Universidade Estadual de Campinas
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Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestra em Clínica Odontológica, na Área de Prótese Dental.

Dissertation presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Master in Clinical Dentistry, in Prosthodontics area.

Orientador: Prof. Dr. Wander José da Silva
Coorientadora: Profª. Drª. Sílvia Carneiro de Lucena Ferreira

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“e ela mesma resolveu escolher
tomar este caminho de cá, louco e
longo, e não o outro, encurtoso.
Saiu, atrás de suas asas ligeiras,
sua sombra também vindo-lhe
correndo, em pós.”
- Guimarães Rosa

RESUMO

A impressão tridimensional (3D) tem se destacado na fabricação de próteses, oferecendo vantagens como menor tempo de confecção, redução de resíduos e maior previsibilidade nos tratamentos. A prótese confeccionada em 3D produz separadamente base-dente, exigindo uma adesão posterior. Por esta razão, foi realizado um estudo *in vitro* com o objetivo de avaliar diferentes protocolos de união entre dentes e bases de próteses fabricadas em 3D. Incisivos centrais maxilares e bases de próteses 3D foram desenhados de acordo com a ISO/TS 19736 e exportados para impressão na Moonray D75, Sprintray. Três protocolos de união química foram analisados: (PZ) resina para impressão 3D (Marketech priZma Bio Denture), (MPZ) pré-condicionamento com monômero + resina 3D (Marketech priZma Bio Denture), e (AD) utilização do adesivo Ivotion Bond Kit 10 (Ivoclar). Além disso, alterou-se a geometria da área de união tanto dos dentes quanto das bases com modificação do design da interface adesiva. Os espécimes foram divididos em dois grupos: com e sem modificação da área adesiva. Após os procedimentos de colagem, as amostras foram randomizadas e metade dos grupos foi submetida à 10.000 ciclos de termociclagem, simulando clinicamente as condições orais e promovendo o envelhecimento térmico do conjunto dente-base. Assim, resultou em doze grupos experimentais (n=10). A interface de união foi avaliada por meio do teste de cisalhamento, medindo a força de união, em MPa, e pela análise fractográfica, utilizando microscopia óptica e eletrônica de varredura. Os dados foram analisados por meio de Análise de Variância (três fatores) seguida do teste de Tukey (α= .05). O modo de falha foi analisado descritivamente com frequências relativas (%) registradas para cada amostra. Os resultados mostraram não haver diferença estatística da força de união entre os agentes químicos testados. (p = 0.8674). A modificação da geometria da interface teve um efeito significativo apenas no grupo PZ sem termociclagem (p = .0076), mas esse resultado não se manteve após o envelhecimento térmico. A termociclagem reduziu significativamente a resistência de união em todos os protocolos (p = .0001), exceto no grupo PZ com modificação, que preservou os valores dos espécimes não termociclados. O efeito combinado das três variáveis não apresentou diferença significativa (p =.5702). Na análise descritiva do modo de falha, observou-se influência do envelhecimento térmico e dos diferentes estágios de polimerização dos agentes químicos. Pode-se concluir que a modificação da interface não influenciou a resistência ao cisalhamento, e os agentes químicos testados tiveram desempenho semelhante. Além disso, todos os protocolos sofreram redução na resistência após o envelhecimento térmico, acompanhada por uma alteração no padrão de falha, indicando instabilidade após o estresse térmico repetitivo.

Palavras-chave: Impressão Tridimensional. Prótese Dentária. Resistência ao Cisalhamento

ABSTRACT

Additive manufacturing is a promising alternative for prosthetic manufacturing, offering benefits such as reduced fabrication time, less material waste, and greater treatment predictability. This prosthesis has tooth and base printed separately, requiring a subsequent adhesion. For this reason, the aim of this *in vitro* study was to evaluate different protocols for bonding 3D-printed denture base and teeth. Central incisors and denture bases were printed following ISO/TS 19736 standards, with and without adding mechanical modification in the interface area, and exported to a DLP printer, Moonray D75, Sprintray. Three chemical bonding protocols were assessed: (PZ) 3D-printed resin (priZma), (MPZ) MMA surface treatment + 3D-printed resin, and (AD) Ivotion Bond kit 10 (Ivoclar). After bonding procedures, half of the groups underwent thermocycling for 10,000 cycles to simulate intraoral conditions and promote thermal aging of the tooth-base interface. As a result, twelve experimental groups were formed (n=10). Bond strength was evaluated through shear bond strength testing (MPa), and fracture analysis was conducted using optical and scanning electron microscopy. Data were analyzed using a three-factor Analysis of Variance (ANOVA), followed by Tukey's test (a=.05). Failure patterns were assessed with relative frequencies (%) for each sample. The results revealed that no statistical difference was found between chemical agents (p=.8674), and mechanical modification on the interface showed a significant effect only in the PZ group without thermocycling (p= .0076). A significant reduction in bond strength (p = 0.0001) was observed across all protocols after thermocycling, except for the modified PZ group, which maintained the same values as the non-thermocycled specimens. Additionally, the combined effect of the three variables showed no significant difference (p = 0.5702). In the descriptive analysis of the failure mode, the influence of thermal aging and the different polymerization stages of the chemical agents was observed. The interface design modification did not influence shear bond strength, and no significant differences were found among the chemical agents used for tooth bonding. Furthermore, all chemical protocols exhibited a reduction in bond strength after thermal aging, accompanied by a change in the failure pattern, indicating long-term stability.

Key words: Printing, Three-Dimensional. Dental Prosthesis. Shear Strength**.**

SUMÁRIO

1 INTRODUÇÃO

O edentulismo será uma das 30 principais doenças globais previstas até 2050, impulsionado pelo crescimento populacional e pela falta de políticas de saúde adequadas (Nascimento et al., 2024). A perda dentária causa alterações estruturais e funcionais que comprometem a mastigação, dificultam a alimentação, afetam a estética e a fala, além de impactar negativamente o bem-estar psicossocial e a qualidade de vida (Rodrigues et al., 2021; Marchi et al., 2012). No Brasil, essa realidade é alarmante: dados da SB Brasil de 2023 indicam que mais de 75% dos idosos entre 65 e 74 anos necessitam de algum tipo de prótese (SB Brasil, 2023). Um dos fatores que contribuem para esse cenário é a crença de que a perda dentária é um processo natural do envelhecimento, levando muitos indivíduos a negligenciarem os cuidados bucais (Cardoso M et al., 2016) e a substituírem progressivamente os dentes naturais por próteses.

Dentro desse contexto, as próteses totais removíveis convencionais continuam sendo a principal opção reabilitadora para muitos pacientes com arcos edêntulos (Kern JS et al., 2016). Tradicionalmente, essas próteses são confeccionadas em resina acrílica à base de poli (metacrilato de metila) termopolimerizável pelo método da cera perdida. Apesar de ser um método de processamento simples, de baixo custo e amplamente utilizado, (Patil et al., 2006; Cunningham et al., 1992) exigem habilidade do operador, demandam tempo e apresentam desafios, como alterações dimensionais na base da prótese (Moslehifard et al., 2022), e o risco de descolagem dos dentes devido ao processamento (Murat Kurt et al., 2016).

Em resposta a essas limitações, a incorporação de tecnologias avançadas tem revolucionado a odontologia, especialmente com a introdução do fluxo digital na fabricação de próteses dentárias (Simoneti et al., 2022). Essa evolução reflete a interseção entre ciências não médicas e ciências médicas, onde conceitos e técnicas das ciências não médicas foram adaptados para otimizar o planejamento e a execução de tratamentos protéticos (Mitsouras et al., 2015). O método CAD/CAM exemplifica esse avanço, integrando etapas digitais ao processo de confecção de próteses. O CAI (inspeção auxiliada por computador) permite a digitalização precisa por meio de scanners intra e extraorais, enquanto o CAD (desenho auxiliado por computador) possibilita o planejamento detalhado e a manipulação virtual do tratamento em softwares especializados. Por fim, o CAM (manufatura auxiliada por computador) materializa o projeto final, salvo em formato STL, por meio de tecnologias aditivas, como impressão 3D, ou subtrativas, como a fresagem de blocos pré-fabricados, garantindo alta precisão e eficiência na produção (Yeslam et al., 2024; Lourinho et al., 2022; Uzun et al., 2008).

Considerando as tecnologias de manufatura aditiva e subtrativa, existem diferenças fundamentais entre os dois métodos. A manufatura subtrativa esculpe objetos a partir de blocos pré-fabricados usando fresas em multieixos. Contudo, gera altos volumes de resíduos, impede a reutilização do material excedente, possui baixa eficiência na produção em larga escala, tem limitações na reprodução de geometrias complexas e sofre desgaste das ferramentas, comprometendo a qualidade das peças (Dewan et al., 2023; Kessler, A. et al., 2020; Kihara et al., 2020; Kalberer et al., 2019) . Por outro lado, a tecnologia aditiva, ou prototipagem rápida, baseia-se na adição de material em camadas por meio de uma impressora 3D até a obtenção do formato final do produto. Esse processo resulta em menor desperdício, uma vez que o material não utilizado pode ser reaproveitado. Além disso, apresenta um tempo de fabricação reduzido, minimizando o número de etapas necessárias para a produção do item, o que resulta em menor intervenção humana e, consequentemente, uma redução nas possibilidades de erros (Sulaiman BDS et al., 2020; Lee et al., 2019; Kalberer et al., 2019; Taormina et al., 2018). Embora a prototipagem rápida otimize a produção e reduza desperdícios, a digitalização direta dos arcos totalmente edêntulos ainda representa um desafio (Kang et al., 2024), o que pode impactar a confecção de próteses totais por ambas as tecnologias.

Na odontologia, a tecnologia impressão 3D mais amplamente utilizada é a polimerização de líquidos (*vat photopolymerization*, VPP), devido ao seu baixo custo, curva de aprendizado acessível e facilidade de manuseio (Caussin et al., 2024). Esse método utiliza fontes de luz, como laser ou ultravioleta (UV), para curar resinas líquidas depositadas nos tanques das impressoras 3D, promovendo a conversão eficiente de monômeros em polímeros presente nas resinas fotossensíveis (Taormina et al., 2018; Low et al., 2017;). Dentre os métodos de fotopolimerização (VPP), a Estereolitografia (SLA), o Display de Cristal Líquido (LCD) e o Processamento Digital de Luz (DLP) destacam-se pela precisão, custo acessível e alta velocidade de impressão (Caussin et al., 2024; Taormina et al., 2018; Yoon et al., 2018). A técnica SLA emprega um laser de alta potência para delinear a camada ponto a ponto (Kessler, A. et al., 2020; Hwang et al., 2019; Han & Cho, 2018). No método LCD, a projeção de toda a camada ocorre simultaneamente, porém a exposição à luz de alta intensidade pode provocar superaquecimento, acelerando a degradação da tela e exigindo substituições frequentes (Kortelainen et al., 2021). Em contraste, o DLP emprega um projetor que direciona a luz para espelhos, os quais refletem um único flash, promovendo a cura completa da camada de resina e aumentando a eficiência do processo (Kessler et al., 2020).

As resinas fotossensíveis utilizadas na técnica DLP são compostas por monômeros, oligômeros—como uretano dimetacrilato (UDMA) e trietileno glicol dimetacrilato (TEGDMA)—, além de fotoiniciadores ou sistemas fotoiniciadores e aditivos (Alsandi et al., 2021; Bayarsaikhan et al., 2021; Lin et al., 2020; Lebedevaite et al., 2019; Skliutas et al., 2018). A polimerização ocorre por meio de radicais livres, gerados pela irradiação dos fotoiniciadores em um comprimento de onda específico, que converte a energia fotolítica em radicais e transforma o material líquido em um polímero termofixo (Bagheri et al., 2019). Esses componentes são ajustados para garantir a cura eficiente e a qualidade estética das próteses. No entanto, a formulação das resinas deve equilibrar resistência e flexibilidade para evitar falhas estruturais (Lebedevaite et al., 2019; Skliutas et al., 2018).

Pela tecnologia DLP, os dentes artificiais não são impressos simultaneamente à base e sim adicionados posteriormente através da união adesiva entre ambas as partes. Esse processo tem a desvantagem de criar uma interface sujeita a falhas de descolagem dos dentes da base causando desconforto estético e social para o paciente, ou, em casos mais graves levar ao risco de deglutição e/ou aspiração desses elementos (Mahadevan et al., 2015; Akin et al., 2014).

Nesse contexto, pesquisas recentes têm investigado diferentes protocolos de união químicos e mecânicos como alternativas para otimizar a resistência de união entre dentes e bases de próteses impressas. Cleto *et al.* (2023) avaliaram a resistência ao cisalhamento utilizando agentes químicos, como o monômero de metil metacrilato (MMA), a resina Duralay e a resina líquida 3D recomendada pelo fabricante. Os resultados indicaram que o pré-tratamento com MMA, combinado com a colagem utilizando a resina fotopolimerizável, proporcionou uma resistência de união superior em comparação aos outros grupos testados. Em contrapartida, a Gibreel *et al.* (2024) não encontrou diferença estatística na utilização monômero de metil metacrilato (MMA) como agente químico para união de espécimes impressas. Adicionalmente, Helal *et al.* (2022) investigaram dois tipos de bases de próteses — fresadas e impressas — juntamente com agentes químicos, como diclorometano (DCM), um solvente orgânico, e métodos mecânicos, como jateamento de alumínio e asperização com brocas. Eles relataram ausência de diferença significativa na resistência ao cisalhamento entre os agentes químicos testados para bases de próteses CAD/CAM.

Modificações da macro e microgeometria da interface de união, como a perfuração na base dos dentes (Pereira et al., 2024; Al-Somaiday et al., 2022), jateamento com óxido de alumínio (Pereira et al., 2024; Kane et al., 2023; Kwanwong B. et al., 2022; Helal et al., 2022; Alharbi et al. 2021; Han et al., 2020) e asperização com brocas das superfícies (Helal et al., 2022) são suportadas pela literatura como recursos mecânicos para melhorar a união entre dente e base protética tanto no fluxo convencional como digital. O jateamento com óxido de alumínio (Corsalini, M et al., 2014) e a asperização com brocas geram microirregularidades/microcavidades na superfície (Helal et al., 2022), aumentando a área de união e facilitando a penetração e difusão dos agentes químicos. Paralelamente, as perfurações ampliam a área de contato e promovem um travamento mecânico mais eficiente (Pereira et al., 2024) Adicionalmente, Cardash *et al.* (1990) relataram tanto aumento quanto redução dessa resistência ao modificar a macrogeometria da interface de união. Já Pereira *et al.* (2024) utilizaram uma combinação de métodos químicos e mecânicos, incluindo a perfuração de todos os espécimes impressos e pré-fabricados, e observaram um aumento na resistência de união quando ambos foram aplicados de forma sinérgica. Ademais, Alharbi *et al.* (2021) utilizou o jateamento de oxido de alumínio 100 mm em amostras impressas com protocolo químico de união com adesivo a base de PMMA, Ivobase CAD bond kit, e houve diferença estatística em relação ao método convencional.

Entretanto, não foram encontrados estudos que avaliassem, simultaneamente, modificações macromecânicas na superfície da base e dos dentes impressos associadas a protocolos químicos de união. Diante desse cenário, o presente estudo testou diferentes protocolos de união, incluindo a modificação da macrogeometria da área adesiva por meio da inclusão de um cone arredondado (Tzanakakis et al., 2023) tanto no dente quanto na base da prótese. Esse recurso, já utilizado de forma semelhante, para modificação dos dentes, em técnicas convencionais e digitais (Pereira et al., 2024; Phukela et al., 2011), pode ser incorporado diretamente no software de design, oferecendo vantagens como travamento mecânico inicial, auxílio no posicionamento dentário e aumento da área de contato entre o dente e a base protética, o que pode favorecer a união. Diante do exposto, o objetivo deste trabalho foi avaliar o efeito de diferentes protocolos de união na resistência da união entre dentes artificiais e a base de próteses totais obtidas pela técnica de impressão tridimensional

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2 ARTIGO: Association of different interface design and adhesion protocols to improve bonding strength of 3D-printed artificial teeth and denture bases

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Association of different interface design and adhesion protocols to improve bonding strength of 3D-printed artificial teeth and denture bases

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Abstract

Purpose: This *in vitro* study aimed to evaluate different protocols for bonding 3D-printed denture bases and teeth. *Materials and Methods:* Central maxillary incisors and cylindrical bases were designed in MeshMixer, following ISO/TS 19736, with and without a modification on the design of adhesion area. Specimens were printed in a Moonray D75 (Sprintray) using PriZma 3D Bio Prov for teeth and PriZma 3D Bio Denture for bases. Three chemical protocols were tested: 3D-printed resin (PZ), MMA + 3D-printed resin (MPZ), and Ivotion Bond Kit 10 (AD). Half of the groups underwent 10,000 thermocycling cycles, totaling twelve groups (*n* = 10). Shear bond strength (N) was tested using a Universal Testing Machine and shear bond stress (MPa) was calculated. Failure patterns were analyzed via light and scanning electron microscopy. Data were evaluated using three-way ANOVA and Tukey's HSD ($\alpha = 0.05$). *Results:* The adhesion interface modification significantly increased bond strength only in the non-thermocycled PZ group ($p = 0.0076$). No statistical difference was found among the different chemical agents tested ($p = 0.8674$). After thermocycling, bond strength decreased ($p = 0.0001$) except in the PZ modified group, which remained stable. The combined effect of the three variables showed no significant difference ($p = 0.5702$). *Conclusions:* Modifying the interface design did not affect shear bond strength and no significant differences were found among the chemical agents. While all protocols met clinical standards, further long-term assessment is necessary to confirm their stability and effectiveness.

Keywords: Computer-Aided Design. Printing, Three-Dimensional. Dental Prosthesis. Shear Strength.

1 Introduction

Additive manufacturing, or 3D printing, has revolutionized dentistry by providing efficient solutions for complete denture production, reducing service time and human errors[1-3]. Digital Light Processing (DLP), a widely used technique, offers high precision, speed, and low porosity, minimizing microorganism colonization[4-6]. Using UV light, the liquid resin it's polymerized layer by layer, with a projector directing the digital design onto a mirror to cure each layer[7-9]. This method enhances production efficiency, enables the simultaneous fabrication of multiple devices, and allows digital storage of patient data for future treatments[8,10,11].

In the DLP technique, teeth and the base are fabricated separately and later bonded using an adhesive[12]. This process creates a failure-prone interface, making tooth debonding a significant concern. Thus, research on effective bonding protocols is essential to prevent such failures, which can compromise function, aesthetics, and patient safety[13,14].

Recent research has investigated various chemical and mechanical bonding protocols as alternatives to optimize bond strength in 3D-printed prostheses. Chemical approaches include the use of methyl methacrylate (MMA) liquid[15-17], IvoBase CAD systems[18-20], Palabond treatments[17, 21], uncured 3D-printing resin [17,18], and resin cement[22]. Cleto *et al*. (2023)[17] evaluated shear bond strength using different chemical agents, including MMA monomer, Duralay resin, and the manufacturer's recommended 3D-printing resin. Their findings indicated that MMA pretreatment, combined with light-cured resin, resulted in superior bond strength. In contrast, Gibreel *et al*. (2024)[23] found no significant difference when using MMA monomer alone.

While mechanical methods include air abrasion with particles[16,20,21,19,23,24], surface roughening with burs[24,27], or incorporating perforations in denture teeth[21], Pereira *et al.*

(2024)[21] combined chemical and mechanical approaches, including perforation of all printed and prefabricated specimens, and observed increased bond strength when both methods were applied synergistically. Additionally, Cardash *et al.* (1990)[25] reported both increases and decreases in bond strength when modifying the bonding interface in conventional prostheses.

Despite conflicting results, studies on mechanical modifications at the interface between the base and 3D-printed teeth, combined with chemical protocols, remain limited. This technique, common in conventional denture fabrication[26,27], may improve bond strength in digital workflows. This *in vitro* study evaluates the effect of adhesion area design modification and chemical agents on the bond strength between artificial teeth and 3D-printed prosthesis bases. The null hypothesis states that bonding protocols will not show statistically significant differences in bond strength.

2 Material and methods

2.1 Experimental design

This *in vitro* study compared six protocols for bonding a 3D-printed central maxillary incisor to a denture base, both fabricated by digital workflow. Two interface designs (with or without design modification) and three chemical agents were tested: (PZ) 3D-printed resin (PriZma), (MPZ) MMA + 3D-printed resin, and (AD) Ivotion Bond Kit 10 (Ivoclar). Half of the groups underwent thermocycling, resulting in 12 groups (n=10). Independent variables included design modification, chemical agents, and thermocycling. Shear bond strength (N) and bond stress (MPa) were measured using a Universal Testing Machine, and failure patterns were analyzed via light and scanning electron microscopy.

2.2 Samples

For this experiment, 120 artificial teeth (crown width: 8.5 mm; crown height: 11 mm) and 120 cylindrical base specimens (25 × 20 mm) were fabricated following the ISO/TS 19736:2017 standard[28,31]. The samples were designed using MeshMixer CAD software and saved as Standard Tessellation Language (STL) files. Two interface designs were created: a flat interface (Fig. 1a) and modified interface (Fig. 1b). The modified design featured an elliptical structure (3 mm × 6 mm × 3 mm), with half anchored to the base as a projection (3 mm × 3 mm × 3 mm), while the other half was subtracted from the tooth. A total of 60 sets of each design were printed.

The specimens were 3D-printed horizontally, perpendicular to the shear force[29], with a 50 µm layer thickness[19, 29]. After printing, they were removed from the platform, sanitized, and placed in an ultrasonic chamber with 99% isopropanol for 5 minutes, following manufacturer recommendations. Post-curing was performed for 20 minutes in a UV LED oven (dOne 3D, Anycubic, 405 nm, 40 W, 70 W heating), ensuring final material properties. Specimens were then sanded with 1200- and 1500-grit sandpaper for 1 minute each, followed by mechanical polishing with a rotating felt disc for 1 minute[30].

2.3 Chemical bonding protocols

Both the flat (n=60) and modified interface (n=60) specimen sets were randomly divided into three groups based on the chemical bonding protocol: (1) PZ – bonding with 3D-printed resin (priZma), (2) MPZ – surface treatment with methyl methacrylate (MMA) liquid followed by bonding with 3D-printed resin (priZma), and (3) AD – bonding with Ivotion Bond Kit 10, a CAD bonding system (Ivoclar) (Fig. 1).

For the PZ chemical protocol, a drop of 3D-printed denture base resin (priZma 3D Bio Denture) was applied, and the denture base resin cylinder was positioned and held under digital pressure for 60 seconds. Excess material was removed with a brush[17], and the specimens underwent post-curing for 20 minutes in a post-curing oven (dOne 3D, Anycubic).

For the MPZ protocol, two drops of monomer were applied to the cervical region of the artificial tooth for 60 seconds, repeated three times (total: 180 s)[31]. Next, a drop of 3D-printed denture base resin (priZma 3D Bio Denture) was applied, and the assembly was held under digital pressure for 60 seconds and excess material was removed with a brush[17], and polymerization was performed in a post-curing oven (dOne 3D, Anycubic) for 20 minutes.

For the AD group, the teeth and denture base were air-particle abraded with 100 µm aluminum oxide for 10 seconds at 2 Bar, following the manufacturer's instructions [19,32]. The surfaces were then conditioned with JET monomer for 30 seconds. The Ivotion monomer and polymer were mixed and applied according to the protocol. Polymerization was completed by securing the assembly with elastic bands and placing it in a pressure pot at 50 °C under 2 to 5 Bar of pressure for 15 minutes.

All bonding procedures were performed by a single operator (M.F.P.), and specimens were stored in distilled water at 37 °C for 48 hours[33]. Materials used are detailed in Table 1.

2. 4 Thermocycling

Specimens of all groups were randomly assigned to thermocycling or non-thermocycling (n = 10). Thermocycled specimens underwent 10,000 cycles using the OMC 300 TSX (Odeme Dental Research, Luzema, SC, Brazil) with temperature alternations between 5°C and 55°C, a transfer time of 10 seconds, and a dwell time of 30 seconds [33,34]. Non-thermocycled specimens were stored in distilled water at 37°C for 24 hours before testing.

2. 5 Shear bond stress

Bond strength was tested using a Universal Testing Machine (Instron Model 4400), with shear bond stress calculated as σ = L/A, where L is the fracture load (N) and A is the adhesion area (mm^2). The load was applied to the palatal surface at a 90° angle, following ISO/TS 19736:2017[28], with a crosshead speed of 1 mm/min. Failure patterns were recorded, ensuring consistent force application across specimens.

2. 6 Failure pattern

After fracture, all specimens were examined under a light microscope (EMF-1; Meiji Techno) to classify the failure pattern as adhesive (fracture at the bond interface), cohesive (fracture within the denture base material or teeth), or mixed (a combination of adhesive and cohesive failure)[35]. Subsequently, three specimens from each group were analyzed using scanning electron microscopy (SEM) (JSM5600LV; JEOL Technics) to evaluate the morphology of the fracture surfaces.

2. 7 Statistics Analysis

Statistical analysis was performed using SAS/LAB (version 9.0, SAS Institute Inc., Cary, NC, USA). Normality (Shapiro-Wilk, $p > .05$) and homoscedasticity (Levene, $p > .05$) were confirmed. A three-way ANOVA was conducted, followed by Tukey's post hoc test ($\alpha = .05$) for significant interactions. Descriptive analysis assessed failure patterns based on relative frequencies (%).

3 Results

The results of the three-way ANOVA regarding the combined effect of interface modification, chemical agents, and thermocycling on bond strength values are summarized in Table 2. The combined effect of the three variables showed no significant difference ($p = .5702$) while significant differences were observed in the interaction between thermocycling and interface modification ($p = .0058$). No significant difference was found among the three chemical protocols tested ($p = .8674$).

Pairwise Tukey's post hoc analysis (Table 2) indicated that the modification in interface design improved bond strength for specimens bonded with priZma (PZ) before thermocycling. For the other groups, no significant difference in bonding strength was observed between the flat and modified interfaces, regardless of thermocycling.

The PZ modified group was the only one that had no significant reduction in bond strength after thermocycling, although the values after aging had no statistically significant difference when compared to the other chemical agents in similar conditions – interface modified.

As shown in Figure 2, most samples exhibited mixed or cohesive failures, and thermocycling led to an increase in cohesive fractures at the expense of mixed failures, especially for the PZ and MPZ groups. The AD group exhibited no adhesive failure, even after thermocycling.

Modified interfaces showed more mixed failures, while flat samples had more cohesive failures (Figure 3). Initially, the mechanical modification reduced adhesive failures, but after thermocycling, adhesive failures remained unchanged, with an increase in cohesive failures and a decrease in mixed failures.

SEM analysis revealed increased porosity within the adhesive interface of the 3D-printed samples (Figure 4a), as well as fracture lines and areas of stress concentration (Figure 4b, 4e, 4f, and 4h). In the AD group, craze lines originated from the bond layer between the resin and the teeth (Figure 4h).

4 Discussion

This in vitro study assessed various protocols to improve bond strength between 3D-printed denture teeth and bases. Three chemical bonding protocols were applied to 3D-printed denture teeth and bases, with an additional interface design modification to evaluate its effect on shear bond strength. The null hypothesis, which proposed that mechanical modification, chemical agents, and thermocycling would not influence shear bond strength, was partially rejected. Although the chemical agents did not show statistically significant differences among themselves, the PZ group was the only one that maintained stability after thermocycling processes for the PZM-NT and PZM-T groups. The interface modification initially had an improved bond strength on the PZF and PZM groups non – thermocycling. However, this effect was not maintained after thermal aging.

Previous studies on both conventional[27,36,37] and digital prostheses[21] support modifying the tooth surface to increase the bonding area and enhance mechanical interlocking. Pereira *et al*. (2024)[21] reported significantly improved bond strength in 3D-printed prostheses by combining mechanical treatments, such as tooth preparation with burs and base sandblasting with aluminum oxide.

In this study, the interface design modification consisted of a digitally designed cone with rounded vertices for both the tooth and the denture base (Fig. 1b). This approach mimics the perforations traditionally made in conventional denture teeth to enhance bond strength[25] before acrylic processing. The literature reports variations in carbide bur numbering (#4, 6, and 8), with diameters ranging from 1 mm[36] to 2.5 mm[38] and hole depths from 0.5 mm[27] to 2 mm[38] in conventional prostheses. Given these discrepancies, a digitally replicable surface modification was chosen to ensure consistency across different software programs.

This mechanical modification did not produce statistically significant differences across all groups, except for the PZ group before thermocycling. This contrasts with the findings of Pereira et al. (2024)[21], who used burs to modify the surface of 3D-printed teeth and found a significant effect. Since burs create microirregularities that enhance adhesion by increasing the surface area and bonding agent diffusion, the absence of these irregularities in the printed modifications may explain the differences observed in this study. Further studies are needed to explore alternative surface design modifications, such as horizontal adjustments, selective alterations to specific regions, and comparisons of roughness between digitally modified and bur-prepared teeth. Notably, integrating these modifications into CAD software did not extend manufacturing time, increase costs, or introduce additional labor steps, reinforcing the viability of a fully digital workflow.

Regarding chemical agents for bonding 3D-printed bases and teeth, resin manufacturers recommend using the uncured 3D resin itself due to its low viscosity, rapid polymerization, and composition similar to that of the denture base resin[17]. The incorporation of surface pre-treatment is also supported in the literature, as its solvent properties improve adhesion between the base and teeth[39]. However, the solvent effect of methyl methacrylate (MMA) monomer was not observed in this study, and the additional laboratory step did not enhance bond strength.

In contrast, Cleto *et al*. (2023)[17] reported a significant increase in bond strength between the denture base and 3D-printed teeth using Duralay monomer as a pre-treatment in non-thermocycled specimens. The differences observed may be due to variations in solvent composition and methodological factors, such as sample diameter and the calculation of shear bond strength in MPa. Notably, Cleto *et al*. (2023)[17] used an adhesive area nearly 2.5 times smaller than in the present study. Since bond strength (MPa) is inversely proportional to adhesive area, this discrepancy could explain the higher values reported by Cleto *et al*. (2023)[17] compared to those obtained in this study, which followed ISO 19736 standards[28]. However, Gibreel *et al.* (2024)[23] found no significant effect with MMA monomer, likely due to its incompatibility with light-polymerized resins, as MMA cannot copolymerize with the di-methacrylate monomers in MMA-free 3D-printed denture base materials[23, 40].

The composition of dental materials is critical, as their chemical characteristics can directly influence bonding failures due to structural and compositional differences, leading to various polymerization reactions[17, 32]. This adhesive designed for bonding CAD/CAM specimens behaves like self-polymerized acrylic resins, expanding at higher temperatures and peaking at 70°C[33]. SEM imaging of the AD group revealed an uneven polymer bead distribution, suggesting incomplete dissolution in the monomer after polymerization in a water bath (Fig. 4h)[33]. The manufacturer's lower polymerization recommendation (up to 50°C) may restrict monomer diffusion, impacting bond strength and failure patterns.

Incomplete polymerization can create stress points, leading to crack propagation[18]. Thermal aging of AD specimens promoted secondary polymerization, forming a more interconnected polymer network, which increased cohesive failures and enhanced bond strength (Fig. 4g). Although incomplete polymerization affected initial adhesion, the AD group did not exhibit complete adhesive failures, suggesting partial polymerization in some areas (Fig. 4i). After thermal aging in the AD group, further polymerization occurred, reducing mixed failures.

This fractographic analysis supports the findings of Güntekin *et al*. (2025)[32], Choi *et al.* (2020)[18], and Alharbi *et al*. (2021)[19], who observed similar polymer beads on the adhesive surface with varying shear bond strength values. Choi *et al*. (2020)[18] reported lower values (2.21 ± 0.86 MPa) without thermal aging, while Güntekin *et al*. (2025)[32]

recorded higher values (6.01 ± 2.0 MPa), and Alharbi *et al*. (2021)[19] reported results in Newtons. These findings emphasize the role of polymer bead distribution and polymerization in adhesion between milled[18,33] and printed[19] teeth and prostheses. Variations may be attributed to differences in specimen size, aging time, manufacturing methods, storage conditions, and resin types.

The dissipation of force and stress concentration in 3D-printed resin depends on layer orientation relative to the applied load[29]. Perpendicular layers promote interlayer sliding, causing a barreling effect (Fig. 4b), which influences failure mode—a key indicator of bonding performance. Adhesive failures are least favorable, mixed failures are moderately acceptable, and cohesive failures are ideal[41]. PriZma resin was the only bonding agent maintaining consistent results with and without thermocycling. However, the PZ non-thermocycled group had the highest adhesive failure rate, which decreased after thermal aging (Fig. 4a). Choi *et al*. (2020)[18] similarly observed adhesive failures in specimens bonded with uncured resin. After thermal aging in the PZ group, adhesive failures disappeared, mixed failures decreased, and cohesive failures increased. All groups remained clinically acceptable per ISO 19736 standards[28], with adhesive failures not exceeding 33% and peaking at 20% in the PZ non-thermocycled group.

Water can act as a plasticizer in composite and acrylic resins, potentially leading to a hardening effect[42], which may have affected both the uncured and cured 3D resin (Fig. 4c). The 3D-printed resin, with its layer-by-layer polymerization technique, tends to have more voids, as confirmed by scanning electron microscopy[34] (Fig. 4b). These voids may trap water within the polymeric network, altering the observed failure pattern[43]. Surface degradation following thermocycling weakened the teeth, increasing the occurrence of cohesive fractures at the force application site. As failures occur at the weakest points under stress, these degradations likely contributed to fracture initiation[44].

Thermocycling was applied to simulate intraoral aging, following established methodologies[23,45,46]. This process significantly reduced bond strength across all bonding agents, likely due to thermal fluctuations inducing mechanical stress and crack propagation along bonded interfaces[32]. Limitations include the use of a single resin type, as viscosity variations may influence adhesion failures[21]. All samples were fabricated using additive manufacturing; future studies should explore different polymerization techniques and compare them with subtractive methods.

5 Conclusion

All tested protocols met clinical standards, with fewer adhesive failures observed, which may be attributed to factors such as material polymerization, chemical interactions, and water exposure. However, the proposed interface design modification did not significantly impact bond strength between the base and teeth of 3D-printed prostheses, regardless of the chemical agent used.

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Table 1. Details of the materials used in the in vitro study.

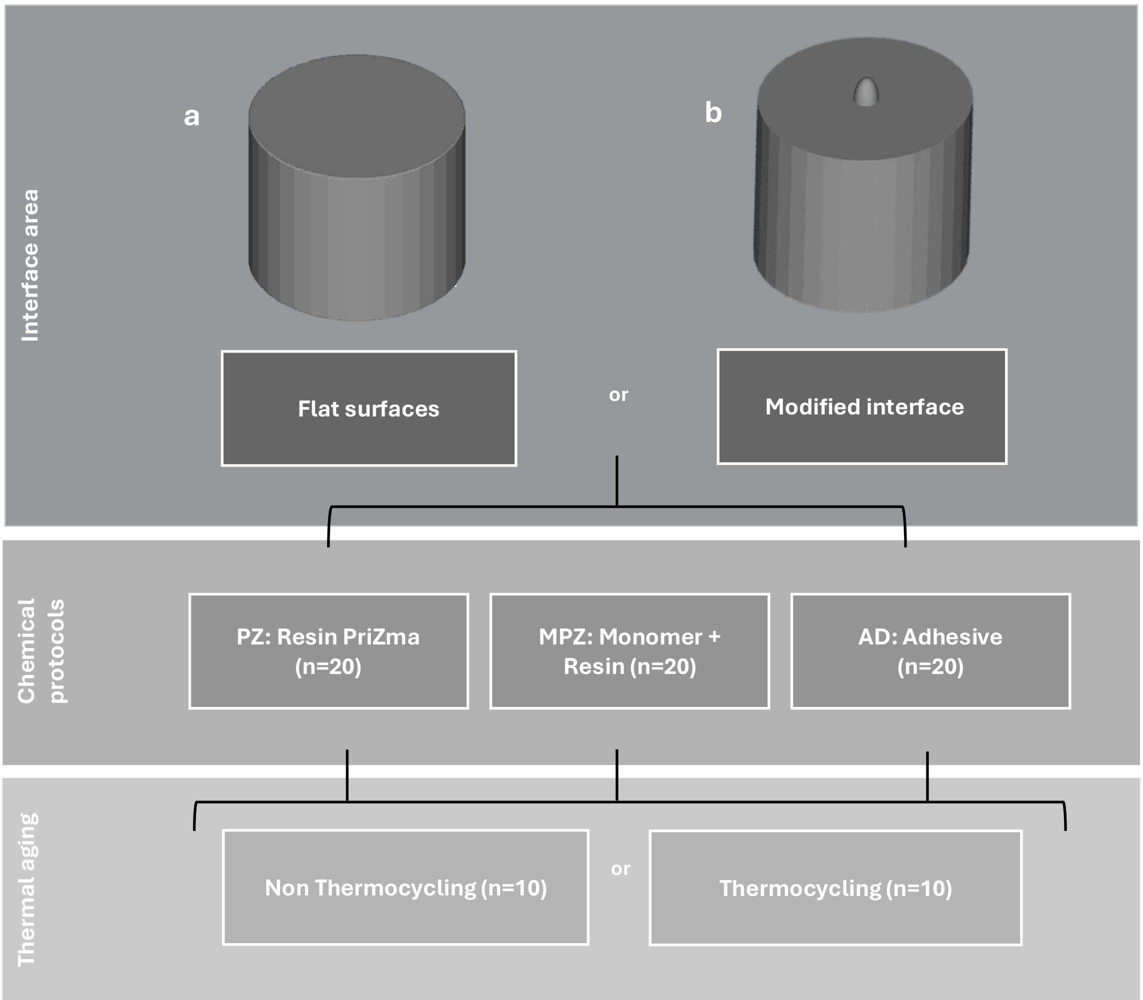
NAME OF PRODUCT	COMPOSITION	MANUFACTURER
PriZma 3D Bio Prov A1	Acrylate and triacrylated monomers (>10%), amorphous silica (≤5%), fillers – proprietary (<10%), proprietary meta-acrylated oligomers (<70%), diphenyl (2,4,6-trimethylbenzoyl)- phosphine oxide (<5%).	Makertech
PriZma 3D Bio Denture	Acrylate monomers (>10%), pigmentation and filler (≤10%), proprietary acrylateoligomers (<65%), diphenyl (2,4,6-trimethylbenzoyl)-phosphine oxide (<5%).	Makertech
Resin acrylic auto JET liquid	Methyl methacrylate, dimethacrylate	Clássico
Ivotion bond kit 10	Powder – Polymethyl methacrylate, softener, benzoyl peroxide, catalyst, pigments;Liquid e modelling liquid – Methyl methacrylate, dimethacrylate, catalyst	Ivoclar Vivadent
Aluminum oxide particles	Aluminum oxide particles – 100µm	Polidental

Table 2. Mean, SDs, and significances of shear bond strength (MPa) for chemical protocols based on interface modification and thermocycling aging.

		Chemical agents		
Thermal aging	**Interface Design**	PriZma Resin (PZ)	Monomer + PriZma (MPZ)	Adhesive Ivotion (AD)
Non Thermocycling	Flat	$4.33 \pm 1.38^{a, A}$	$5.01 \pm 1.62^{a,A}$	$4.64 \pm 0.92^{a,A}$
	Modified	$6.28 \pm 0.93^{b, A}$	$6.51 \pm 1.07^{a,A}$	$6.01 \pm 1.29^{a, A}$
Thermocycling	Flat	$2.49 \pm 0.56^{c, A}$	$2.92 \pm 0.80^{bd, A}$	$2.81 \pm 0.42^{ac, A}$
	Modified	$3.56 \pm 1.07^{bc, A}$	$2.83 \pm 1.25^{cd, A}$	$2.87 \pm 0.42^{bc, A}$
		p- thermocycling	**<.0001**	
		p- interface	**<.0001**	
		p- chemical	= 0.8674	
		p- termocycling * interface	**=.0058**	
		p-thermo*interface*chemical	= .5702	

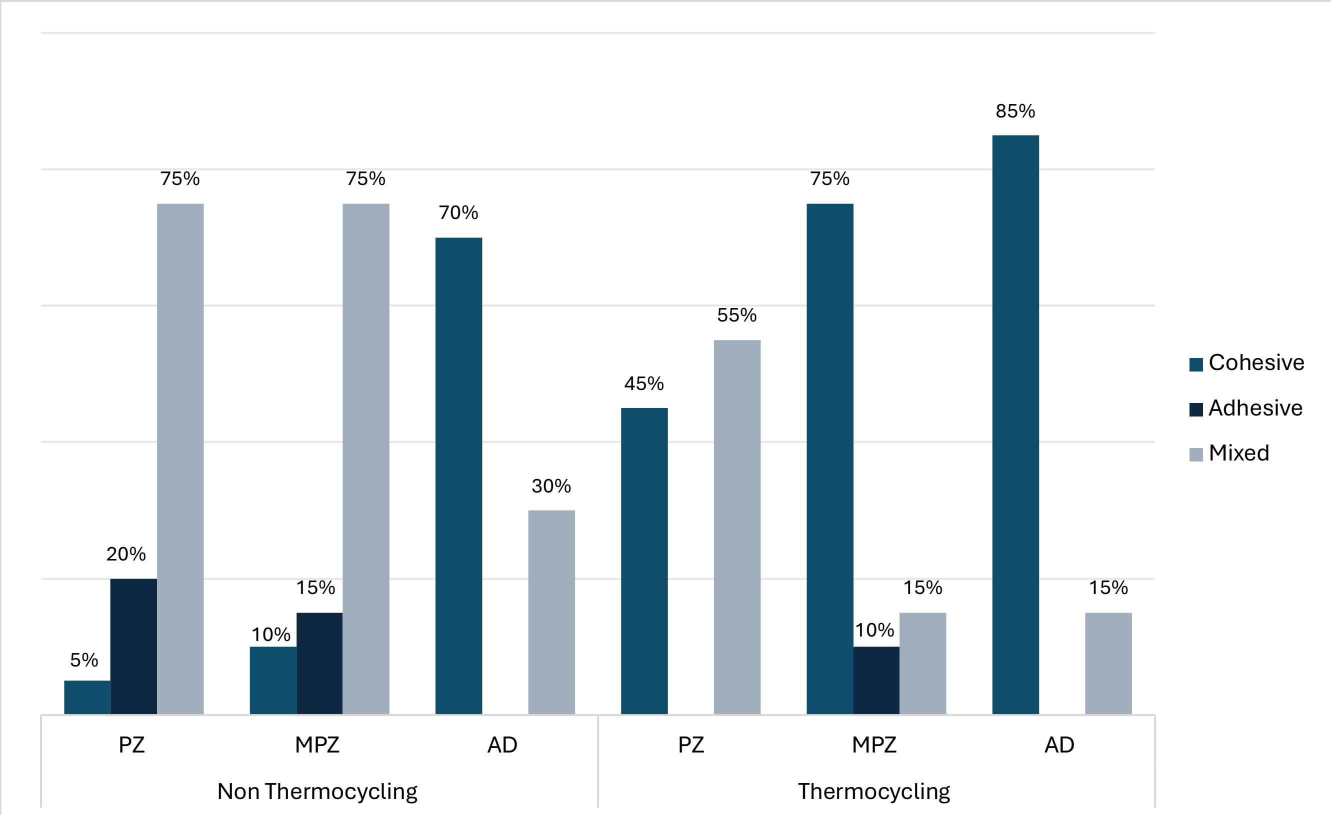
Different lowercase superscript letters indicate significant differences in the same column ($p<.05$). Different uppercase superscript letters indicate significant in the same differences line ($p < .05$)

Figure 1. Flowchart of *in* vitro study. On (a), flat surface and (b) a modified sample.



PZ: Resin priZma; MPZ: Methylmethacrylate (MMA) liquid + Resin priZma; AD: Adhesive Ivotion Bond.

Figure 2. Percentage of failure pattern according to chemical protocols and thermocycling (n=20)



PZ: Resin priZma; MPZ: Monomer+ Resin priZma; AD: Adhesive Ivotion Bond.

Figure 3. Frequence relative about interface design and thermocycling (n=30).

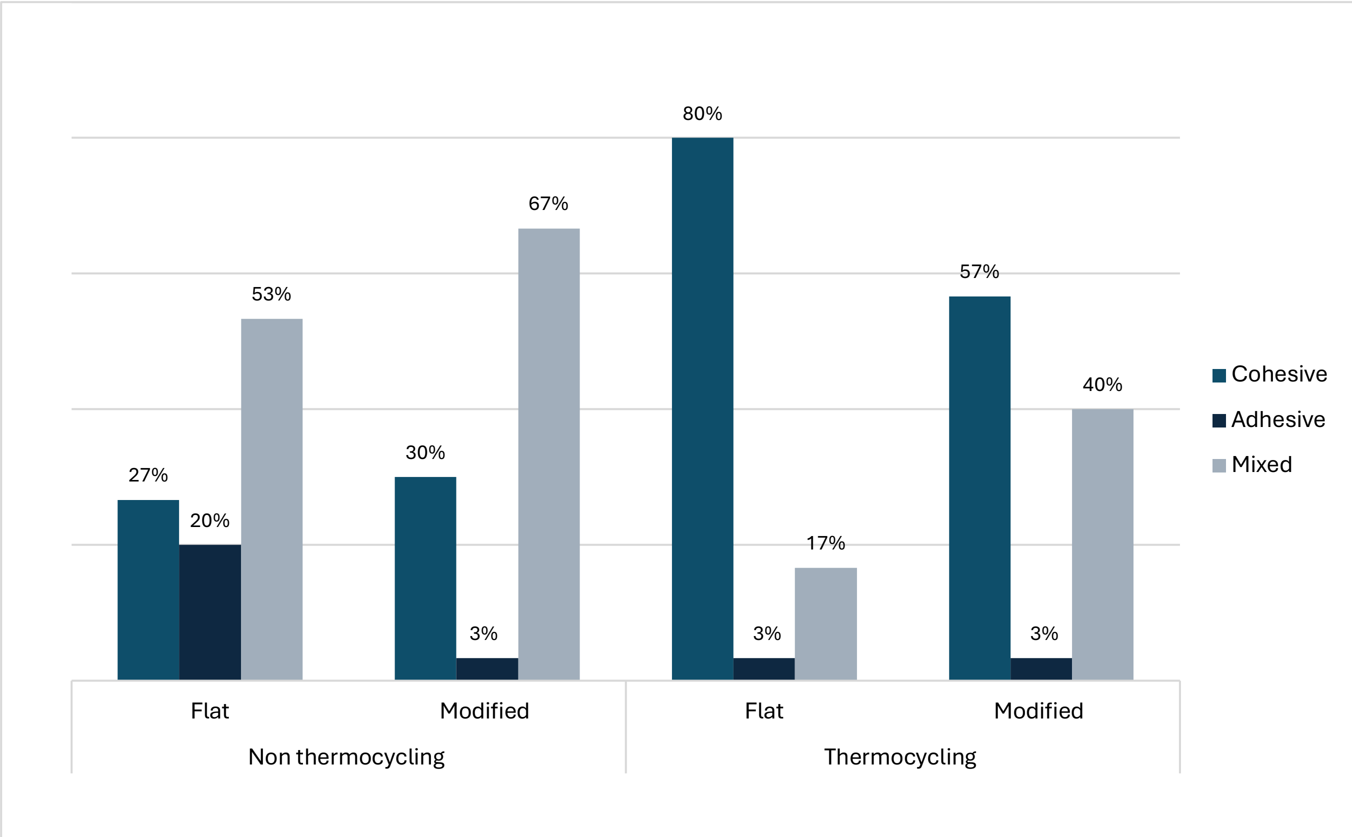


Figure 4. Scanning electron microscopy analysis.

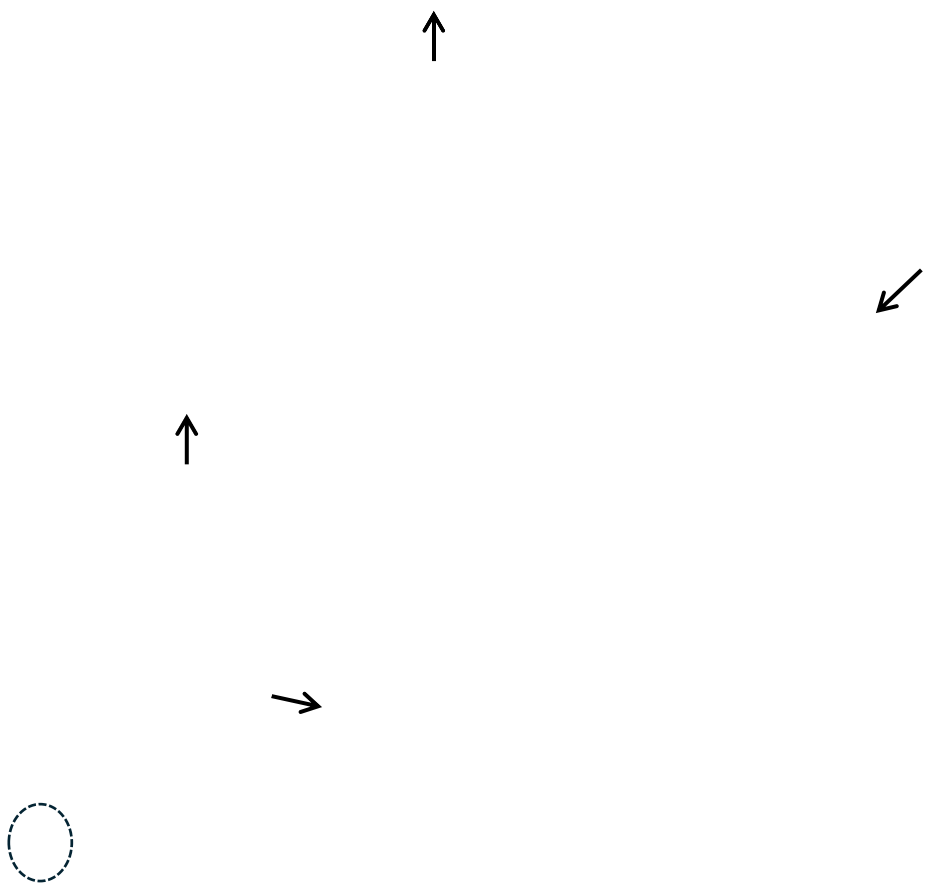


Figure 4. (a) Adhesive failure in PZM-T. (b) Porosities (white arrow) and failure propagation (black arrow) creating a barrel effect. (c) Plastic deformation post-thermal aging: loss of sharp fracture features (white arrow). (d) Mixed failure in ADM-NT: intact adhesive area (white arrow), detachment within the tooth (black arrow). (e) Stress concentration in mechanical modification (white arrows). (f) Mixed failure around modification: failure area (white arrow), stress lines (black arrow). (g) Cohesive failure in AD: tooth fragment attached to the base (white arrow), detached mechanical feature (dashed circle). (h) Stress lines near adhesive surface (white arrow), adhesive interface (black arrow). (i) Subpolymerized polymeric spheres (white arrow).

3 CONCLUSÃO

- Os protocolos de união não influenciam a resistência de união entre dentes artificiais e bases de próteses impressas em 3D.
- A resistência de união em espécimes impressos é negativamente impactada pela termociclagem.
- A análise do modo de falha revelou que, apesar da ausência de diferenças estatísticas entre os protocolos de união, o comportamento da interface adesiva varia de acordo com os agentes de união utilizados.

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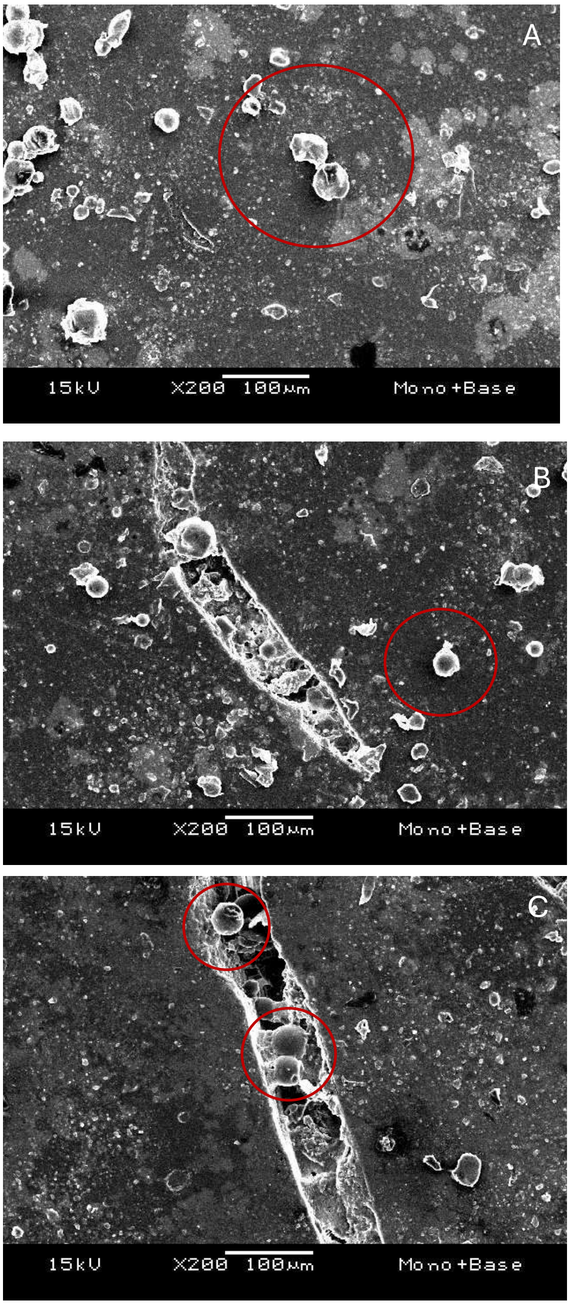
APÊNDICE 1- IMAGEM DE MEV APÓS PRÉ – TRATAMENTO COM MONÔMERO

Apêndice 1 (A): Imagem de MEV mostrando diversas partículas (círculo vermelho) na superfície dos espécimes após fricção com monômero durante o pré-tratamento. **(B):** Fenda na base e algumas partículas observadas após fricção. **(C):** Esferas poliméricas não dissolvidas observadas na superfície pré-condicionada.



ANEXOS

ANEXO 1- COMPROVANTE DE SUBMISSÃO

Dear Ms. Maryana Fernandes Praseres,

We are pleased to inform you that the manuscript "Association of different interface design and adhesion protocols to improve bonding strength of 3D-printed artificial teeth and denture bases" has been submitted to Journal of Prosthodontics by Wander José da Silva.

Please note that during the review process, the submitting author is solely responsible for communicating with the journal and providing any requested updates.

If you believe you were incorrectly listed as a co-author, please contact the editorial office by replying to this email.

Kind regards,
Journal of Prosthodontics

ANEXO 2- VERIFICAÇÃO DE ORIGINALIDADE E PREVENÇÃO DE PLÁGIO

Adesão de dentes artificiais a bases fabricadas por impressão tridimensional: Efeito da modificação da interface associada a agentes químicos na resistência ao cisalhamento

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