UNIVERSIDADE FEDERAL DE UBERLÂNDIA
FACULDADE DE ODONTOLOGIA

JÚLIA BORGES RESENDE

Influência do tratamento de placas de EVA na delaminação de protetores bucais customizados

Influence of the treatments for ethylene-vinyl acetate on delamination of custom-fitted mouthguard

UBERLÂNDIA
2022

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Trabalho de conclusão de curso apresentado a Faculdade de Odontologia da UFU, como requisito parcial para obtenção do título de Graduado em Odontologia

Orientador: Prof. Dr. Carlos José Soares

UBERLÂNDIA
2022

AGRADECIMENTOS

Em primeiro lugar gostaria de agradecer aos meus avós, Aurora e Geraldo, por todo o apoio, carinho, auxílio e paciência por permitir morar com eles, tornando possível meu acesso e continuidade à faculdade. Agradeço também aos meus pais, Marina e Júlio, os quais me deram a vida, a força e o incentivo a persistir nessa jornada. Aos meus colegas de curso que se tornaram amigos de vida, Amanda Monise, Sara Saramago, Andressa Souza, Letícia Gondin, Ana Luisa, Isabela Felix, Thiago Peres, Carola Leyton, Calebe de Melo, aquele meu muito obrigada por terem me dado a mão nos momentos difíceis, nas pressões que a faculdade nos expõe constantemente e também nos ótimos momentos, aqueles de comemorações, gargalhadas e fofocas diárias, essa conquista da formatura é nossa! Sou eternamente grata aos ensinamentos que a Universidade Federal de Uberlândia me proporcionou, e tenho a honra de ter sido orientada por professores que admirei desde o primeiro momento, Robinson Sabino e Carlos José Soares, vocês foram minha grande inspiração ao longo destes anos. Guardarei ótimas lembranças do meu curso, principalmente, devido a presença de cada um de vocês na minha vida.

RESUMO

A delaminação precoce pode ser desenvolvida devido a falhas no processo de fabricação do protetor bucal, a delaminação das placas de Etileno Vinil Acetato (EVA) diminui o desempenho, qualidade e durabilidade dos protetores bucais. Nenhuma diretriz de tratamento é determinada pelas empresas. O objetivo foi avaliar a eficácia do tratamento de superfície na colagem de placas de EVA. Cada placa foi caracterizada medindo a espessura (mm) com paquímetro digital e a medida Shore A (in) em 3 locais diferentes antes e depois. Para cada grupo, 10 placas diferentes foram selecionadas aleatoriamente para serem submetidas a um tratamento de superfície diferente: 1 - No, sem tratamento; 2 - IsoAc, álcool Isopropílico, 3 - Chlo, clorofórmio, 4 - AcRm, monômero de resina acrílica, 5 - 70Alc, álcool 70%. Trinta corpos de prova para cada grupo de tratamento de superfície foram confeccionados e ensaiados conforme ISO 37 (2017)[1], tração (MPa) ensaiada para obter a força máxima de ruptura (N) e dividida pela área de união individual (mm²) das amostras, que também foram caracterizados quanto à sua espessura (mm) e dureza (in) para a área de adesão e as extremidades livres. Tanto para resistência de delaminação (LBS) quanto para alongamento máximo (ME), o grupo AcRm apresentou desempenho semelhante ao grupo Chlo, Chlo ao 70Alc, 70Alc ao IsoAc ($P<0,001$). A redução do ângulo de contato da água demonstrou aumento na energia superficial promovida pelo tratamento de superfície. O padrão de falha não foi definido pelo tipo de tratamento de superfície. Em conclusão, os resultados indicaram que o tratamento com monômero de resina acrílica resultou em resistência delaminação semelhante ao clorofórmio e superior aos demais métodos testados. Todos os tratamentos realizados resultaram em desempenho superior ao grupo controle definido pela ausência total de tratamento da placa.

Palavras-chaves: Trauma dentoalveolar, protetor bucal, etileno vinil acetato, tratamento de superfície, delaminação.

SUMMARY

Influence of the treatments for ethylene vinyl acetate on delamination of custom-fitted mouthguard

Influence of the treatments for ethylene vinyl acetate on delamination of custom-fitted mouthguard

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Running title: Surface treatment influence on bonding of mouthguard plates

CONFLICT OF INTEREST

The authors confirm that they have no conflict of interest.

Acknowledgements

This study was supported by grants from CAPES – Finance code 001 (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior), National Council for Scientific and Technological Development (CNPq) and FAPEMIG (Fundação de Amparo à Pesquisa de Minas Gerais). This study was carried out at the Biomechanics, Biomaterials and Cell Biology Research Centre – CPBIO at UFU (Federal University of Uberlândia).

AUTHORS CONTRIBUTIONS

Júlia Borges Resende: literature search, pilot test development, models generation, data acquisition, manuscript preparation, and manuscript editing.

Calebe de Melo: pilot test development, models generation, data acquisition, data analysis, literature search, manuscript preparation, and manuscript editing.

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Carlos José Soares: literature search, pilot test development, conception and design, data analysis, manuscript editing, and manuscript revision.

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Influence of the treatments for ethylene vinyl acetate on delamination of custom-fitted mouthguard

ABSTRACT

Background/Aims: The contamination on the ethylene vinyl acetate (EVA) during the mouthguard fabrication can generate the delamination. The EVA treatment are not well stablished. The aim was to evaluate the effect of different EVA surface treatment on the contact angle, laminated bond strength and elongation capacity.

Materials and Methods: EVA plates (Biaort) were characterized measuring the thickness (mm) using digital caliper, and the Shore A in 3 different locations before and after thermo-plasticization. The EVA plates were randomized in pars and received 5 different surface treatment protocols: NoT, No treatment (Control); IsoAc, Isopropyl Alcohol; Chlo, Chloroform; AcRm, Acrylic resin monomer; 70Alc, 70% Alcohol. The bonding plate area was standardized and after plasticization the specimens were made and tested according to ISO 37-2017 (n = 30). The maximum breaking force (N) and maximal elongation (ME, mm) were recorded at the specimen rupture using universal test machine (Instron E3000). The laminated bond strength (LBS, MPa) was obtained dividing the maximal maximum breaking force (N) by bonding area (mm^2). The failure modes were classified regarding the rupture location in 5 levels. The contact angle surface was measured using ImageJ software. The LNS and ME data were analyzed by one-way ANOVA followed by Tukey's test and Dunnet test. The failure mode data was analyzed by Chi-square test (α = 0.05).

Results: The EVA surface treatment influenced significantly the LBS and ME (P < 0.001). The control group showed higher CAN and significantly lower LBS and ME than all tested EVA surface treatment (P < 0.001). The AcRm and Chlo had similar LBS, ME and CAN values and higher than other protocols (*P* <0.001). Failure modes were not influenced by the EVA surface treatment (*P* = 0.604).

Conclusions: All tested protocols resulted in higher LBS and tended to increase the ME of the EVA than control group. The acrylic resin monomer and chloroform surface treatments resulted in lower EVA CAN and higher LBS and ME than other tested protocols.

Keywords: Dentoalveolar trauma, mouthguard, ethylene vinyl acetate, surface treatment, delamination.

INTRODUCTION

The practice of sports activities impact in the psychological well-being and physical health of the individual, community, and population being a strategy to face this pandemic due to the disorders that COVID-19 has caused.[1] High-risk sports with little or no use of sports protective material generate increased demand for emergency care.[2]

Dental trauma is recognized as one of the most prevalent injuries/diseases in the world affecting more than one billion people.[3] Orofacial injuries may involve tooth fracture, avulsion, lateral luxation, soft tissue laceration, and temporomandibular joint damage.[4-6] This type of complication represent 18% of all injuries during sports practices, with 50% directly affecting the teeth.[7] Tooth protrusion and lack of lip sealing double the occurrence of dental trauma,[8] being the maxillary anterior teeth and mainly the central incisors the most affected teeth due the positioning in the dental arch.[5,9,10]

The custom-fit mouthguard made with ethylene vinyl acetate (EVA) is recommended to protect teeth and surrounding regions due the capacity of absorbing and dissipating stress and deformations caused by dental trauma.[5] According to the American Society of Testing and Materials (ASTM) the custom-fit mouthguard is the most recommended due to its ability to provide greater impact protection and better adaptation. Additionally, they can cause less interference with breathing, speech and consequently athletic performance when they are produced in proper thickness.[11-13] The efficiency of mouthguards is also related to the type of EVA used and the appropriate physical and mechanical properties to absorb impacts.[13] Based on the American National Standards Institute (ANSI),[14] EVA must have low water absorption, adequate hardness, impact resistance and low delamination.[12,15] Therefore, to optimize impact absorption, it has been recommended the thermoforming two plates to reach the ideal thickness of 4mm.[13,16]

Mouthguards must be replaced periodically due the deterioration or permanent deformation, which reduces the protection capacity and reduces mouth stability.[17] Over time of use, delamination of EVA plates may occur,[18,19] consequently contamination and increased water sorption becomes possible.[12,20] The delamination and resultant defects can decrease the mouthguards performance, quality, and durability, reducing the protection capacity of the teeth and adjacent structures.[19] Early delamination can occur due the failures during the mouthguard fabrication process.[18,21] The plasticization of the first EVA plate is followed by cutting and edge wear that results in surface contamination EVA that will be in contact with the second layer.[12]

The EVA surface treatment aiming the plates union by plasticization is not provided by manufactures and are not well established.[12,22] Some studies have recommended the heating of the first EVA plate, (REF) other studies recommended the application of the application of alcohol or even do not perform any type of treatment.[12]+ REF This lack of protocol standardization can increase the defects in bonding plates undergo delamination. Considering that leading market companies of EVA do not determine any treatment guidelines for their plates, studies are needed to improve the quality of the mouthguard fabrication. Therefore, the aim of this study was to evaluate the effect of the EVA surface treatment performed with different solvent products on the biomechanical interaction, expressed by contact angle, scanning electronic microscopy characteristic, laminate bond strength and elongation between two EVA plates used for mouthguard fabrication. The null hypothesis was that the surface EVA treatment would not influence the biomechanical interaction of the thermos-formed EVA used for mouthguard fabrication.

METHODS AND MATERIALS

EVA thickness measurement

The soft colored circular EVA plates (Bio-Art Dental Equipment, Sao Carlos, SP, Brazil) with 15 mm in diameter and 3 mm in thickness were used in pars to obtain specimens with two EVA with a final thickness of 4mm (n = 30). The thicknesses of EVA plates were measured using a digital caliper (Mitutoyo, Kawasaki, Japan) on three locations for each quadrant before and after plasticization.

Shore A hardness of EVA

Shore A hardness (Model CV06-113, CV Instruments Europe BV) of the standards EVA plates (n = 50) were measured before and after plasticization. The indenter was applied at three locations in each plate from a vertical position without shock and read 10 second after the 10N load application.[21,23] Shore A values were recorded, and the mean values of each plate was considered an experimental unit.

EVA plates plasticization and Surface Treatment

The first EVA plate was heated in a vacuum plasticizer (PlastiVac P7, Bio-Art) for 2 minutes, a rectangular metal model measuring 75 x 70mm was made and used to standardize the EVA plasticization. Two central holes with 5mm in diameter were made in the metal model to improve the EVA adaptation generated by vacuum forming for 20 seconds, following the manufacturer's recommendation. The first EVA plate was allowed to cool for 15 minutes at room temperature then was removed from the metal plate. The preparation was standardize for all first EVA plate for 1 minute finishing time, the edge cutting was made using silicon carbide stone drill (DHPro, Paraná, Brazil), and finishing and polishing was made using rubber point (G2052F, DHPro) on the low-speed handpiece, at the end a quick air spray was applied on the plate surface.

The delimitation of the bonding area was performed with a digital caliper (Mitutoyo, Kawasaki, Japan), allowing a delimited the bonding area of 15 mm x 70 mm.

Five surface treatments were applied over the limited area of the EVA plate were applied using soaked gauze was active applied on top of the entire surface of the first EVA plate and bottom of the entire surface of the second EVA for 10 seconds:

1) No, no treatment: no treatment, only the standardized preparation.

2) IsoAc, isopropyl alcohol: active application of isopropyl alcohol 100% (Quality, Brasilia, Brazil).

3) Chlo, chloroform: active application of chloroform 99,8% (Alphatec, Cajuru, Brazil).

4) AcRm, acrylic resin monomer: active application of resin monomer Vipi Flash (VIPI Odonto Products, Pirassununga, São Paulo, Brazil);

5) 70Alc, 70% alcohol: active application of 70% alcohol (Ciclo Farma, Serrana, Brazil).

Over the first EVA plate, two pieces of baking paper were added covering the no treated surface on both sides of centralized treated surface. No hand contact was done on the treated surfaces. The second plate was thermo-formed for 2 minutes and bonded over the first EVA plate and stored at room temperature (25 $\pm$ 1 °C) (Figure 1).

EVA laminate tensile bond strength and failure mode analysis

After 24 hours the two EVA plates formed were cut with an ISO 9001 certified hand pressure cutting machine (SOMEH Projects Products and Services, Joinville, Santa Catarina, Brazil) and type dumbbell knive producing 3 dumbbell shape according to ISO 37-2017.[24] The bonding area was measured by a digital caliper (Mitutoyo, Kawasaki, Japan) and cut in half with sharp scissors. The width and length of each specimen bonding area was measured with a digital caliper (Mitutoyo). Six specimens of each bonded plates were obtained, culminating in a 7.5 ± mm x 4 ± mm rectangular bonding area.[21]

The specimens were attached to two pneumatic clamps (2712 Series Pneumatic Action Grips, Instron Corporation, Norwood, MA, USA) leaving the standardized testing area. The specimens were then subjected to a tensile strength test using50 mm/min crosshead speed in a universal testing machine (ElectroPuls® E3000, Instron). The maximum displacement (mm) and the maximum rupture force (N) at the specimen failure were recorded by dedicated software (Blue Hill 2, Instron). The laminated bonding strength (MPa) was calculated dividing the rupture force (N) by the bonding area (mm2) for each specimen (Figure 3).[25]

The failure mode was classified after the test by visual analysis according to failure mode in 6 levels:

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

Contact Water Angle and Wettability

Contact angle measurement was carried out using standardized image obtained by digital camera (DXM-l200; Nikon, Tokyo, Japan). The angle measurements were carried out with distilled water drop deposited over the EVA specimen at controlled temperature (25 ± 2 ºC). The volume of the sessile drop was maintained as 5µl in all cases using a micro pipette (Peguepet, Santa Catarina, Brazil). The contact angle was captured after 60 s of the water drop. The images were used to calculate the water contact angle with the EVA plate with different treatment surfaces using public domain software (ImageJ, National Institutes of Health, Bethesda, MD, USA) (Figure 2).

Statistical Analysis

The laminated bonding strength (MPa), maximum elongation (mm) and contact angle data were tested for normal distribution (Shapiro–Wilk) and equality of variances (Levene's test), then were analyzed by one-way analysis of variance (ANOVA) followed by Tukey's and Dunnet tests. Failure modes were analyzed using Chi-square test. All tests employed $\alpha = 0.05$ significance level. All analyses were carried out with the statistical package Sigma Plot version 13.1. The SEM imagens were analyzed qualitatively.

RESULTS

The Laminated bonding strength means and standard deviation values for all EVA treatment surfaces are shown in Figure 4. The Dunnet test showed that all tested surfaces had significantly higher Laminated bonding strength than control group ($P < 0.005$). ANOVA showed significant difference among experimental tested EVA treatment. AcRm showed similar LBS than and Chlo significant higher values than 70Alc and IsoAc. Chlo had similar LBS mean values than 70Alc and higher than IsoAc. No significative difference between the 70Alc and IsoAc.

The maximal elongation means and standard deviation values for all EVA treatment surfaces are shown in Figure 5. The Dunnet test showed that AcRm, Chlo 70Alc had significantly higher ME values than control group (P < 0.005). The IsoAc had similar ME than control group The Tukey test showed that AcRm and Chlo had similar ME and significantly higher than IsoAc. The 70Alc and IsoAc had similar ME values.

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

For Contact Water Angle and Wettability shown in Figure 2 is shown that surface treatments reduced the contact angle when compared to the group without surface treatment.

DISCUSSION

The EVA plate surface treatment had significantly influenced on the laminated bonding strength, specimen elongation and the contact angle, therefore the null hypothesis was rejected. All tested EVA surface treatments tested in this study showed significantly higher laminated bonding strength and specimen elongation and resulted in lower contact angle of EVA surface.

The EVA surface treatment performed on the first thermoformed EVA plate showed to be important in the production of custom-fitted mouthguards for reducing the board delamination.[26] A study carried out through questionnaires reported that 73.2% of mouthguards users had difficulty wearing them.[26] The unpleasant taste and smell frequently caused by board delamination represented 20.7% of these individuals.[26] The importance of creating an effective EVA sealing between the plates and avoid delamination can directly contribute with greater durability and facilitate the mouthguard use.

Mouthguard thicknesses of 3 to 4 mm are recommended for custom-fitted mouthguards due the protective effect that results in lower stresses and strains in teeth during trauma impact.[13] Then, the use two EVA plates, making lamination is an essential part of the custom-fit mouthguard fabrication process.[13] The first EVA plate is thermoformed and needs to be cut and prepared using bur, abrasive rubber and brushes to adapt at the limited of custom-fit mouthguard.[13,18] During this process the profession needs to manipulate the first EVA plate and consequently glove powder, the oils and residues of the EVA preparation can create the contamination of the EVA surface. The adhesion between two materials is due to the interaction between their molecules. The intermolecular forces lead to EVA bonding with another EVA plate. (REF) 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.[27] Lower contact angles are associated with better bonding interaction. (REF) The use of AcRM and Chlo reduced the contact angle, creating more reactive EVA surface demonstrated by lower contact angle than other groups. This aspect can explain the higher laminated bond strength for these groups.

Chloroform is already indicated for cleaning and treatment of EVA plates.[28,29] However, this production needs specific license requirements for acquisition making complex the access for clinicians. (REF) The tested EVA surface treatment analyzed in this study involved products easily available in dental offices. The acrylic resin monomer, and 70% alcohol are products presented and used in the routine of clinicians in private and public dental services. Isopropyl alcohol is more specific and maybe more difficult to have in dental offices, but it has no controlled process for acquisition.

The higher LBS values showed for all tested EVA surface treatment when compared with control group, can be related to the cleanness capacity of different solvents used. The acrylic resin monomer demonstrated to be a great alternative for EVA surface treatment. The methylmethacrylate composition can interact with the EVA surface increasing of surface energy, for broking the superficial tension. Distilled water presented in 70% alcohol seems to reduce the effectiveness on the EVA surface treatment. The better penetration on the superficial molecules resulted in a more catalytic action increasing plate surface energy and breaking surface tension.

The prevalent failure mode observed, regardless of the EVA treatment performed, the rupture at the bonded area clearly showed that the test used in this study was efficient for analyze the EVA bonding mechanism. Increasing wettability (Figure 2) through the plate treatment becomes possible to see the loss of the EVA shine after application of tested products, mainly for AcRM and Chlo. Consequently, becoming less hydrophobic and diminishing contact angle being directly related to the improved adhesion between the EVA plates.

It is important that the clinician be careful regarding the mouthguard fabrication process. The delamination, even if partial area of the mouthguard board can increase the oral fluid retention and consequently increasing unpleasant odor.

These aspects can contribute for non-use for long time of the mouthguard, increasing the necessity of substitution. The use of all tested products in this study demonstrated to be a better alternative to non-surface treatment. However, considering the facility and the familiarity of acrylic resin monomer use in dental offices, the use of this product for cleaning the thermoformed first and also the second plate during the fabrication of custom-fit mouthguard should be recommend.

CONCLUSIONS

Within the limitations of design of this study the following conclusions can be drawn:

- All tested EVA surface treatment significantly reduced the contact angle of the EVA surface.
- The failure modes were predominantly adhesive for all groups and were not influenced by surface treatment.
- The laminated bond strength was significant higher when all tested surface treatment was used compared with control group.
- The higher laminated bond strength resulted in higher EVA elongation.
- The use of acrylic resin monomer and chloroform resulted in higher laminated bond strength, reduced contact angle.

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Table 1. EVA plate materials used.

Shape	Type	Dimension	Batch Numbers	Manufacture
Circular	Soft plate mixed colors	13cm Thickness - 3.0 mm	41712/ 48952/ 54492 / 51901	Bioart, São Carlos, SP, Brazil.

Table 2. Materials used for surface EVA plates treatment Materials.

Treatment	Composition	Batch Number	Manufacture
Self-curing acrylic liquid	Methylmethacrylate; EDMA; DMT; Inhibitor; Fluorescent	180214	Vipiflash, Pirassunga, Brazil
Isopropyl Alcohol	Isopropyl Alcohol	15748	Quality, Cidade, Pais
70% alcohol	Distilled Water; Isopropyl Alcohol	0036/012020	Ciclo Farma, Serrana, Brazil
Chloroform	Chloroform; Distilled Water	25270	Alphatec, Cajuru, Brazil

*Information provided by manufacturers.

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
EVA plates – before plasticization	3.0 ± 0.1	82.9 ± 0.6
First EVA Plate after plasticization	2.5 ± 0.4	79.3 ± 6.6
Second EVA Plate after plasticization	2.6 ± 0.4	79.9 ± 3.0
Bonding specimen area (2 EVA plates)	4.7 ± 0.5	77.8 ± 4.2

Figures

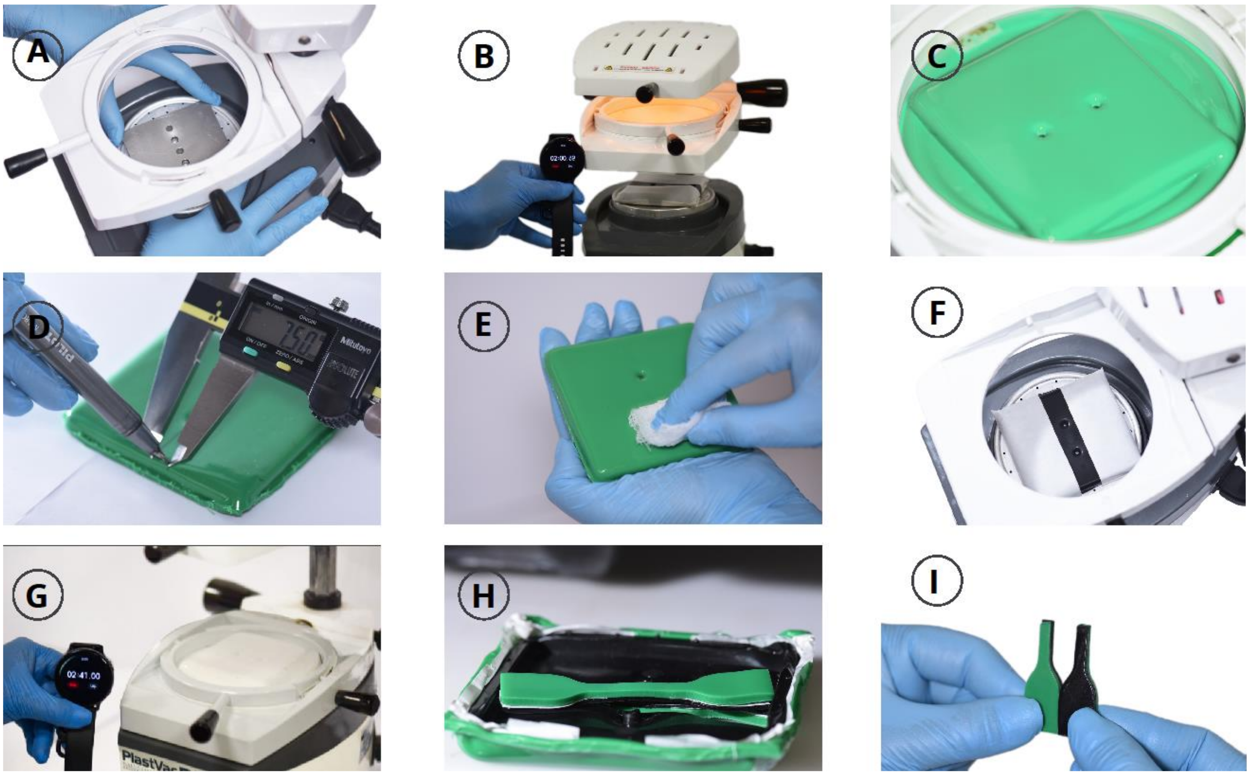

Figure 1. Specimen fabrication: A. Metal model positioned in vacuum laminator machine; B. EVA plate heating for 2 minutes; C. Metal plate laminated by the first EVA plate; D. Adhesion area measures; E- Surface treatment; F. Wrapped plate positioned in vacuum laminator; G. Heating and lamination of the second plate respecting a cooling time of 2 minutes; H. Dumbbell cut performed; I. Final aspect of the samples extracted from de plates union.

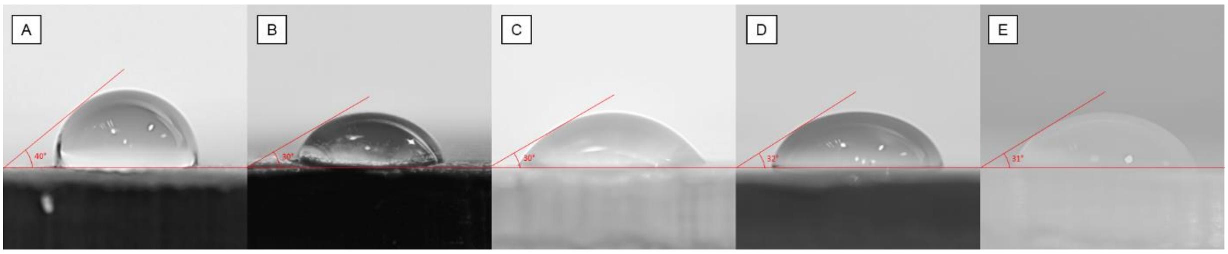

Figure 2. Contact water angle: A No treatment (control); B. Isopropyl alcohol; C. 70% alcohol; D. Chloroform; E. Acrylic resin monomer.

Figure 3. Laminate bond strength test using a universal test machine with a pneumatic griping for fixation of the EVA specimen.

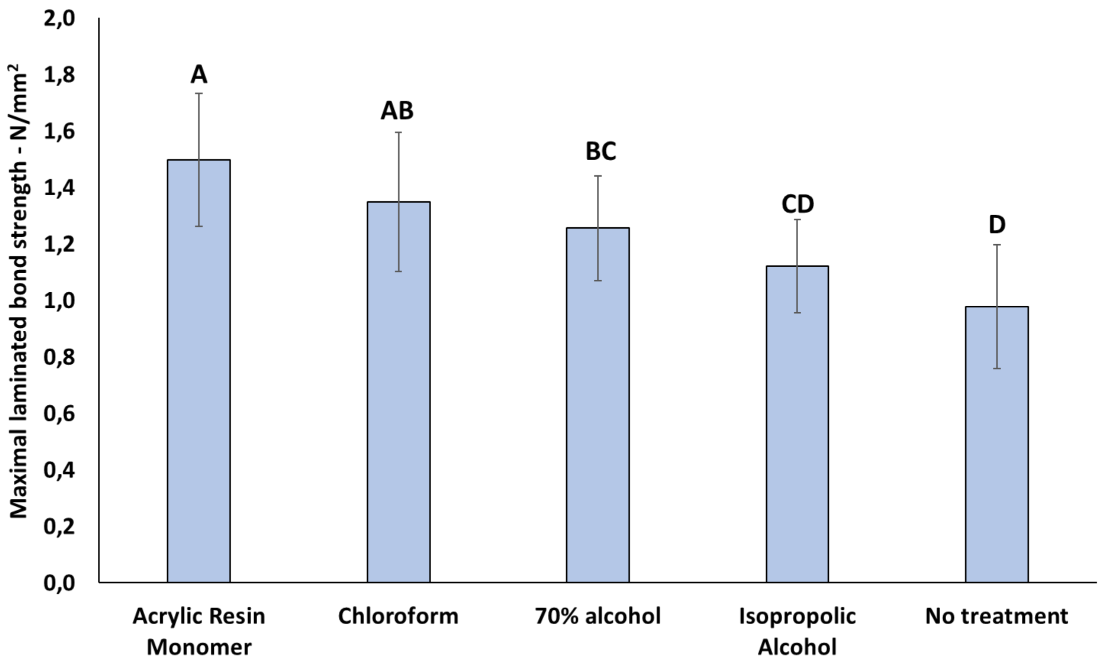

Figure 4. Maximal laminated bond strength (N/mm²) of different surface treatments. Different letters indicate a significant difference calculated using two-way RM ANOVA ($P < 0.05$).

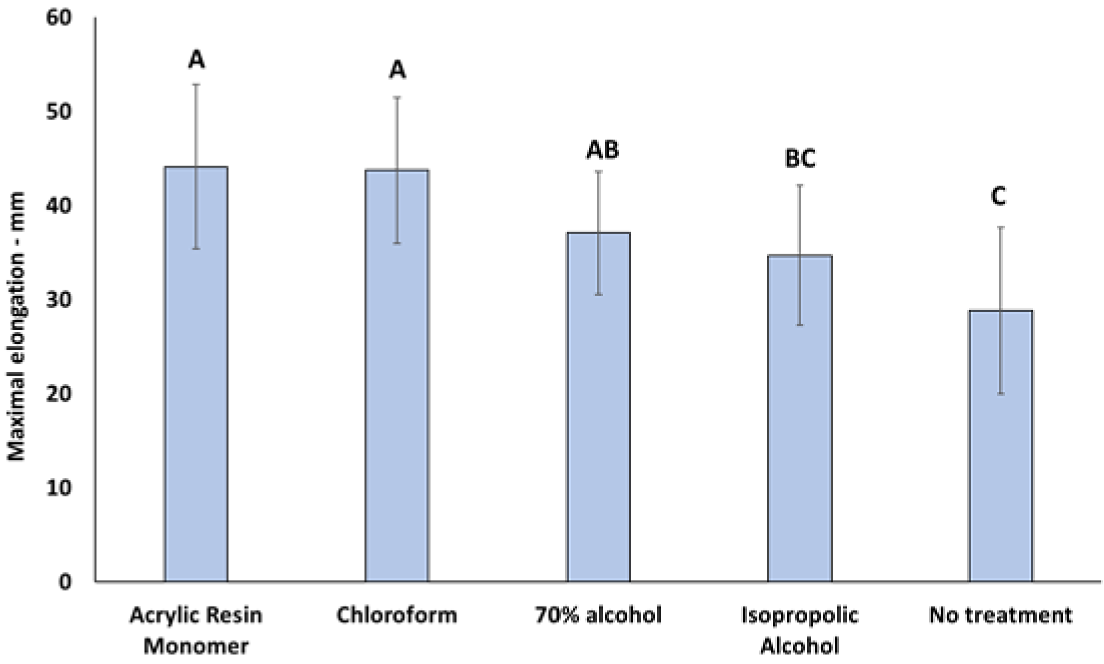

Figure 5. Maximal sample elongation (mm) of different surface treatments. Different letters indicate a significant difference calculated using two-way RM ANOVA ($P < 0.05$).

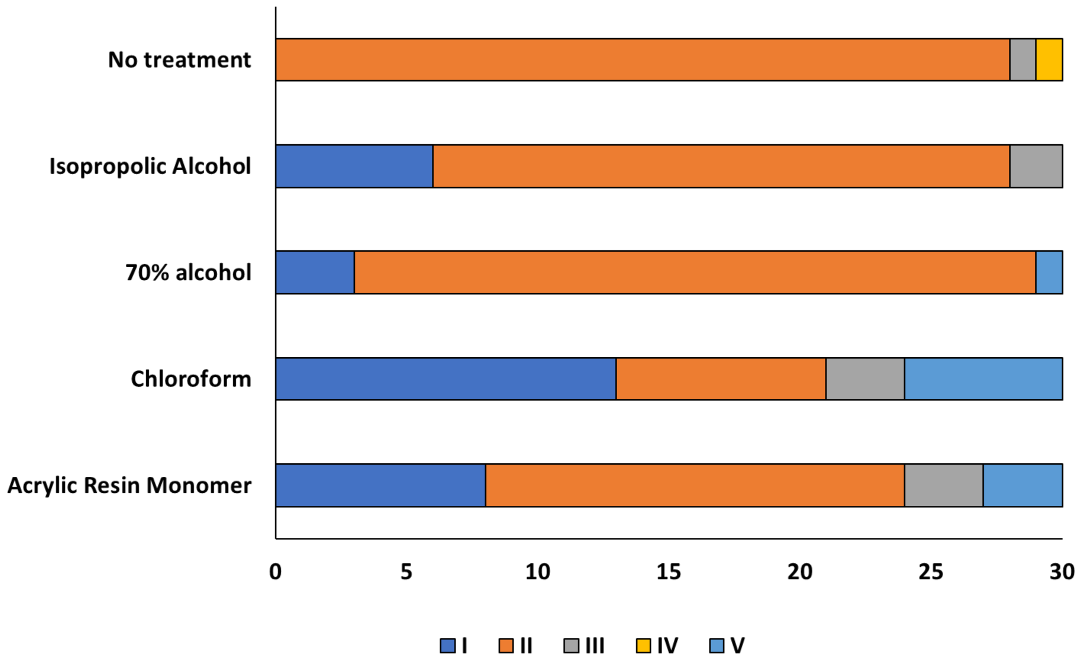

Figure 6. Failure mode distribution of different surface treatments. I. No rupture between the plates; II. Adhesive Rupture in the bonded area; III. Rupture predominance of the first plate in the bonded area; IV. Rupture predominance of the second plate in the bonded area; V. Rupture predominance of the first plate outside the bonded area.

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