

Isabela Guimarães Cruz

**Avaliação Comparativa das Propriedades Físicas de Diferentes Tipos de Resinas
Utilizadas na Confeção dos *Attachments* Ortodônticos**

*Comparative evaluation of the physical properties of different types of resins used in the
fabrication of orthodontic attachments*

Dissertação apresentada à Faculdade de Odontologia da Universidade Federal de Uberlândia, para a obtenção do Título de Mestre em Odontologia, Área de Clínica Odontológica Integrada.

Uberlândia, 2023

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Orientador: Prof. Dr. Guilherme de Araújo Almeida

Banca Examinadora:
Prof. Dr. Guilherme de Araújo Almeida
Prof. Dra. Gisele Rodrigues da Silva
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Uberlândia, 2023



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Dedico este trabalho aos meus pais, *Marco Antônio e Rosiane*,

Obrigada por sempre estarem ao meu lado me apoiando e me incentivando.

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RESUMO

Introdução: O objetivo deste estudo *in vitro* foi avaliar as propriedades físicas de diferentes resinas utilizadas para confecção dos *attachments* ortodônticos. **Métodos:** Seis resinas foram utilizadas neste estudo: Z350 XT (3M), Transbond XT (3M), Vittra APS Unique (FGM), Filtek Bulk Fill Flow (3M), Orthocem (FGM) e Vitta APS Unique Flow (FGM). A estabilidade de cor, a resistência de união, a resistência flexural e módulo de elasticidade foram obtidos pelo espectrofotômetro, pela microtensile OM100 e pelo teste de flexão em 3 (três) pontos. Os materiais foram divididos em diferentes tipos de superfícies: resina composta Z250 XT (3M), dissilicato de lítio (e-max) e esmalte bovino. Foram utilizados água destilada e café como soluções de imersão para envelhecimento das resinas. Os dados foram analisados pelos testes ANOVA one-way/two-way e testes Tukey ($\alpha = 0,05$). **Resultados:** O café apresentou uma variação de cor significativamente maior que a solução controle ($P < 0,001$). Os grupos Vittra APS Unique e Filtek Bulk Fill Flow apresentaram maior variação de cor. Houve diferença significativa entre os tipos de superfícies ($p = 0,036$). Os grupos Z350 XT e Transbond XT apresentaram maior resistência flexural e módulo de elasticidade. **Conclusão:** As resinas foram consideradas acima do limiar de aceitação visual com o consumo de café. Filtek Z350 XT e Transbond XT mostraram-se mais eficazes para confecção de acessórios ortodônticos em comparação as outras resinas testadas.

Palavras chaves: *attachments*, resinas compostas, estabilidade de cor, resistência de união, resistência à flexão.

ABSTRACT

Introduction: The objective of this *in vitro* study was to evaluate the physical properties of different resins used in the fabrication of orthodontic attachments. **Methods:** Six resins were used in this study: Z350 XT (3M), Transbond XT (3M), Vittra APS Unique (FGM), Filtek Bulk Fill Flow (3M), Orthocem (FGM), and Vitta APS Unique Flow (FGM). Color stability, bond strength, flexural strength, and modulus of elasticity were obtained using a spectrophotometer, the microtensile testing machine OM100, and three-point bending test. The materials were divided into different types of surfaces: Z250 XT composite resin (3M), lithium disilicate (e-max), and bovine enamel. Distilled water and coffee were used as immersion solutions for aging the resins. Data were analyzed using one-way/two-way ANOVA and Tukey tests ($\alpha = 0.05$). **Results:** Coffee showed significantly greater color variation than the control solution ($P < 0.001$). The Vittra APS Unique and Filtek Bulk Fill Flow groups exhibited greater color variation. There was a significant difference between the types of surfaces ($p = 0.036$). The Z350 XT and Transbond XT groups showed higher flexural strength and modulus of elasticity. **Conclusion:** The resins were considered above the threshold for visual acceptance with coffee consumption. Filtek Z350 XT and Transbond XT proved to be more effective for the fabrication of orthodontic attachments compared to the other tested resins.

Keywords: *attachments*, composite resins, color stability, bond strength, flexural strength.

1. INTRODUÇÃO E REFERENCIAL TEÓRICO

Os tratamentos ortodônticos realizados por meio dos alinhadores invisíveis, requerem o uso de *attachments* para melhorar a eficácia da movimentação dentária, proporcionando apoio extra e atrito entre os alinhadores e os dentes. Eles são confeccionados por meio de resinas fotopolimerizáveis com carga, que possui uma cor próxima a coloração do dente do paciente e fixados com um sistema adesivo na superfície dentária (Barreda et al., 2017; Nojima et al., 2019). Segundo Weir (2017) a espessura, a forma e o espaço de colagem dos *attachments*, são o que determinam o seu efeito desejado e o resultado no movimento dentário. Desde que foram vistos como ferramentas essenciais, melhorando, auxiliando e guiando a força aplicada sobre os dentes, é muito importante que eles mantenham sua integridade e forma (Simon *et al.*, 2014; Dasy *et al.*, 2015; Rossini *et al.*, 2015). Esses acessórios não devem passar pelo processo tradicional de polimento, pois o seu design é predefinido na fase de planejamento e não podem ser modificados após a sua colagem (Barreda et al., 2017; Chami et al., 2022).

O uso dos *attachments* pode trazer um excelente controle em todos os tipos de movimentos dentários individuais, desde movimentos de intrusão, extrusão, inclinação não controlada e controlada, rotação e até translação de corpo. Entretanto, o design dos *attachments* e cada fase do tratamento com alinhadores, são importantes no resultado da movimentação dentária ao longo do tratamento (Moya, Zafra, 2021; Silva, Alessio Jr, 2023). Entre os tipos existentes de *attachments*, encontramos os convencionais (elipsoidal, retangular e retangular biselado) e os otimizados (rotação, extrusão anterior, extrusão multi-dentes, extrusão posterior, multiplano posterior, controle radicular anterior, multiplano anterior, apoio com formato elipsoidal e inseridos nos incisivos laterais superiores, mordida profunda, ancoragem, retração, controle de ancoragem, expansão posterior, expansão e rotação), sendo indicados com base as necessidades do paciente e do plano de tratamento (Silva, Alessio Jr, 2023).

Como critério de escolha, foi selecionado resinas de diferentes composições e polimerização, que apresentam alta resistência ao desgaste e que proporcionasse um melhor custo/benefício para os profissionais; além de que, são opções presentes e viáveis, de

marcas nacionais e internacionais, facilitando o dia a dia e otimizando o tempo clínico. Pensando em todos esses cuidados e na estética do paciente, foi selecionado resinas novas no mercado para que atendesse vários objetivos, como as unicromáticas, visto que elas conseguem mimetizar de forma mais natural a cor do dente.

Portanto, o objetivo deste estudo foi avaliar propriedades físicas das resinas utilizadas para confecção dos *attachments* em alinhadores invisíveis. As hipóteses nulas testadas eram de que não haveria diferença entre as propriedades físicas das resinas compostas e ortodônticas utilizadas para a confecção de *attachments* e que a resistência de união não seria influenciada pelo tipo de substrato.

2. CAPÍTULO ÚNICO

ARTIGO 1 - Comparative evaluation of the physical properties of different types of resins used in the fabrication of orthodontic *attachments*

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Comparative evaluation of the physical properties of different types of resins used in the
fabrication of orthodontic *attachments*

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Comparative evaluation of the physical properties of different types of resins used in the fabrication of orthodontic *attachments*

Abstract

Introduction: The objective of this *in vitro* study was to evaluate the physical properties of different resins used in the fabrication of orthodontic attachments. **Methods:** Six resins were used in this study: Z350 XT (3M), Transbond XT (3M), Vittra APS Unique (FGM), Filtek Bulk Fill Flow (3M), Orthocem (FGM), and Vitta APS Unique Flow (FGM). Color stability, bond strength, flexural strength, and modulus of elasticity were obtained using a spectrophotometer, the microtensile testing machine OM100, and three-point bending test. The materials were divided into different types of surfaces: Z250 XT composite resin (3M), lithium disilicate (e-max), and bovine enamel. Distilled water and coffee were used as immersion solutions for aging the resins. Data were analyzed using one-way/two-way ANOVA and Tukey tests ($\alpha = 0.05$). **Results:** Coffee showed significantly greater color variation than the control solution ($P < 0.001$). The Vittra APS Unique and Filtek Bulk Fill Flow groups exhibited greater color variation. There was a significant difference between the types of surfaces ($p = 0.036$). The Z350 XT and Transbond XT groups showed higher flexural strength and modulus of elasticity. **Conclusion:** The resins were considered above the threshold for visual acceptance with coffee consumption. Filtek Z350 XT and Transbond XT proved to be more effective for the fabrication of orthodontic attachments compared to the other tested resins.

Keywords: *attachments*, composite resins, color stability, bond strength, flexural strength.

INTRODUCTION

Orthodontic treatments carried out using invisible aligners necessitate the usage of attachments to enhance dental movement efficiency by providing additional support and friction between the aligners and teeth. These attachments are crafted from load-bearing light-cured resins, closely resembling the patient's tooth color, and affixed with an adhesive system onto the tooth surface (Barreda et al., 2017; Nojima et al., 2019). According to Weir (2017), the thickness, shape, and bonding space of attachments determine their desired effect and the outcome in dental movement. Being perceived as essential tools that improve, assist, and guide the force applied to the teeth, it's crucial for them to maintain their integrity and shape (Simon et al., 2014; Dasy et al., 2015; Rossini et al., 2015). These accessories shouldn't undergo traditional polishing processes as their design is predetermined during the planning phase and cannot be altered post-bonding (Barreda et al., 2017; Chami et al., 2022).

The utilization of attachments can provide excellent control over various types of individual tooth movements, including intrusion, extrusion, controlled and uncontrolled tipping, rotation, and even bodily translation. However, both the design of attachments and each phase of aligner treatment play significant roles in the outcome of dental movement throughout the treatment process (Moya, Zafra, 2021; Silva, Alessio Jr, 2023). Among the available attachment types are conventional ones (ellipsoid, rectangular, and beveled rectangular) and optimized versions (rotation, anterior extrusion, multi-tooth extrusion, posterior extrusion, posterior multiplane, anterior root control, anterior multiplane, ellipsoid-shaped support and insertion in upper lateral incisors, deep bite, anchorage, retraction, anchorage control, posterior expansion, expansion, and rotation), recommended based on the patient's needs and treatment plan (Thiesen, 2020; Silva, Alessio Jr, 2023).

Selection criteria involved choosing resins with different compositions and polymerization, ensuring high wear resistance and providing better cost-effectiveness for professionals. These options, available from national and international brands, ease daily practice and optimize clinical time. Considering these aspects alongside patient aesthetics, new resin types available in the market were selected to meet various objectives, such as monochromatic ones, capable of more naturally mimicking tooth color.

Therefore, the objective of this study was to evaluate the physical properties of the resins used for creating attachments in invisible aligners. The null hypotheses tested were that there would be no difference in the physical properties of composite and orthodontic resins used for attachment fabrication and that bond strength would not be influenced by the substrate type.

MATERIAL AND METHODS

This study utilized the following composite resins: Z350 XT (3M-ESPE), Filtek Bulk Fill Flow (3M), Vittra APS Unique (FGM), and Vittra APS Unique Flow (FGM); as well as orthodontic resins: Transbond XT (3M) and Orthocem (FGM) (Table 1). All materials were light-cured using a professional LED light-curing unit (Radii Xpert, SDI, Victoria, Australia) with a light intensity of 1500 mW/cm² for 20 seconds. Color stability was determined using a spectrophotometer. Bond strength was measured using the microtensile test. Additionally, flexural strength (MPa) and the modulus of elasticity (MPa) were determined through a three-point bending test. The materials were divided into different surface types: composite resin (Z250 XT), lithium disilicate (E-max), and enamel (bovine teeth). Two immersion mediums were employed for resin

Table 1. Resin composites composition

Material	Shade	Batch number	Composition (**)	Filler % (wt)	Manufacturer
Z350 XT	A1	2226200559 2303300808	Silica filler, zirconia filler, aggregated zirconia/silica cluster filler, BisGMA, UDMA, TEGDMA, PEGDMA e BisEMA.	78.5	3M Oral Care, St. Paul, MN, USA
Transbond XT	-	2233000625 2300500626	Silane-treated quartz, BisGMA, bisphenol-A bis (2 hydroxyethyl ether) dimethacrylate, silane-treated silica, diphenyliodonium hexafluorophosphate.	77	3M Oral Care, St. Paul, MN, USA
Vittra APS Unique Flow	unicromática	110822 030622 280223	Mixture of methacrylate monomers, photoinitiator composition (APS), co-initiators, stabilizers, silane, barium-aluminum-borosilicate glass, silicon dioxide.	58 - 62	FGM, Joinville, SC, Brazil
Filtek Bulk Fill Flow	A1	2225700490 2231100319 2302400257	Treated silanized ceramic, UDMA, Substituted Dimethacrylate, Ytterbium Fluoride, BisGMA, BisEMA, TEGDMA.	64.5	3M Oral Care, St. Paul, MN, USA
Orthocem	-	140722 170123	BisGMA, TEGDMA, methacrylate phosphatized monomers, stabilizers, canforoquinone, co-initiators, silicon dioxide loading and pigment.	48 - 50	FGM, Joinville, SC, Brazil
Vittra APS Unique	unicromática	251022 161122	Mixture of methacrylate monomers, photoinitiator composition (APS), co-initiators, stabilizers, silane, barium-aluminum-borosilicate glass.	72 - 80	FGM, Joinville, SC, Brazil
Ambar APS Universal	-	060223	MDP (10-methacryloyloxydecyl dihydrogen phosphate), methacrylate monomers, photoinitiators complex (APS), co-initiators, stabilizers, inert load (silica particles) and vehicle (ethanol).	-	FGM, Joinville, SC, Brazil

(**) Abbreviations: BisGMA, bisphenol A-glycidyl methacrylate; BisEMA, ethoxylated bisphenol-A dimethacrylate; PEGDMA, Polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

aging: distilled water (control) and coffee.

Tooth Selection

For this *in vitro* study, ten healthy bovine incisors with similar size and anatomical shape were selected, cleaned, and stored in a container with distilled water at a constant temperature of 37°C in a bacteriological incubator. Subsequently, the crowns were separated from the roots at the enamel-cement junction using a diamond bur (KG Sorensen, Barueri, SP, Brazil) under constant water cooling, leaving only the coronal portion for embedding and sample preparation.

Preparation of Samples for Microshear Bond Strength Evaluation (Mpa)

Sample preparation on bovine tooth surfaces

As described above, ten healthy bovine incisors of similar dimensions were selected and adequately prepared. The crowns were fixed onto a utility wax and enclosed within a standardizing device. Subsequently, they were embedded using a self-polymerizing crystal resin, which was manipulated and inserted inside PVC cylinders. After embedding, the enamel surface was abraded with silicon carbide sandpaper #600, 1200, 1500, and 2000 (Norton, Guarulhos, SP, Brazil) for 30 seconds for standardization, followed by polishing with 3 µm diamond paste (Arotec, Cotia, SP, Brazil).

The vestibular surface of the bovine enamel was conditioned with 37% phosphoric acid (Condac Porcelain, FGM, Brazil) for 15 sec, rinsed with water jets for 30 sec, and dried with compressed air. Two layers of adhesive were then applied for 10 sec each and dried with a gentle air stream (Ambar Universal APS, FGM, Brazil), followed by photoactivation for 10 sec for each tooth, by the manufacturer's instructions.

Sample preparation on lithium disilicate (e-max) surface

Ten lithium disilicate CAD/CAM ceramic blocks (IPS e.max CAD LT A2, Ivoclar Vivadent) were used as substrates for microshear bond strength testing (MPa). The metal base of the blocks was removed, and each sample was then cut to a thickness

of 2 mm using a precision metallographic cutter (Isomet 1000, Buehler, Lake Bluff, IL, USA). After achieving the desired thickness, the blocks were sent to a dental laboratory (located in Uberlândia, MG) for finishing and crystallization of the specimens. Subsequently, the samples were embedded in PVC cylinders using crystal resin (Aerojet, Santo Amaro, SP, Brazil).

Following the manufacturer's recommendations, the surfaces of the ceramic blocks were treated with 10% hydrofluoric acid (Condac Porcelain, FGM, Joinville, PR, Brazil) for 20 sec, cleaned with filtered water spray for 1 min, and dried with a jet of air. Next, a silane coupling agent (Prosil, FGM, Joinville, PR, Brazil) was applied to the ceramic surface for 1 min and dried with a gentle stream of air (11). After silane application, two layers of adhesive (Ambar Universal APS, FGM, Joinville, PR, Brazil) were applied for 10 sec each and dried with a gentle air stream for 10 sec to evaporate the solvent. Then, the adhesive was photopolymerized for 10 sec.

Sample preparation on composite resin surface

Ten composite resin discs were fabricated using Filtek Z250 XT shade A2 (3M Oral Care, St. Paul, MN, USA) with a matrix measuring 2 mm in height and 10 mm in internal diameter. To create the samples, we placed the matrices on a glass slide and applied increments of composite resin inside. Next, a polyester strip and a glass slide were placed on top of the composite resin to ensure a smooth surface, and they were then photoactivated for 20 sec. The photoactivation time adhered to the manufacturer's recommendations.

The composite resin discs were embedded in standardized PVC cylinders with a crystal resin. After embedding, the surfaces of the composite resins were abraded with silicon carbide sandpaper #600, 1200, 1500, 2000 (Norton, Guarulhos, SP, Brazil) for 30 sec for standardization, followed by polishing with 3 µm diamond paste (Arotec, Cotia, SP, Brazil).

Surface treatment was performed with 37% phosphoric acid (Condac Porcelain, FGM, Brazil) for 15 sec, followed by rinsing with water jets for 30 sec and drying with compressed air. Two layers of adhesive (Ambar Universal APS, FGM, Brazil) were then applied for 10 sec each and lightly dried with an air stream, following

the manufacturer's instructions (12). After the adhesive application, it was photoactivated for 10 sec.

Microshear Bond Strength

To insert the composite and orthodontic resins on the surface of different substrates (bovine tooth, ceramic, and composite resin), a PVC nasogastric microtube (4FR, Ningbo Foyomed Medical Instruments Co., Ltd., China) with an internal diameter of 1 mm and a height of 2 mm was used as a mold, respectively. Six specimens were made on the surface of each block, one specimen for each resin used in the study. Subsequently, the samples were light-cured for 20 sec. The microtube was then carefully removed with the aid of a scalpel blade, leaving the test specimens adhered to the substrate surface. To differentiate the resin group in each sample, six different colored markers were used, referencing each studied resin to a specific color. After preparing the test specimens, they were placed in containers and stored in distilled water at 37°C for 24 hours before undergoing the bond strength test (13).

The microshear bond strength test was performed using a Microtensile OM100 mechanical testing machine (Odeme Dental Research, Luzerna, SC, Brazil), in which the samples were placed in a specialized metallic device of the equipment, positioned in a way that they were aligned with the direction of the force application. A 0.2 mm diameter orthodontic wire (NiCr, Morelli, Sorocaba, SP, Brazil) was attached to encircle the specimens and the load plate of the machine. The equipment was set at a constant speed of 0.7 mm/min until displacement occurred. The bond strength values (MPa) for each sample were obtained using the following formula: $R = (F/A)$, where R is the bond strength, F is the force required for rupture (N), and A is the adhesive area of the specimens (mm²).

Color Stability

Sixty samples ($n=10$) were prepared using six types of restorative and orthodontic resins in color A1, as presented in Table 1. The samples were made with standardized measurements (8 mm in diameter and 2 mm in thickness) using a previously made mold, which had a central hole with these dimensions to accommodate the composite resins. After placing the resin in the mold's hole, a polyester strip was

positioned, and a glass plate was pressed onto the material. Subsequently, they were light-cured for 20 sec. The specimens were kept in Eppendorf tubes with distilled water for 24 hours before starting the test, at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$ in the absence of light.

Ten specimens from each of the six resins were divided into two groups for immersion, according to the following experimental solutions: G1 – Distilled Water (control) and G2 – Coffee (aging protocol). Next, all specimens underwent an initial color evaluation using a portable spectrophotometer (Ci6X, X-Rite Pantone®, Grand Rapids, MI, USA). The spectrophotometer measures colors using values corresponding to the CIE $L^* a^* b^*$ scale, where L^* indicates the brightness of the object, with an average ranging from 0 (absolute black) to 100 (absolute white). The a^* and b^* values correspond to hue, with a^* representing saturation on the red (positive a^*) or green (negative a^*) axis, and b^* on the yellow (positive b^*) or blue (negative b^*) axis (14).

After the initial color assessment, the samples were stored in individual Eppendorf tubes (2.0 ml) and completely immersed in the respective group solutions. These Eppendorf tubes were then placed in a plastic box with a lid and kept for six days at 37°C in an incubator. For the samples subjected to the coffee aging protocol, the solution was prepared by dissolving 3.0 g of coffee powder (Nescafé Original, Nestlé®, São Paulo, Brazil) in 150 ml of distilled water (15), heated to a temperature of 92°C . The beverages were renewed daily to prevent bacterial or yeast contamination, and with each change, the samples were rinsed with running water. According to Ertas et al. (16) and Chami et al. (7), one day of immersion in an incubator corresponds to 30 days (one month) of consumption. In other words, six days of immersion simulated six months of consumption of each beverage.

Subsequently, at the end of the six days of immersion, the specimens were washed with distilled water in an ultrasonic bath (Cristófoli, Campo Mourão, Paraná, Brazil) for 10 min and dried with absorbent paper. Then, to determine whether there was any staining, spectrophotometric analysis of the final color of the composite resins was performed using the same spectrophotometer and parameters. The color measurement after aging the samples was determined by the color difference or ΔE_{00} parameters calculated using the CIEDE 2000 method, represented by the equation:

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_{LSL}} \right)^2 + \left(\frac{\Delta C'}{K_{CSC}} \right)^2 + \left(\frac{\Delta H'}{K_{HSH}} \right)^2 + RT \left(\frac{\Delta C'}{K_{CSC}} \right) \left(\frac{\Delta H'}{K_{HSH}} \right) \right]^{1/2},$$

where the differences in brightness, chroma, and hue between the compared samples are represented by $\Delta L'$, $\Delta C'$, $\Delta H'$, and RT is the rotation function that depicts the interaction between chroma and tonal differences in the blue region. The weighting functions SL, SC, and SH are weighting factors, and the parametric factors KL, KC, KH are correction terms for experimental conditions.

Flexural Strength (FS) and Elastic Modulus (EM)

For these tests, the ISO 4049 standard was employed (17), and the flexural strength of all groups was determined using a three-point flexural test setup with a Universal Testing Machine (Instron ElectroPulsTM E3000, High Wycombe, UK) and using software (Bluehill Universal, Instron Training Center, Norwood, MA, USA) to calculate the values after setup. The tests were conducted at a speed of 0.5 mm/min and a load of 50N until failure. Sixty samples ($n=10$) were fabricated in stainless steel molds (25 x 2 x 2 mm) in a dark room with yellow light. The mold was placed on a glass plate with a polyester strip to prevent the presence of bubbles and acquire a smooth surface. After inserting the resin in a single increment into the mold, a second strip and glass plate were placed to remove excess material. The specimens were light-cured for 20 sec at three points of light, spreading throughout the length of the sample. The faces of the samples were sanded with #600 grit sandpaper (Norton, Guarulhos, SP, Brazil) to remove any excess resin and then stored in an incubator at 37°C for 24 hours.

The results of flexural strength were obtained using the following formula: $\alpha = 3FL/2bh^2$ where "F" is the maximum force applied (N); "L" is the distance between the support points (mm); "b" is the width (mm), and "h" is the thickness of the sample (mm). To measure the elastic modulus (MPa), the tip of the flexural deflectometer (W-E401-J, E-Series Deflectometer, Instron, Norwood, MA, USA) was fixed at the center of the specimen's base during the test, allowing for the measurement of deflection during the loading application. The elastic modulus (MPa) was determined using the following formula: $E = FL^3/4BH^3d$, where "F" is the maximum load (N); "L" is the length of the specimen (mm); "B" is the width of the sample (mm); "H" is the height of the sample (mm), and "d" is the deflection (mm) corresponding to the load F (18).

Statistical Analysis

After the results were analyzed using the Shapiro-Wilk normality test and found to have a normal distribution for color stability, bond strength, flexural strength, and modulus of elasticity tests, these data were subjected to a two-way analysis of variance (ANOVA) for color stability and bond strength and a one-way ANOVA for flexural strength and modulus of elasticity analysis. Subsequently, the Tukey test ($p < 0.05$) was applied to compare differences between the groups.

RESULTS

Color stability

The values of color change (ΔE_{00}) and standard deviations for the six resins and immersion solutions are presented in Table 2. Two-way ANOVA indicated a significant difference in the factor of attachments material, immersion solution, and the interaction between these two factors ($p < 0.001$). Coffee showed significantly greater color variation than the control solution ($p < 0.001$). The ΔE_{00} results ranged from 0.96 for distilled water (control) in the Orthocem group, considered perceptible and clinically acceptable, to 13.50 for coffee in the APS Unique group, considered extremely unacceptable (Figure 1). The APS Unique group, followed by the Filtek Bulk Fill Flow group, exhibited the highest color change when compared to the other resin groups during the immersion period.

Table 2. Mean (*SD*) of the color difference (ΔE_{00}) comparing attachments material and immersion solution.

Attachments material	Distilled water				Coffee			
Unique Flow	2.88	±	0.55	Aa	8.51	±	0.97	Bb
Transbond XT	2.81	±	0.42	Aa	7.70	±	0.74	Bab
APS Unique	2.38	±	0.63	Aab	13.50	±	1.54	Ba
Filtek Bulk Fill Flow	2.05	±	0.46	Aab	13.20	±	1.80	Ba
Z350 XT	1.71	±	0.64	Aab	6.38	±	0.91	Bc
Orthocem	0.96	±	0.51	Ab	9.58	±	1.50	Bb

Distinct letters (uppercase comparing immersion solution in row, lowercase comparing attachments material in column) indicate statistical difference at Tukey's test ($p < 0.05$).

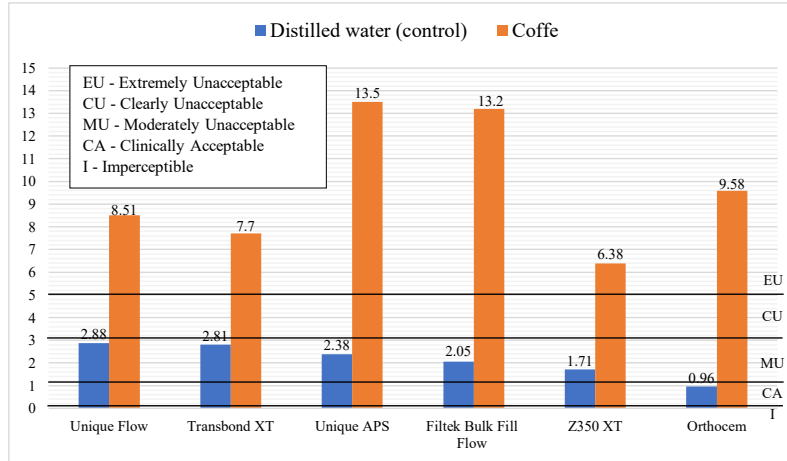


Figure 1. Ratings for perceptibility thresholds and visual acceptability for ΔE_{00} of composite resins in different immersion solutions (20).

Microshear bond strength

The mean values of bond strength and standard deviation for all resin groups and tested surface types are presented in Table 3. Two-way ANOVA found a significant difference between surface types ($p = 0.036$), but no significant differences were observed between the attachment material factor ($p = 0.053$) and the interaction between the attachment material factor and the surface factor ($p = 0.323$). When analyzing the different types of resins used for attachment fabrication, the Transbond XT group exhibited higher bond strength on the Emax and Enamel surfaces, while the APS Unique group showed higher values on the Resin surface. The grouped mean showed superior bond strength for the Emax group, which was similar to the Resin group, which in turn was similar to the Enamel group. The Enamel group was statistically different from the Emax group ($p = 0.029$).

Table 3. Mean ($\pm SD$) Microshear bond strength values (MPa) comparing attachment material and surface.

Attachment material	Resin			Dissilicate ceramic			Enamel		
Orthocem	19.60	$\pm$	5.51	18.40	$\pm$	2.83	17.20	$\pm$	4.11
Z350 XT	20.00	$\pm$	3.42	18.60	$\pm$	3.29	19.00	$\pm$	3.47
APS Unique	21.10	$\pm$	4.14	21.80	$\pm$	4.63	17.30	$\pm$	4.20
Unique Flow	18.70	$\pm$	4.65	21.10	$\pm$	4.13	18.90	$\pm$	4.22
Filtek Bulk Fill Flow	16.10	$\pm$	4.79	19.10	$\pm$	4.04	18.00	$\pm$	3.59
Transbond XT	19.50	$\pm$	5.31	23.50	$\pm$	5.10	20.00	$\pm$	4.75
Pool average	19.17	$\pm$	4.64	^{AB} 20.42	$\pm$	4.00	^A 18.40	$\pm$	4.06 ^B

Distinct letters indicate statistical difference at Tukey's test ($p < 0.05$).

Flexural strength (FS) and elastic modulus (EM)

The results for flexural strength and standard deviations for the six composite resins are shown in Table 4. Significant differences were found between the composite resins ($p < 0.001$). The Z350 XT group exhibited higher flexural strength, while the Orthocem and APS Unique groups showed lower flexural strength compared to the other groups.

The results for the modulus of elasticity and standard deviations for the six composite resins are also shown in Table 4. One-way ANOVA revealed significant differences between the composite resins ($p < 0.001$). The Z350 XT and Transbond XT groups had the highest modulus of elasticity values, the APS Unique, Orthocem, and Unique Flow groups had intermediate values, and the Filtek Bulk Fill Flow group had the lowest modulus of elasticity values.

Table 4. Mean ($\pm$ SD) Flexural strength and elastic modulus values (MPa) comparing attachment materials.

Attachment material	Flexural Strength (Mpa)				Elastic Modulus (Mpa)			
Z350 XT	118.00	$\pm$	18.20	A	8.82	$\pm$	0.92	A
Transbond XT	106.00	$\pm$	9.25	AB	9.23	$\pm$	1.26	A
Unique Flow	102.00	$\pm$	21.80	AB	4.25	$\pm$	0.93	C
Filtek Bulk Fill Flow	99.70	$\pm$	8.19	AB	2.77	$\pm$	0.42	D
Orthocem	89.50	$\pm$	25.60	B	5.60	$\pm$	1.10	BC
APS Unique	84.20	$\pm$	6.46	B	6.00	$\pm$	1.48	B

For each outcome (FS and EM), distinct letters indicate statistical difference at Tukey's test ($p < 0.05$).

DISCUSSION

The first null hypothesis was rejected because the groups showed different behaviors, with statistically significant differences in mechanical performance between the composite and orthodontic resins. The second null hypothesis was accepted because the bond strength exhibited similar behavior among the different types of substrates tested.

The CIEDE 2000 system was chosen because it is a standard method and is considered the most up-to-date formula regarding human eye perception (19). It is used to visually interpret color changes based on perceptibility and acceptability thresholds.

According to Paravina, Pérez, and Ghinea (20), color changes with ΔE_{00} values < 0.8 (clinically imperceptible) and ΔE_{00} values between 0.8 and 1.8 (perceptible but clinically acceptable) are currently accepted, where there is no need to replace the material. However, ΔE_{00} values > 1.8 are considered clinically unacceptable and are divided into moderately unacceptable ($\Delta E_{00} 1.8 > 3.6$), clearly unacceptable ($\Delta E_{00} 3.6 > 5.4$), and extremely unacceptable ($\Delta E_{00} > 5.4$). For all groups evaluated after aging in the coffee solution, ΔE_{00} obtained values greater than 5.4 (Figure 1), which is considered extremely unacceptable, confirming the coffee's staining effect on the resins and the need to replace these materials.

The color stability of resin may be associated with the structural composition of the matrix and its filler particles, depth of polymerization, and the type of dye (21,22). According to Chen (23), resins containing triethylene glycol dimethacrylate (TEGDMA) exhibit high levels of discoloration due to their hydrophilic character and greater water and water-soluble dye absorption capacity. Therefore, the more hydrophilic the resin, the greater its capacity to absorb water and water-soluble dyes, leading to more intense resin composite color (7). However, bisphenol A-glycidyl methacrylate (Bis-GMA) is a hydrophobic monomer and can also exhibit discoloration when exposed to solutions (24).

In this study, the nanoparticulate resin Filtek Z350 XT showed less pigmentation in the coffee solution, possibly due to its higher volume of filler content and smaller particles, which likely contribute to better mechanical properties, surface smoothness, and resistance to color changes (23). On the other hand, Filtek Bulk Fill Flow and Vittra APS Unique resins exhibited lower color stability when immersed in coffee. This higher degree of pigmentation in these resins was also observed in the studies by Karadas and Demirbuga (25), Oliveira et al. (26), and Pedrosa et al. (27), where they reported greater discoloration in Filtek Bulk Fill Flow resin. They believe that these different results may be influenced by various factors such as the type, size, and quantity of filler particles in the composite resins. Furthermore, according to Salgado et al. (28) and Oliveira et al. (26), the more translucent the restorative materials, the higher their susceptibility to color changes.

Regarding the monochromatic resin Vittra APS Unique, due to its new technology, there is limited information available about its use. Manufacturers provide

little detail about its exact composition, stating that it contains a mixture of methacrylate monomers but not specifying the types or concentrations, making interpretation of results challenging. In the present study, Vittra APS Unique composite exhibited lower color stability after aging in coffee, consistent with Martins (29). This author evaluated two composite resins immersed in solutions and described that the differences in results could be influenced by variations in the basic chemical composition of these resins, including matrix composition and photoinitiator characteristics when exposed to the challenges of storage in different solutions (27,29,30,31). Other studies described that the resin presented a combination of different photoinitiators and increased translucency of the material after polymerization (27,32).

Flexural strength represents the maximum stress that composite resins can withstand before failure, while the modulus of elasticity describes their rigidity (33,34). These characteristics are important to ensure that these materials offer sufficient flexural strength to withstand masticatory forces without fracturing. According to ISO 4049-2000 standards, composite resin materials evaluated using this standard methodology should have a minimum strength value of 80 MPa (17). Therefore, all groups tested in this study exhibited adequate flexural strength. In terms of flexural properties, Filtek Z350 XT and Transbond XT composites showed the highest flexural strength and modulus of elasticity compared to other resins. Previous studies have shown similar results, where these composites exhibited higher flexural strength and modulus of elasticity than bulk-fill composites (35,36) and low-shrinkage composites (36,37). Generally, the filler content is responsible for enhancing the mechanical properties of the material (38,39).

The modulus of elasticity is directly related to the volume and level of filler content in composite resins (40). Therefore, composites with higher filler volumes, such as Filtek Z350 XT (59.5% [vol]) and Transbond XT (70 to 80% [vol]), are expected to have higher resistance than those with lower filler volumes. Additionally, using an organic matrix composed of monomers with more rigid structures could contribute to achieving higher flexural properties (36,41). Thus, these observations justify the lower modulus of elasticity values found in the Filtek Bulk Fill Flow (42.5% [vol]) group in this study. Another study described that, when comparing different products, Filtek Bulk Fill Flow resin also exhibited lower modulus of elasticity values than conventional ones.

This is believed to be attributed to its relatively lower filler content and the matrices of the resins used (34).

For the fabrication of attachments, bond strength must be sufficient to withstand masticatory forces and stresses exerted by aligners. The microshear test aims to evaluate the bond strength and the maximum force that these materials can withstand before failure (42). Although there were no significant differences between the tested resins, the present study showed that Transbond XT composite exhibited higher bond strength, indicating good adhesion to e-max and enamel surfaces, consistent with the studies by Romano (43) and Souror, Maaly, and Khawandanah (44). For bonding various orthodontic appliances, a bond strength range of 6 to 8 MPa is required, which is considered suitable for most orthodontic requirements, as it can withstand orthodontic and masticatory forces (44). In this study, it was found that the bond strength of the tested resins was above this range. However, Scribante et al. (45) reported that to avoid enamel loss after debonding, adhesive forces should not be too strong (40–50 MPa).

When comparing different surface types, the substrates made of lithium disilicate ceramic (e-max) had slightly higher average bond strength than enamel substrates, likely due to the different surface treatments used in this study. While there is limited research on attaching to different surfaces, some scientific observations have shown that adhesion quality can be influenced by bonding mechanisms, and specific surface treatments are used to improve micromechanical or chemical retention of materials (46,47,48). Additionally, other authors reported that silane improved bond strength in applications on ceramics, regardless of surface preparation (49,50,51).

As a limitation of this study, it should be noted that it is an *in vitro* study and cannot precisely simulate the conditions of the oral cavity, such as the presence of saliva and patient oral hygiene. Additionally, the aging protocol based on Ertas et al.'s study (2006) should be clinically considered with caution and a critical scenario, as this hypothesis was solely related to coffee consumption, based on the assumption that the population's total coffee intake is approximately 3.2 cups/day with 15 min/cup consumption, without also considering the frequency with which the population consumes other immersion solutions. Several variables limit and can influence this study, such as evaluation time, choice of surface treatment method, evaluation methods (instrumental and/or visual), absence of information regarding the exact composition of

the resin, commercial brand of composite resin, material color, and even variations in composition that may affect mechanical capabilities.

Considering the study by Duc et al. (52), the use of unpolished samples to simulate the clinical conditions of resin composites in attachments was a notable strength. In this study, unpolished composite resins exhibited a 30% greater color change compared to polished samples, which could be attributed to the presence of free radicals on the unpolished surface (7). Finally, clinical studies should be conducted to confirm the results of this in vitro study, and further studies addressing various variables of these composites, especially those with single shade resins, are needed due to the scarcity of references on this new material.

CONCLUSION

According to this in vitro study, the following conclusions can be drawn:

- The composite resins used in this study were considered above the threshold of visual acceptance when exposed to coffee consumption, which may negatively impact aesthetics during orthodontic treatment.
- In decrescent order, Transbond XT and Filtek Z350 XT resins proved to be effective for the fabrication of orthodontic attachments compared to the other tested resins, showing better results in terms of color stability, bond strength, tension, and stiffness.

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