroform; (C) 70% alcohol; (D) isopropyl alcohol; (E) no treatment (control).

statistical package Sigma Plot version 13.1. The SEM images were analyzed qualitatively.

### 3 | RESULTS

The Shore A hardness and thickness (mm) of the specimens are presented in Table 3. One-way ANOVA showed no significant effect on the Shore A hardness of EVA before and after plasticization. The EVA thickness reduced after plasticization from two plates of 3.0 mm each to a final thickness of 4.5 mm (Table 3).

Representative contact angle images are shown in Figure 2, and their values are presented in Table 4. The Dunnet test showed that all surface treatment groups had lower contact angles than that in the control group. The Tukey test showed that the AcRm group

had a significantly lower contact angle than that in all other groups ( $p < .001$ ).

The representative SEM images of the EVA surfaces of all tested groups are shown in Figure 5. The AcRm and IsoAlC groups showed a molten surface, and the 70Alc and Chlo groups showed scratches on their surface. The control group showed a smooth surface with debris generated during peripheral EVA preparation.

The laminate bond strength (MPa) of all EVA treatment groups is shown in Table 4. The Dunnet test showed that all the tested surfaces had significantly higher laminate bond strength than the control group ( $p < .005$ ). ANOVA showed significant difference between the experimental groups. The laminate bond strength of the AcRm group was similar to that of the Chlo group and significantly higher than that of the 70Alc and IsoAlc groups. The mean laminate bond strength of the Chlo group was similar to that of the 70Alc group

and higher than that of the IsoAlc group. No significant difference between the 70Alc and IsoAlc groups was found.

The maximum elongation (mm) following all EVA treatment surfaces is shown in Table 4. The Dunnet test showed that the AcRm, Chlo, and 70Alc groups had a significantly higher maximum

elongation than the control group ( $p < .001$ ). The Tukey test showed that the AcRm and Chlo groups had similar maximal elongation, significantly higher than that of the IsoAlc group. The maximum elongation in the IsoAlc group was similar to that in the 70Alc group.

The failure mode distributions for different surface experimental and control group are shown in Figure 6. Adhesive failure was the most frequent failure mode regardless of the surface treatment performed. The failure pattern was not influenced by the surface treatment.

#### 4 | DISCUSSION

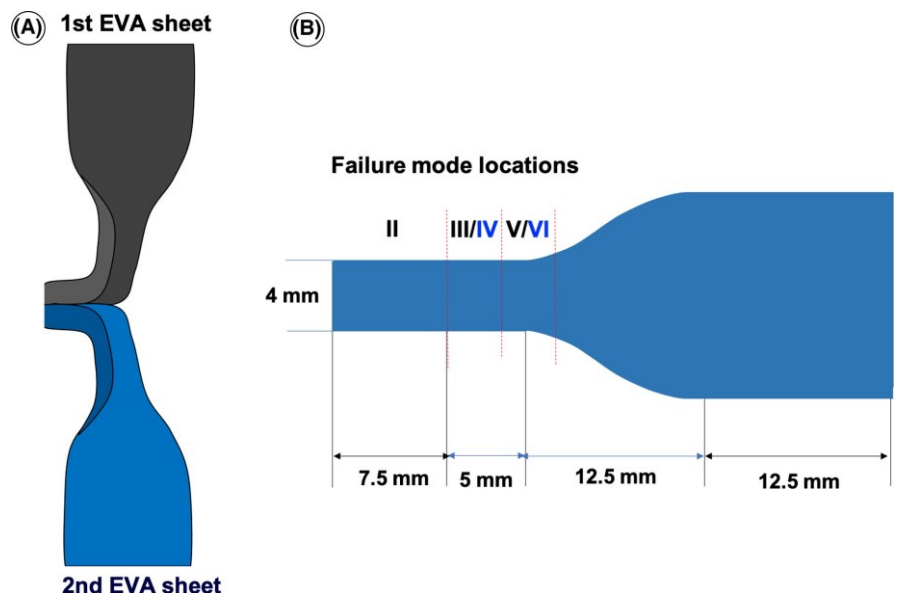
The EVA plate surface treatments significantly influenced the laminate bond strength, specimen elongation, and contact angles of the specimens. Therefore, the null hypothesis was rejected. All the experimental surface treatments showed significantly greater laminate bond strength and elongation and a lower contact angle of the EVA surface.

This study involved the characterization of the EVA surface, mechanical alteration, and the bonding interaction between the double-layer EVA. This comprehensive analysis using complementary tests could explain the benefit of the different products applied not only for bonding two EVA plates but also to show the effect of the products used on the performance of the final mouthguard.<sup>24,27</sup> The tensile strength test, which was used to calculate the bond strength, and the elongation of the EVA associated with failure mode were used to differentiate the surface protocols for better interaction of the double layer EVA mouthguard.<sup>12,25</sup> Appropriate SEM techniques, which adhered to fundamental physical and chemical test protocols, were used to elucidate morphological features involved in EVA mouthguard fabrication.<sup>28</sup> The hardness of EVA influences the form, retention, occlusal support, shock absorption, and durability of



**FIGURE 3** Laminate bond strength test using a universal test machine with a pneumatic grip for fixation of the ethyl vinyl acetate specimen.

**FIGURE 4** (A) Laminate bond strength specimen; (B) Failure locations after test for characterization of failure modes.



the mouthguards.<sup>29</sup> Characterization of the possible effects on the hardness of EVA is also important to assess the final performance of mouthguards.

The EVA material can influence the biomechanical performance of a mouthguard.<sup>29</sup> The EVA material used in this study had an elastic modulus of approximately 35 MPa.<sup>29</sup> A questionnaire study reported that 73.2% of mouthguard users had difficulty wearing it.<sup>30</sup> The unpleasant taste and smell frequently caused by delamination accounted for the reluctance in wearing a mouthguard among 20.7% of adolescents.<sup>30</sup> Creating an effective EVA seal between the plates can help avoid delamination, increase the durability of the mouthguard, and facilitate its use.

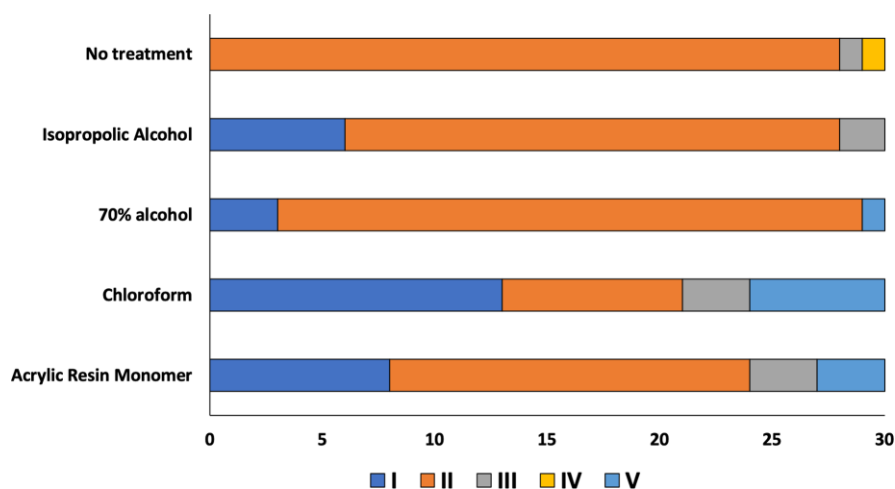
**TABLE 3** Thickness and Shore A mean and standard deviation values for EVA plates before and after thermo-plasticization.

EVA plates	Thickness (mm)	Shore A hardness
EVA plates—before plasticization	3.0 ± 0.1	82.9 ± 0.6 A
First EVA plate after plasticization	2.2 ± 0.4	79.3 ± 6.6 A
Bonding area—2 EVA plates	4.5 ± 0.5	79.9 ± 3.0 A

Note: Different letters mean significant difference among tested groups.

EVA surface treatments	Contact angle (θ)	Laminated bond strength (MPa)	Maximal elongation (mm)
NoT, no treatment (control)	72.5 ± 1.8	28.8 ± 7.1	0.98 ± 0.50
AcRM, acrylic resin monomer	39.2 ± 4.7 A*	44.1 ± 5.8 A*	1.50 ± 0.50 A*
Chlo, 99.8% chloroform	54.0 ± 5.3 B*	43.8 ± 7.0 A*	1.35 ± 0.50 AB*
70Alc, 70% alcohol	62.9 ± 1.2 C*	37.1 ± 5.4 AB*	1.26 ± 0.50 BC*
IsoAlc, 100% Isopropyl alcohol	66.3 ± 1.6 C	34.7 ± 5.8 B	1.12 ± 0.50 C

Note: Different letters mean significant difference among tested experiment protocols (Tukey test,  $p < .05$ ); \* means significant difference between each experimental tested protocol and control group (Dunnet test,  $p < .05$ ).

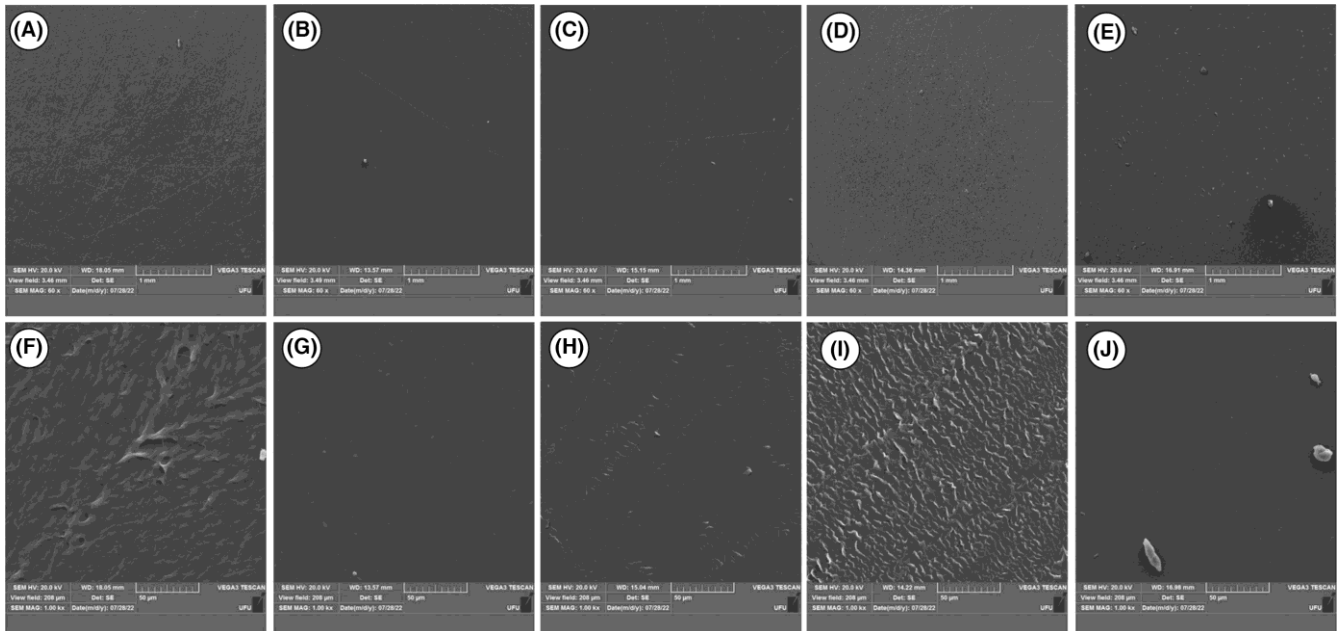


**TABLE 4** Contact water angle (θ), Laminated bond strength (MPa), and maximum elongation (mm) of the EVA submitted to different surface treatments.

**FIGURE 5** Failure mode distribution of different surface treatments: (I) no fracture between the plates; (II) adhesive rupture in the bonded area; (III) fracture predominantly on the first plate in the bonded area; (IV) fracture predominantly on the second plate in the bonded area; (V) fracture predominantly on the first plate outside the bonded area.

A plate thickness of 3–4 mm is recommended for custom-fit mouthguards due the protective effects of the plate, including lowered stress and strain on teeth during trauma impact.<sup>13</sup> When the 3.0-mm EVA plates were plasticized, their thickness reduced significantly. The use of two laminated EVA plates is therefore essential for custom-fit mouthguard fabrication.<sup>13</sup> The first EVA plate is thermoformed and needs to be cut and prepared using a bur, abrasive rubber and brushes to adapt the EVA to the custom-fit mouthguard area.<sup>13,18</sup> Glove powder, oils, and residues can contaminate the surface of the first EVA plate during its manipulation. The union between two materials is due to the interaction between their molecules. Intermolecular forces help the first EVA plate bond with the second one.<sup>20,31</sup> This adhesion is mainly affected by the surface energy of EVA. A reduction in interfacial tension or interfacial energy results in stronger attraction forces and interactions between different materials.<sup>30</sup> Lower contact angles are associated with better bonding interaction.<sup>32</sup> The use of acrylic resin monomer reduced the contact angle, creating a more reactive EVA surface than that in the other groups. This can further explain the higher laminate bond strength in these groups.

Chloroform is already indicated for cleaning and treatment of EVA plates.<sup>12,23,31</sup> However, its production needs specific license requirements, making it relatively inaccessible for clinicians.<sup>33</sup> The inhalation of chloroform vapors, which have also been found to be



**FIGURE 6** Scanning electron microscopy surface images of all tested groups. Images with 60× magnification: (A) self-cure acrylic resin monomer; (B) chloroform, 99.8%; (C) ethyl alcohol, 70%; (D) isopropyl alcohol, 100%; (E) no treatment (control); Images with 1000× magnification: (F) self-cure acrylic resin monomer; (G) chloroform, 99.8%; (H) ethyl alcohol, 70%; (I) isopropyl alcohol, 100%; (J) no treatment (control).

cytotoxic, may lead to symptoms such as shortness of breath and dryness of the mouth and throat.<sup>34,35</sup> Other symptoms of chloroform vapor inhalation include excitement and nausea followed by dizziness and drowsiness.<sup>34,36</sup> Considering that there are alternative products with good performance to use in the EVA cleaning process, the use of chloroform should be avoided. Specific products produced for this purpose, such as ENTFETTER Dressing Agent (ERKODENT), are composed of a solution of *n*-hexane and petroleum distillate. These products also have some hazardous effects; repeated exposure may cause skin dryness, cracking, and irritation and, similar to chloroform, they may cause drowsiness or dizziness.<sup>37</sup> However, these effects can also be associated with prolonged use of acrylic resin monomer.<sup>38</sup> Occupational training should emphasize that to prevent sensitization, better work practices should be developed so that there will be no direct contact with acrylic products.<sup>38</sup> The tested EVA surface treatment analyzed in this study involved products easily available in dental offices. The acrylic resin monomer and 70% alcohol are routinely used by clinicians in private and public dental services. Although isopropyl alcohol may be more difficult to procure in dental offices, it has no controlled process for acquisition.

The higher laminate bond strength achieved with all treated EVA surfaces in comparison with the control group can be attributed to the cleansing effect of the solvents used.<sup>24,25</sup> The acrylic resin monomer seemed to be good alternative for EVA surface treatment. Its methylmethacrylate composition can interact with the EVA surface, reducing the superficial tension. The better interaction on the superficial molecules resulted in a more catalytic action, increasing plate surface energy. Distilled water present in 70% alcohol seems to reduce its effectiveness during EVA surface treatment.

The most common failure mode, regardless of the EVA treatment performed, was a rupture at the bonded area, indicating that the test used in this study was efficient for analyzing the EVA bonding mechanism. The acrylic resin monomer and chloroform treatments resulted in a loss of surface shine after becoming less hydrophobic, with a reduced contact angle, which was directly related to the improved adhesion between the EVA plates.

Clinicians should be careful during the fabrication of the mouthguard.<sup>24,29</sup> Delamination, even in a partial area of the mouthguard, can increase the oral fluid retention and consequently increase unpleasant odor.<sup>18</sup> These aspects can contribute to prolonged non-use of the mouthguard, increasing the need to replace it. Considering the accessibility of the acrylic resin monomer in dental offices and its hazardous effects, the use of this product for cleaning the thermoformed first and second EVA plates during the fabrication of custom-fit mouthguards is recommended.

The following conclusions can be drawn:

- All tested EVA surface treatments significantly reduced the contact angle of the EVA surface.
- The failure modes were predominantly adhesive for all groups and were not influenced by surface treatments.
- The laminate bond strength was significantly higher in the experimental surface treatment groups than in the control group.
- A higher laminate bond strength resulted in greater EVA elongation.
- The use of acrylic resin monomer and chloroform resulted in a higher laminate bond strength and reduced contact angle.
- The chloroform should be avoided due its hazardous effects.

**AUTHOR CONTRIBUTIONS**

Calebe de Melo contributed to literature search, pilot test development, model generation, data acquisition, data analysis, manuscript preparation, and manuscript editing. Júlia Borges Resende contributed to literature search, pilot test development, model generation, data acquisition, manuscript preparation, and manuscript editing. Maribí Isomar Terán Lozada and Lilibeth Carola Leyton Mendoza contributed to model generation and data acquisition. Maria Tereza Hordones Ribeiro contributed to pilot test development. Priscila Barbosa Ferreira Soares contributed to conception and design, manuscript revision, and data acquisition. Carlos José Soares contributed to literature search, pilot test development, conception and design, data analysis, manuscript editing, and manuscript revision. All the authors read and approved the final manuscript.

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**CONFLICT OF INTEREST STATEMENT**

The authors confirm that they have no conflict of interest.


**DATA AVAILABILITY STATEMENT**

The data that support the findings of this study are available from the corresponding author upon reasonable request.

**ORCID**

Calebe de Melo  <https://orcid.org/0000-0003-3263-5951>

Júlia Borges Resende  <https://orcid.org/0000-0002-4425-0357>

Maribí Isomar Terán Lozada  <https://orcid.org/0000-0002-7094-4571>

Lilibeth Carola Leyton Mendoza  <https://orcid.org/0000-0003-1608-4299>

Maria Tereza Hordones Ribeiro  <https://orcid.org/0000-0002-5126-7931>

Priscila Barbosa Ferreira Soares  <https://orcid.org/0000-0002-4492-8957>

Carlos José Soares  <https://orcid.org/0000-0002-8830-605X>

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# Conclusão

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## CONCLUSÃO

Podemos concluir que o tratamento na superfície do etileno acetato de vinila (EVA), material essencial na confecção de protetores bucais, é comprovadamente eficaz na melhoria da resistência de adesão, alongação e energia de superfície do EVA. Dentre os tratamentos testados, incluindo o monômero de resina acrílica autopolimerizável e o clorofórmio, demonstraram aumentar consideravelmente a resistência de adesão e a máxima alongação, além de diminuir o ângulo de contato em comparação ao grupo controle. No entanto, é importante enfatizar a necessidade de evitar o uso de clorofórmio, devido aos seus efeitos adversos e perigosos para o organismo humano.

Por meio de tratamentos de superfície adequados, é possível aprimorar a funcionalidade e a interação entre as placas de EVA, potencializando a qualidade dos protetores bucais. A continuidade das pesquisas nessa área é fundamental para a otimização do design e da funcionalidade dos protetores bucais, tornando-os ainda mais benéficos para todos os atletas, independentemente do esporte praticado.

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