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Orientador: Prof. Dr. Murilo de Sousa Menezes

Banca Examinadora:
Prof. Dr. Murilo de Sousa Menezes
Prof. Dra. Gisele Rodrigues da Silva
Prof. Dr. Paulo César Freitas Santos Filho
Prof. Dr. André Luís Faria-e-Silva
Prof. Dr. Crisnicaw Veríssimo

Uberlândia, 2022

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Matrícula do Discente:	11813ODO015				
Nome do Discente:	Thaís Souza Maia				
Título do Trabalho:	Efeitos dos líquidos para modelagem nas propriedades físico-mecânicas de resinas compostas				
Área de concentração:	Clínica Odontológica Integrada				
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Projeto de Pesquisa de vinculação:	Propriedades Físicas e Biológicas dos materiais Odontológicos e das estruturas dentais				

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Dedico esse trabalho à minha mãe e às minhas tias professoras, verdadeiras guerreiras.

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EPÍGRAFE

“Se você tiver que confiar em alguém, confie em si mesmo.
Quem acredita sempre alcança”

Renato Russo

SUMÁRIO

Resumo

Efeitos da aplicação dos líquidos para modelagem nas propriedades físico-mecânicas de resinas compostas – THAÍS SOUZA MAIA – Tese de Doutorado – Programa de Pós-Graduação em Odontologia – Faculdade de Odontologia – Universidade Federal de Uberlândia.

RESUMO

O objetivo geral desse estudo foi investigar a influência da aplicação de diferentes materiais para modelagem (Composite Wetting resin, Scotchbond Multipurpose, Singlebond Universal) nas propriedades físico-mecânicas de resinas compostas (Vittra, Z350 XT, Forma). Este estudo foi dividido em 3 capítulos, de acordo com cada objetivo específico: **capítulo 1)** avaliar a rugosidade superficial e parâmetros de cor de diferentes líquidos para modelagem em resina composta submetida a exposição de manchamento e escovação simulada; **capítulo 2)** avaliar o efeito de materiais para modelagem na dureza, ângulo de contato, rugosidade superficial, e na formação de biofilme bacteriano em resinas compostas; **capítulo 3)** avaliar o efeito de agentes adesivos na resistência de união e no modo de falha do reparo imediato de resina composta, por meio do ensaio mecânico de microcisalhamento e microscopia óptica. As variáveis respostas obtidas foram rugosidade superficial, alteração de cor, índice de brancura, opacidade, topografia de superfície, dureza, ângulo de contato, adesão bacteriana por meio da contagem de unidade formadora de colônia e formação de biofilme, resistência adesiva e padrão de falha. Após a análise estatística dos dados, conclui-se que os adesivos testados Scotchbond Multipurpose e Singlebond Universal conferiram menores valores de rugosidade superficial, melhor estabilidade de cor, maior índice de brancura e os menores valores de opacidade após ao manchamento com vinho tinto e escovação simulada; apresentaram ainda redução na dureza, aumento do ângulo de contato e não influenciaram negativamente na rugosidade superficial e adesão bacteriana das resinas compostas; e apenas o procedimento de limpeza com jato de ar pode ser realizado para alcançar uma resistência de união aceitável no reparo imediato da resina composta, dispensando a utilização de um agente de união.

PALAVRAS-CHAVE: resinas compostas, cor, aderência bacteriana, resistência ao cisalhamento.

Abstract

ABSTRACT

The main objective of this study was to investigate the influence of the application of different materials for modeling (Composite Wetting Resin, Scotchbond Multipurpose, Singlebond Universal) on the physical- mechanical properties of resin composites (Vittra, Z350 XT, Forma). This study was divided into 3 chapters, according to each specific objective: chapter **1)** to evaluate the surface roughness and color parameters of different modeling liquids of resin composite subjected to staining and simulated toothbrushing; chapter **2)** to evaluate the effect of modeling liquids on hardness, contact angle, surface roughness, and bacterial biofilm formation in resin composites; chapter **3)** to evaluate the effect of modeling agents on the bond strength (μSBS) and on the failure mode of the immediate repair of resin composite, through the microshear mechanical test and optical microscopy. The response variables used were surface roughness, color stability, whitening index, opacity, surface topography, hardness, contact angle, bacterial adhesion by colony forming unit count and biofilm formation, bond strength and failure mode. After statistical analysis of the data, it was concluded that the tested Scotchbond Multipurpose and Singlebond Universal adhesives provided lower surface roughness values, better color stability, higher whitening index and lower opacity values after staining with red wine and toothbrushing; they also showed a reduction in hardness, an increase in the contact angle and did not negatively influence the surface roughness and bacterial adhesion of the resin composites; and only the air jet cleaning procedure can be performed to achieve an acceptable bond strength in the immediate repair of the resin composite, without the use of a bonding agent.

KEYWORDS: Composite resins; Surface properties; Color; Hardness; Wettability; *Streptococcus mutans*

INTRODUÇÃO E REFERENCIAL TEÓRICO

1. INTRODUÇÃO E REFERENCIAL TEÓRICO

As resinas compostas avançaram em longevidade e durabilidade nas últimas duas décadas e se tornaram o material restaurador mais comumente utilizado tanto para procedimentos minimamente invasivos como para reabilitações estéticas mais complexas.[1-3] A qualidade da camada superficial das resinas compostas tem um efeito decisivo na estética das restaurações, bem como na estabilidade de cor, e pode ser afetada pelo tamanho das partículas de carga e composição da matriz orgânica.[4] Ao longo do tempo, restaurações com resinas compostas tornam-se menos estáveis devido à exposição prolongada a solventes orgânicos e cargas mecânicas na cavidade bucal[5,6], o que pode promover degradação hidrolítica. O monômero amplamente utilizado em materiais restauradores odontológicos é o bisfenol dimetacrilato diglicidil (BisGMA), que por apresentar alto peso molecular e consequentemente alta viscosidade, é associado a monômeros diluentes de menor viscosidade para melhorar o grau de conversão, e também reduzir componentes capazes de sofrer lixiviação.[7] A inclusão de monômeros adicionais na composição química destes materiais pode alterar distintas características, como a sua reação de polimerização e as propriedades mecânicas.[8,9]

Outros fatores inerentes às resinas compostas no meio bucal são a ação de enzimas salivares, variações no pH e temperatura, presença de dieta cariogênica, acúmulo de biofilme, desgaste por pastas dentárias abrasivas e pela escovação dentária, além da fadiga oclusal.[10,11] Existem ainda fatores dependentes do profissional, sendo estes relacionados com a habilidade de quem as executam, e podem ser reduzidas ou controladas por meio da escolha correta do material e da técnica restauradora. Combinados, todos estes aspectos influenciam de alguma forma na contração do polímero e, consequentemente, na tensão de contração residual, que por sua vez podem desenvolver tensão na interface dente/restauração, redução da adesão, infiltração marginal, sensibilidade pós-operatória e/ou cáries secundárias.[12,13]

Considerando as técnicas restauradoras utilizadas para restaurações com resinas compostas, um desafio da técnica incremental é a viscosidade demasiada de alguns compósitos, que pode dificultar a aderência da resina

composta no instrumental odontológico, a adaptação às paredes cavitárias e ainda dificultar o processo de escultura e definição das margens das restaurações.[14,15] Por consequência, uma restauração em resina composta que não apresenta um selamento marginal satisfatório pode se tornar um fator de susceptibilidade à formação de fendas e poros ou defeitos coesivos entre as camadas de resina, sendo um fator determinante para penetração de fluidos orais e bactérias[16,17] e fator de predisponibilidade à desmineralização dentária.

No intuito de melhorar a adversidade relacionada ao manuseio da resina composta, foram propostos líquidos específicos para modelagem.[18] Tais líquidos, possuem em sua composição química monômeros diluentes como o trietileno glicol dimetacrilato (TEGDMA) e uretano dimetacrilato (UDMA)[19,20] de acordo com os fabricantes. Sua utilização visa facilitar a modelagem da resina composta antes da técnica de polimerização.[20] A inserção dos líquidos para modelagem no mercado odontológico ocorreu nas últimas décadas, porém o conceito de lubrificar a espátula de manipulação para favorecer o manuseio de resinas compostas é encontrado na literatura há mais tempo. Por volta da década de 1980, já existiam estudos com o uso de álcool, acetona ou mesmo adesivos para este fim[21-23], embora seja uma manobra que não é sugerida pelos fabricantes.

Complementarmente, a vantagem dos chamados líquidos para modelagem é que, ao contrário de alguns sistemas adesivos, são livres de 2-hidroxietil metacrilato (HEMA), minimizando a sorção de água, e consequentemente o efeito de degradação ao longo do tempo.[24] Outro ponto importante é o efeito benéfico na redução de defeitos superficiais presentes na restauração,[14] como bolhas e poros no corpo da restauração, mas pouco se sabe ainda acerca de seu efeito na adesão bacteriana. O microrganismo *Streptococcus mutans* é considerado um dos principais agentes etiológicos da cárie dentária.[25] Este se adere nas superfícies duras formando as placas bacterianas cariogênicas e inicia a produção de ácidos e enzimas degradantes que influenciam na longevidade das restaurações.[26] Estudos relatam que as características das resinas compostas influenciam na adesão, de maneira que os materiais hidrofílicos atraem mais biofilme oral do que os hidrofóbicos[27,28], e

as superfícies rugosas mais do que as superfícies lisas.[29,30] No entanto, existem controvérsias que relatam a insignificância entre rugosidade da superfície e quantidade de adesão bacteriana[25,31] nas resinas compostas. Nesse sentido, surge a necessidade de uma investigação acerca dos monômeros hidrofílicos e dos solventes presentes nos adesivos que são expostos na camada superficial quando utilizados como líquidos para modelagem[32-34].

Tendo em vista a utilização cada vez mais frequente dos líquidos para modelagem e de diferentes resinas na construção de restaurações estéticas, é de suma importância compreender melhor as propriedades físicas[35-37] que afetam a capacidade de combinar resinas compostas com líquidos para modelagem. Logo, considerando o uso desses materiais pelos profissionais e a escassez de informações das possíveis implicações clínicas, este estudo foi concebido para investigar particularmente o efeito do uso de diferentes líquidos para modelagem na superfície de diferentes resinas compostas. A aplicação dos diferentes líquidos para modelagem não influenciará nas propriedades físico-mecânicas das resinas compostas testadas.

Capítulos

2. CAPÍTULOS

Essa tese foi subdividida em três capítulos:

2.1 Capítulo 1) Artigo aceito no periódico Brazilian Oral Research*:* "EFFECT OF MODELING LIQUIDS ON RESIN COMPOSITE ROUGHNESS AND COLOR PARAMETERS AFTER STAINING AND TOOTHBRUSHING".

O objetivo deste estudo foi avaliar a rugosidade superficial, estabilidade de cor, índice de brancura e opacidade de diferentes líquidos para modelagem aplicados em resina composta submetida a exposição de manchamento e escovação simulada.

2.2 Capítulo 2) Artigo nas normas para ser submetido ao periódico Journal of dentistry: *"*INFLUENCE OF MODELING LIQUIDS ON PHYSICAL PROPERTIES AND ADHERENCE ANALYSIS OF STREPTOCOCCUS MUTANS IN THE RESIN COMPOSITES".

O objetivo deste estudo foi avaliar o efeito de líquidos para modelagem na dureza, ângulo de contato, rugosidade superficial, e na formação de biofilme bacteriano em resinas compostas.

2.3 Capítulo 3) Artigo nas normas para ser submetido no periódico Journal of Esthetic and Restorative Dentistry*:* "EFFECT OF THE RESIN COMPOSITE IMMEDIATE REPAIR USING DIFFERENT MODELING LIQUID ON THE BOND STRENGTH".

O objetivo deste estudo foi avaliar o efeito de agentes adesivos na resistência de união (μSBS) e no modo de falha do reparo imediato de resina composta, por meio do ensaio mecânico de microcisalhamento e microscopia óptica.

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Capítulo I

2.1 Capítulo 1

Original Research Report

Thematic area: Dentistry

Effect of modeling liquids on resin composite roughness and color parameters after staining and toothbrushing

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Effects of modeling liquids on resin composite roughness and color parameters after staining and toothbrushing

Abstract: This study evaluated surface roughness, color stability, whitening index, and opacity of different types of modeling liquids for resin composite coating after exposure to staining and toothbrushing. Disc-shaped resin composite (Vittra APS, FGM) specimens were fabricated and divided into four groups (n=10 each): control group, Composite Wetting resin (Ultradent Products), Adper Scotchbond Multipurpose adhesive (3M ESPE), and Adper Universal adhesive (3M ESPE). Surface roughness (Ra) was measured using a rugosimeter, while color stability (ΔE_{00}), whitening index (WI), and opacity (%) were measured using a spectrophotometer. Assessments were made at four time points: after polishing (baseline, T1), after immersion in red wine for 24 h (T2), and after 5,000 (T3) and 10,000 (T4) cycles of toothbrushing. Scanning electron microscopy images were captured to analyze the scratches created. The data were statistically analyzed by two-way repeated-measures analysis of variance and Tukey's honestly significant difference tests ($\alpha = .05$). Modeling with the Wetting resin resulted in higher surface roughness ($p < 0.05$) and low color stability, which were attributable to porosities. Higher color change values were observed in the control group after staining. Both adhesives showed the lowest mean ΔE_{00} values ($p < 0.005$). WI decreased after staining, except with the use of the Universal adhesive ($p < 0.005$). The lowest opacity values were observed at baseline for all groups ($p < 0.005$). The Universal and Scotchbond adhesives had lower surface roughness, better color stability, higher WI, and the lowest opacity values after staining with red wine and toothbrushing.

Keywords: Resin composites; Color; Toothbrushing; Wine.

Introduction

Resin-based composites has become the most common restorative material used in anterior and posterior teeth because of its wide clinical applicability, excellent esthetics, acceptable biocompatibility, and appropriate physical and mechanical properties.[1-3] However, composites undergo constant degradation when exposed to different conditions in the oral cavity.[4,5] Changes in pH, absorption of pigments present in beverages and foods, and toothbrushing, among other factors, can cause the loss of restorative material and tooth structures.[5-7]

Surface quality of resin composites plays a significant role in maintaining the esthetic appearance of restorations. This quality is key in patients' perception of and dissatisfaction with restorations; it is also a the major reason for frequent replacement of restorations.[7-10] Many factors can lead to loss of surface quality — mainly, changes in surface color and roughness.[11,12] A rougher surface is more prone to plaque accumulation,[11] may have a deleterious effect on the wear of the antagonistic natural teeth, reflects a lesser amount of light than smooth surfaces, and increases tooth staining.[4,7,11,13-15]

Owing to an increase in patients' high esthetic demands and the pursuit of a harmonious smile, techniques and materials are continually being improved and developed, thereby enabling restorative dentists to leverage the direct composite technique.[8,9] Nevertheless, this technique has a learning curve as it requires operator skill at handling the material and the sticky consistency of several composites can hinder their placement and sculpting.[16,17] Therefore, specific low-viscosity liquids are available for relatively easy build-up restorations.[18-20] These liquids, applied during or over the last increment while building up a restoration, are beneficial to reducing tooth surface tension,

which smooths the incremental layer of the resin composite, improves the surface adaptability of the composite, and fills microstructural defects, having a sealing effect.[19-22] While some clinicians have used specific liquids for modeling resin composites, the use of adhesives has gained popularity for this purpose.[19,20]

In this context, the use of a modeling liquid to increase the handling of the final composite increment can improve some of its physical properties.[20,23-25] The present *in vitro* study compared the surface roughness, color stability, whitening index (WI), and opacity of different modeling liquids subjected to erosive and abrasive challenges by staining and toothbrushing simulation to answer the following question: "Does the application of modeling resin on composite restorations maintain their optical properties after simulated degradation by combining red wine staining and toothbrushing?". The first null hypothesis was that surface roughness would not vary between the different types of modeling liquid coatings. The second null hypothesis was that staining and toothbrushing would not influence the color stability of resin composite specimens coated with modeling liquids.

Methodology

Specimen preparation

Disc-shaped specimens (8-mm ø × 1.5-mm height) were built up in a single increment of resin composite (Vittra; A2 for enamel; FGM, Joinvile, Santa Catarina, Brazil). After inserting the increment into a Teflon matrix, the excess composite was removed by moving a glass plate parallel to the surface of the matrix. A spreadsheet (Excel; Microsoft New Mexico, USA) containing random numbers was used to randomly

allocate the specimens into one of the four experimental groups (n=10 each), according to the modeling liquid used. One group served as the control (no model liquid) and three groups received a type of modeling liquid, as follows: Composite Wetting resin (Ultradent Products Inc., South Jordan, Utah, USA), Adper Scotchbond Multipurpose (3M ESPE, St. Paul, Minnesota, USA), or Adper Universal (3M ESPE, St. Paul, Minnesota, USA). The polymer matrix composition, filler characteristics, and content are displayed in Table 1.

Except for the control, the composite surface was smoothed using a brush (#4, Kota, Cotia, São Paulo, Brazil) and covered with the modeling liquid randomized for each experimental condition. The modeling liquid was applied with the brush performing six movements in the same direction to prevent the formation of porosities and to obtain a surface similar to that observed clinically. The adhesives were agitated before application and the solvent was evaporated using a gentle air blast for 5 s before light-curing. The increment was individually light-cured with a Valo LED-based unit (irradiance, 1000 mW/cm^2; Ultradent Products Inc., South Jordan, UT, USA) for 20 s. After storage in an incubator (Solab, Piracicaba, São Paulo, Brazil) in distilled water at 37.7 °C for 24 h, the specimens were polished with a series of aluminum oxide discs (medium, fine and, extra-fine abrasiveness; Sof-lex, 3M ESPE, St. Paul, Minnesota, USA) for 20 s per disc by a single trained operator. Subsequently, each disc was washed for 20 s. Upon the conclusion of the polishing cycle, the specimens were immersed in an ultrasonic bath (Thornton, Vinhedo, São Paulo, Brazil) for 10 min. The final thickness of each specimen was measured using a digital caliper (Absolute AOS Digimatic, Mitutoyo, Tokyo, Japan), and specimens < 1.45 mm or > 1.55 mm were replaced. All measurements were performed at

baseline (after polishing, T1), 24 h after specimen immersion in red wine (T2),[24] and after 5,000 (T3) and 10,000 (T4) brushing cycles.[13]

Surface roughness measurement

The surface roughness (Ra) of each specimen was obtained using a surface roughness tester (Surftest 301 J, Mitutoyo, Kanagawa, Japan) at a speed of 0.25 mm/s, using a cut-off of 0.8 mm. The mean value of three readings was computed and used for subsequent statistical analysis.

Measurement of color parameters

Color parameters were measured using a digital spectrophotometer (SP64, X-Rite, Grand Rapids, MI, USA) in reflectance mode, with a D65 illuminant, and a wavelength range of 400–700 nm, including a specular light (SPIN mode), and an observer angle of 10°. The L*a*b* color system defined by the Commission Internationale de l'Éclairage (CIE) was used. This system consists of three parameters, where L* indicates lightness (black to white) and a* and b* are the chromaticity coordinates for the red-green and yellow-blue axes, respectively. The color measurements were performed in triplicate for each specimen, and the mean values were recorded as L0*, a0*, and b0*. The color parameters were measured against white ($L^*_{white} = 86.70$, $a^*_{white} = -1.17$, $b^*_{white} = 1.60$) and black ($L^*_{black} = 29.96$, $a^*_{black} = 0.42$, $b^*_{black} = 1.49$) backgrounds to obtain the opacity of the specimens, which was auto-calculated using a spectrophotometer. The device was adjusted to a small-area view, with a total area of 4 mm. The WI was calculated using the following formula:[26]

$$[\text{Formula 1}]\ WI = 0.551 \times L\ - 2.324\ \times a - 1.1\ \times b$$

Staining procedure

The specimens were embedded in transparent nail polish to cover the unpolished surfaces during the staining procedure. The specimens were immersed in plates containing 10 mL of red wine (Cabernet Sauvignon Concha Y Toro Reservado, Concha y Toro, Chile) and kept in an incubator at 37.7 °C for 24 h.[24] The pH of the wine (2.6) was measured using a pH meter (JK-PHM-005, JKI, Shang Hai, China). After staining, the specimens were subjected to ultrasonic cleaning in distilled water for 10 min and dried before repeating the measurement of all parameters (T2).

Toothbrushing cycles

The specimens were subjected to mechanical brushing with soft-bristled toothbrushes (Colgate Essential Clean, Colgate Oral Pharmaceuticals Inc, Toronto, Ontario, Canada Inc, lot No. PBR5311687) attached to a toothbrushing simulation device (Odeme, Luzerna, SC, Brazil). The toothbrush heads (one per specimen) were cut off and then fitted into the clamp of the machine. The toothbrushes moved back and forth horizontally at 2.5 cm/s under a 200 g load. As 10,000 to 14,600 brushing cycles are considered equivalent to 1 year of *in vivo* toothbrushing,[13] 5,000 and 10,000 cycles were performed to simulate 6 months and 1 year of brushing, respectively. After the first 5,000 cycles, the brushes were replaced. A dentifrice (Colgate Total 12, Colgate Palmolive, Canada) was used to make a slurry (90 g of dentifrice in 180 mL of distilled water) with which the specimens were brushed. After 5,000 cycles, the specimens were subjected to ultrasonic cleaning for 10 min to remove dentifrice remnants. At the end of each set of 5,000 brushing cycles, new measurements were performed (T3 and T4).

Color changes

The overall color changes (ΔE_{00}) caused by the staining procedures and brushing cycles were calculated for T2, T3, and T4 using the following formula[27]:

$$[\text{Formula 2}]\ \Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \frac{\Delta C'}{K_C S_C} \frac{\Delta H'}{K_H S_H}}$$

where $\Delta L'$, $\Delta C'$, and $\Delta H'$ are the changes in lightness, chroma, and hue, respectively. SL, SC, and SH are weighting functions for each component. RT is the interaction term between the chroma and hue differences. Although CIE76 (ΔE_{ab}) has been widely used in previous studies, the formula CIEDE2000 (ΔE_{00}), was chosen because it reflects the color differences perceived by the human eye better than CIE76 (ΔE_{ab}).[28]

Topographical analysis

Surface topography was analyzed, relative to smoothness and scratches, using scanning electron microscopy (EVO MA 10, Carl Zeiss, London, UK). One specimen per experimental condition was randomly selected and sputter-coated with gold/palladium for 120 s. Images were obtained at 20 kV at a working distance of 12 mm and ×5000 magnification.

Statistical analysis

The data for each outcome were individually analyzed by two-way repeated-measures analysis of variance after defining the "assessment time interval" as a repetition factor. Normal distributions and equal variances of the data were analyzed using Shapiro-

Wilk and Levene's tests, respectively. Multiple comparisons were performed by Tukey's honestly significant difference tests. The significance level was set at $\alpha = 0.05$ for all analyses.

Results

Surface roughness

The results showed that only the "treatment" ($p = 0.001$) affected roughness; however, roughness remained unchanged upon evaluation by the "assessment time intervals" ($p = 0.193$). The interaction between the evaluated factors was also nonsignificant for the roughness values ($p = 0.226$) (Table 2). Irrespective of the assessment time interval, the use of Wetting resin resulted in rougher surface values compared to those yielded by the Scotchbond and Universal adhesives. Intermediate Ra values were observed for the control, without significant differences for the other treatments.

Color parameters

Both "treatment" ($p < 0.001$) and "assessment time interval" ($p < 0.001$) affected the overall color changes (ΔE_{00}), with a significant interaction between these factors ($p = 0.009$) (Table 3). Higher color change values were observed in the control group after the staining procedure. The specimens modeled with adhesives had similar and the lowest mean color change values. Similar results were observed at T4, but the specimens modeled with the Universal adhesive showed color changes similar to those observed for the control specimens and those modeled with Wetting resin. Except in the case of the Wetting resin, toothbrushing of the specimens reduced the color changes produced by the

staining procedures. However, all final values were beyond the ΔE_{00} acceptability threshold ($\Delta E_{00} = 1.77$).[29]

Figure 1 shows the color parameters measured throughout the experiment. Irrespective of treatment, a reduction in lightness of baseline values was observed after the staining procedure. In general, while toothbrushing of the specimens increased their lightness, the final values remained lower than those observed at T1. When the specimens were modeled with adhesives (highest a* values at baseline), the staining procedure increased the specimen's redness values, which were reduced by toothbrushing. A slight reduction in redness was observed in the control specimens after toothbrushing, while the a* values remained stable for specimens modeled with Wetting resin throughout the experiment. Except for the Universal adhesive, the staining procedures increased the yellowness of the specimens and produced slight changes in b* values observed after toothbrushing.

Cylinders were drawn using CorelDraw Graphics Suite X8 (Corel Corporation, Ottawa, ON, Canada) and colored with the RGB values calculated previously to facilitate the visualization of color changes that occurred during the experiment (Figure 2). The discs in the Wetting resin group exhibited intermediate changes in color. The Scotchbond and Universal specimens were a shade lighter than the control and Wetting resin specimens (Figure 2).

Both "treatment" ($p < 0.001$) and "assessment time interval" ($p < 0.001$) affected the WI, with a significant interaction between these factors ($p < 0.001$). The WI results are presented in Table 4. Except for the Universal adhesive (stable WI), the staining procedure caused a WI reduction, whereas toothbrushing cycles did not increase the WI. At other assessment time intervals, specimens modeled with the Scotchbond and

Universal adhesives showed similar WI values and were a shade whiter than those that received the other treatments.

While both "treatment" ($p < 0.001$) and "assessment time interval" ($p < 0.001$) affected opacity, the interaction between these factors was not significant ($p < 0.785$) (Table 5). Irrespective of treatment, the lowest opacity values were observed at T1. Modeling the specimens with either Universal or Scotchbond adhesives resulted in more translucent specimens compared to the control specimens. The use of the Wetting resin did not change the opacity observed in the control specimens.

Topographical analysis

The scanning electron microscopy images (Figure 3) showed that the Wetting resin had the most irregular surface among the groups, observed immediately after 24 h of immersion in red wine. All groups showed some degradation, resulting in irregular surfaces, superficial scratches, and areas of debonding, after staining and toothbrushing. However, there were limited specific differences between the adhesive groups, and so it was hard to differentiate them from each other.

Discussion

The present study compared surface roughness, color stability, WI, and opacity of different modeling liquids after staining and toothbrushing challenges. In this study, all null hypotheses were rejected. The results demonstrate different degrees of color change after immersion in red wine, depending on the material. Use of the Wetting resin increased the material's susceptibility to surface roughening and color changes compared

to the other adhesives. Interestingly, the staining procedure resulted in reduced roughness when a Wetting resin or an adhesive was used.[30] The ethanol content and low pH of wine led to resin matrix degradation;[31] thus, the use of a modeling liquid might help prevent this adverse effect by reducing the occurrence of porosities on the composite surface.[32]

The differences in roughness, discoloration, and other color parameters between the modeling resin and adhesives indicated the importance of the composition of these materials.[33] Among resin-based dental composites, specifically the Wetting resin, resin monomers containing diurethane dimethacrylate (UDMA) have a high molecular weight, which increases the viscosity of this material. Moreover, the Wetting resin contains triethylene glycol dimethacrylate (TEGDMA) in the same proportion as UDMA. TEGDMA is more sensitive to changes in pH and solvent composition; therefore, it may potentially absorb and react with pigments.[34] Our findings suggest that the changes in color and roughness were more affected by the viscosity of the modeling materials than by the presence of solvents in their composition. Despite the presence of an acidic functional monomer, using a Universal adhesive as a modeling liquid, there were smaller changes in color and roughness, which were similar to those observed in the use of the Scotchbond. A previous study has also reported the reliability of the Universal adhesive use for this purpose.[6] In addition, Scotchbond as modeling liquid has already been shown not to affect the cohesive strength of the resin composite.[18] 2,2-bis-[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]-propane) (bis-GMA) and hydroxyethylmethacrylate (HEMA), without the combination of solvents, form molecules with high molecular weight and, consequently, a better bond at the interfaces.[18] It is known, however, that the presence of solvents can compromise some mechanical properties, which were not evaluated in the present study.

In general, we found a higher degree of staining in the control group after immersion. Moreover, toothbrushing was not effective in reducing this color change. This can be problematic in patients with resin composite restorations in the esthetic zone. Other studies on these color changes have immersed test specimens in various solutions.[7,12,15] In the present study, the specimens were continuously immersed in red wine for 24 h; thus, it was possible to combine the effects of staining, erosion, and degradation, as wine is acidic. Acidic beverages commonly consumed by people negatively influence the physical and mechanical properties of composites.[5]

Most studies have attributed the changes in specimen color to the effects of experimental staining challenges, without considering the influence of toothbrushing. Therefore, another important observation from the present study was that the toothbrushing procedures allowed the inclusion of another condition of the oral environment; namely, the abrasive challenge. Simulated toothbrushing reduced the color changes caused by immersion in red wine but did not increase WI. All values of ΔE_{00} in this study exceeded the acceptability threshold ($\Delta E_{00} = 1.77$). This threshold was defined as the color difference between two objects, which required acceptance by 50% of observers to consider it clinically acceptable.[29] The surface stains caused by the staining protocol used in the present study were removable; and they were removed using a toothbrush and regular dentifrice, consistent with the findings of other studies.[12,15] Nevertheless, red wine had the highest staining potential.[5] The final mean values of all the groups were considerably above the confidence interval (CI: 1.23–2.37).[29] Thus, in the present study, wine caused an irreversible stain that could not be completely removed to increase the WI. The dentifrice used in the present study had a relative dentin abrasion index of 70 (the scale ranges from 0 to 250), which is considered moderately abrasive.

This dentifrice was chosen because it is commonly used and available to patients; however, it did not have sufficient abrasive potential to remove the surface staining caused by wine.[13,14]

As the composite surfaces were polished, the differences in smoothness among the treatments could be related to changes in the composite properties caused by the modeling liquid. The Wetting resin showed higher surface roughness and color change values compared to those in the other treatments. The higher viscosity level of this resin may have contributed to this finding because it produced irregular surface thickness owing to air bubbles trapped within the coating layer.[21] The numerous porosities present on the surface after the Wetting resin application with a brush, as well as the large voids resulting from the abrasion of the organic matrix and removal of inorganic fillers from the surface during polishing and toothbrushing, may also have contributed to these findings. These surface porosities caused losses of mass and water sorption, which may have promoted higher roughness and color change.[25] However, none of the modeling liquids used in the present study reduced the roughness measured at baseline compared to the control. Thus, the findings suggest the need to polish restorations even when a modeling liquid is used.

The possible explanations for the reduction in surface roughness and susceptibility to staining between the two types of adhesives tested include the following: low viscosity, which reduced the presence of defects in the bulk of the composite, and the relative hydrophobic composition, which may have protected the composite from hydrolysis and further deleterious effects.[25] Despite the presence of hydrophilic monomers and solvents, the Universal adhesive showed higher color stability in the present study. The predominance of 60–70% of BISGMA monomer resulted in higher

viscosity in the Scotchbond adhesive, when compared to the Universal adhesive, which contained only 15–25% of BISGMA. Another explanation for the better outcomes of the Universal adhesive could be the higher b* values of the Universal adhesive, which were probably directly related to the greater amount of amine in the material.[35]

A limitation of the present study was that the specimens were immersed in red wine for a long period that did not reproduce the clinical environment. Therefore, the color changes observed in this study were likely overestimated. Moreover, the data observed for the materials evaluated in the present study cannot be extrapolated to other materials because differences in composition could affect the outcomes. Besides the afore-mentioned effect of the resin monomer composition, the inorganic content of the modeling liquids used in this study may also have affected the properties of the materials.[17] Nonetheless, the lack of complete information about these commercial formulations made it difficult to evaluate these differences. Lastly, the two adhesives evaluated were commonly available at dental offices and the clinician was not required to have a material specifically designed for use as a modeling liquid. Thus, studies that evaluate different materials and staining liquids and the amount of modeling liquid used may contribute to a better understanding of the clinical reality. Considering that modeling liquids are applied directly on the last layer of the resin composite during restoration, modeling with adhesives is an alternative[20,25,30] to reduce color change and surface roughness, consequently improving the surface quality of a resin composite.

Conclusions

Based on the findings of this study, it can be concluded that the Wetting resin showed the highest surface roughness and staining potentials. Toothbrushing reduced the

color changes (ΔE_{00}) produced by wine staining, except for the Wetting resin. Both adhesives were beneficial as a modeling liquid, promoting lower surface roughness, better color stability, higher WI, and lower opacity values.

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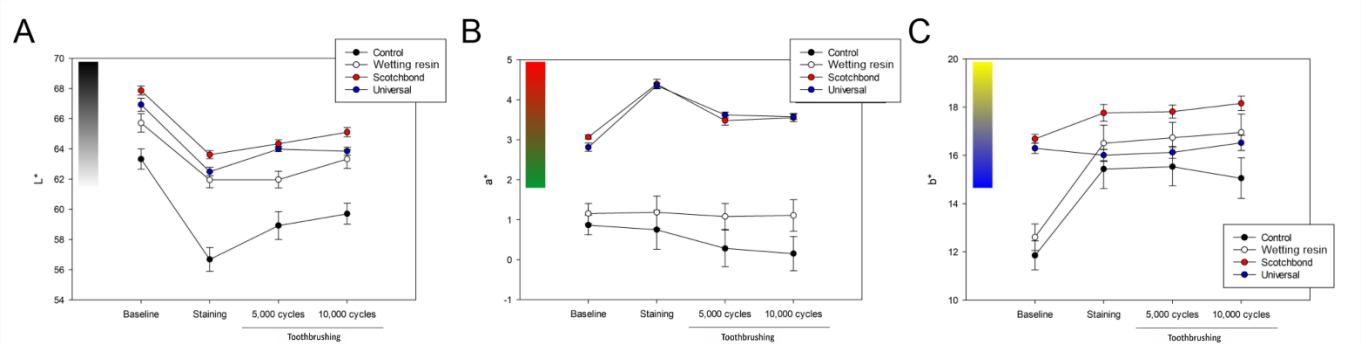
Figure legends

Figure 1. Behavior of color parameters according to treatment and assessment time. (A) parameter L* on the black-to-white axis, (B) parameter a* chromatic coordinates on the red-to-green axis, and (C) parameter b* chromatic coordinates on the yellow-to-blue axis.

Figure 2. Illustrative disc-shaped resin composite specimens, based on data from L*a*b* converted to the RGB system.

Figure 3. Scanning electron microscopy representative images of resin composite surfaces after being modeled with coatings at 5000x. Line 1: Control group; Line 2: Wetting resin; Line 3: Scotchbond adhesive; Line 4: Universal adhesive. The letters corresponded to baseline (a), after staining - (T1) (b), after 5,000 cycles - (T2) (c) and after 10,000 toothbrushing cycles - (T4) (d).


A
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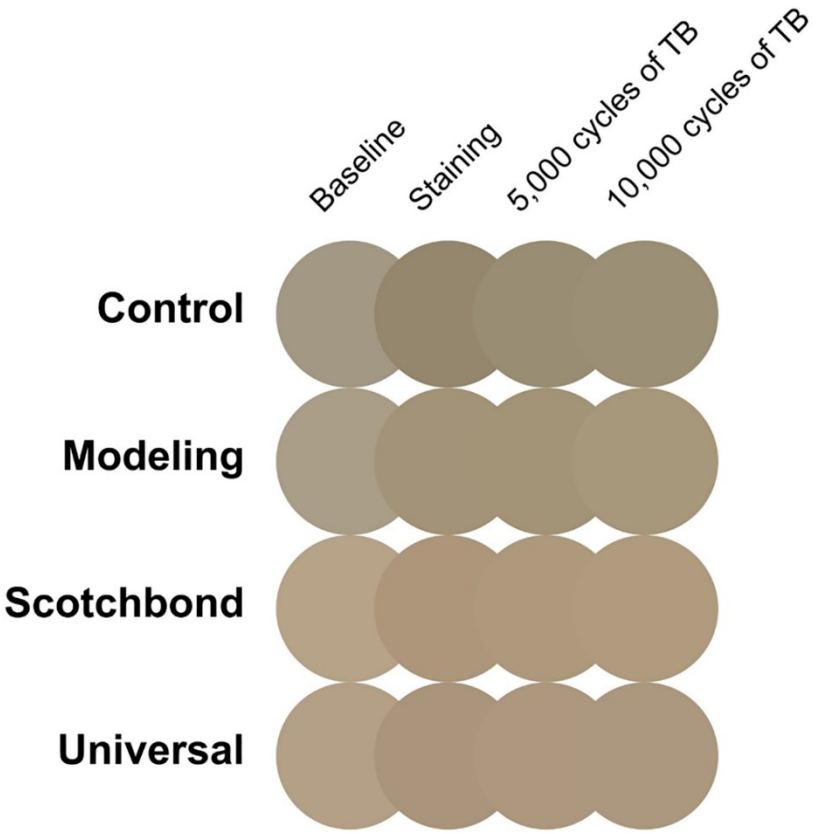
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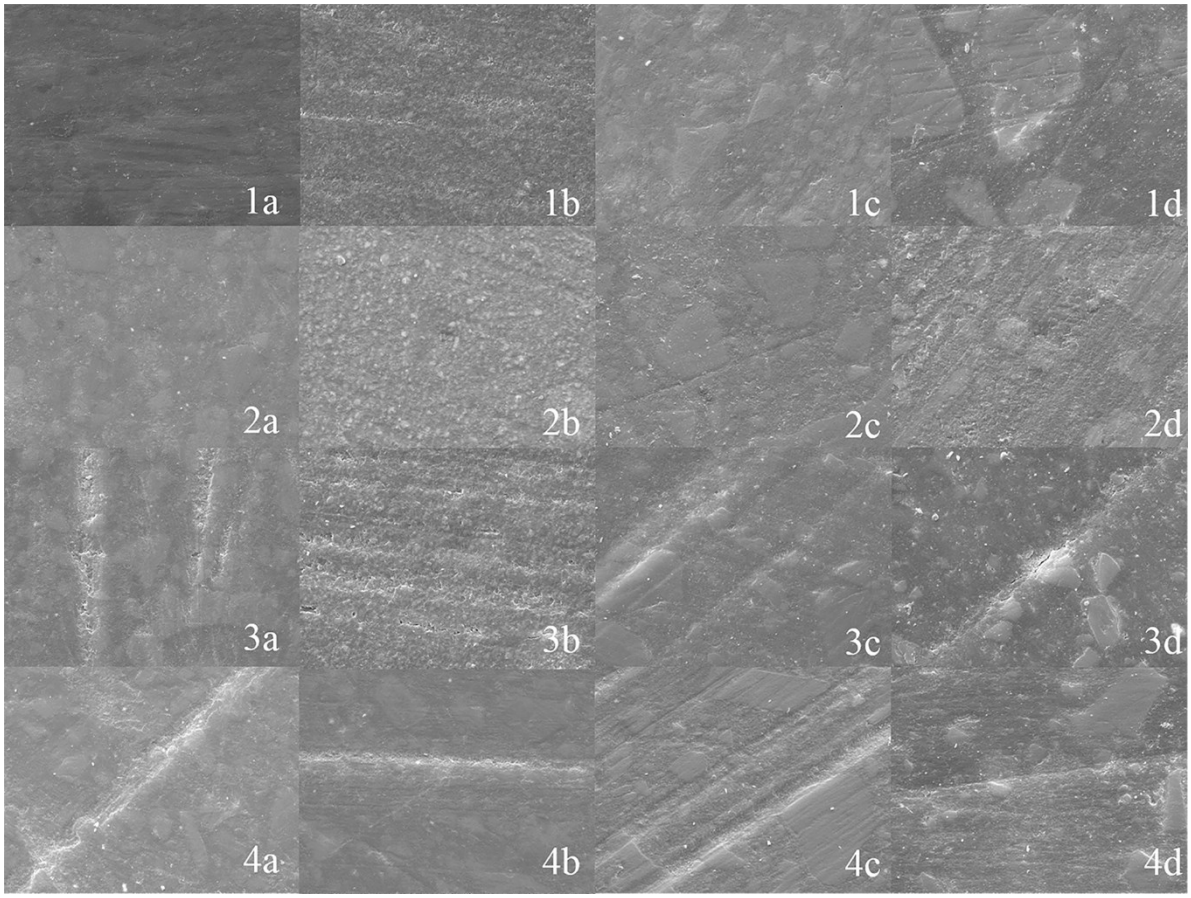

Table 1. Description of the evaluated materials.

Material (manufacturer)	Monomers and solvents	Filler content
Vittra APS (FGM, Joinville, SC, Brazil)	UDMA, TEGDMA	Silica-zirconia
Composite Wetting Resin (Ultradent Products Inc, South Jordan, UT, USA)	TEGDMA, DUDMA	Silica
Scotchbond Multipurpose (3M ESPE, St. Paul, MN, USA)	BisGMA, HEMA	-
Single Bond Universal (3M ESPE, St. Paul, MN, USA)	MDP, dimethacrylate resins, HEMA, polyacrylic acid methacrylate copolymer, polyalkenoic acid, ethanol and water.	Silica

UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; DUDMA: diurethane dimethacrylate; TMSPM: Bis-GMA: bisphenol A glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

Table 2. Mean and standard deviation (SD) of Ra values according to treatment and assessment time intervals[a].

Treatment	Baseline	After staining	Toothbrushing		Pooled average
			5,000 cycles	10,000 cycles	
Control	0.31 (0.13)	0.38 (0.38)	0.79 (1.44)	0.43 (0.19)	0.48 (0.51) ^{AB}
Wetting resin	0.58 (0.23)	0.48 (0.17)	0.50 (0.25)	0.44 (0.12)	0.50 (0.14) ^{A}
Scotchbond	0.44 (0.29)	0.32 (0.16)	0.21 (0.05)	0.27 (0.05)	0.31 (0.14) ^{B}
Universal	0.34 (0.08)	0.25 (0.08)	0.28 (0.09)	0.25 (0.06)	028 (0.08) ^{B}

[a] *For pooled average, different letters indicate statistical difference shown by Tukey's test ($p<0.05$).*

Table 3. Mean and standard deviation (SD) of ΔE_{00} values from baseline data according to treatment and assessment time intervals[a].

Treatment	After staining	Toothbrushing	
		5,000 cycles	10,000 cycles
Control	6.48 (1.76) ^{Aa}	4.92 (2.56) ^{Ba}	4.25 (1.79) ^{Ba}
Wetting resin	4.71 (1.07) ^{Ab}	4.67 (1.53) ^{Aa}	4.16 (0.94) ^{Aa}
Scotchbond	3.86 (0.68) ^{Ab}	3.07 (0.91) ^{ABb}	2.52 (0.61) ^{Bb}
Universal	4.12 (0.15) ^{Ab}	2.61 (0.82) ^{Bb}	2.78 (1.07) ^{Bab}

[a] *Different letters (capital for line, lowercase for row) indicated statistical difference Tukey's test ($p<0.005$).*

Table 4. Mean and standard deviation (SD) of whitening index values according to treatment and assessment time intervals[a].

Treatment	Baseline	After staining	Toothbrushing	
			5,000 cycles	10,000 cycles
Control	20.4 (0.8) ^{Aa}	12.7 (2.8) ^{Cc}	13.3 (2.8) ^{BCb}	14.2 (2.8) ^{Bb}
Wetting resin	21.2 (1.3) ^{Aa}	14.8 (1.2) ^{Bb}	14.5 (1.9) ^{Bb}	15.3 (1.3) ^{Bb}
Scotchbond	19.8 (0.6) ^{Aa}	17.3 (1.6) ^{Ba}	17.0 (1.4) ^{Ba}	17.1 (1.2) ^{Ba}
Universal	19.4 (0.8) ^{Aa}	18.9 (0.7) ^{Aa}	18.8 (0.8) ^{Aa}	18.3 (1.0) ^{Aa}

[a] *Different letters (capital for line, lowercase for row) indicated statistical difference Tukey's test ($p<0.005$).*

Table 5. Mean and standard deviation (SD) of opacity values according to treatment and assessment time intervals[a].

Treatment	Baseline	After staining	Toothbrushing		Pooled average
			5,000 cycles	10,000 cycles	
Control	87.7 (4.4)	90.7 (3.5)	92.2 (2.9)	91.8 (2.1)	90.6 (3.7) ^{A}
Wetting resin	86.1 (2.3)	87.6 (3.2)	89.5 (5.3)	88.2 (3.6)	87.9 (3.8) ^{A}
Scotchbond	80.9 (3.2)	83.9 (1.5)	83.5 (2.1)	83.7 (2.4)	83.0 (2.6) ^{B}
Universal	81.8 (1.8)	84.5 (2.0)	85.6 (2.8)	86.1 (4.2)	84.5 (3.2) ^{B}
Pooled average	84.1 (4.1) ^{B}	87.6 (3.8) ^{A}	87.7 (4.8) ^{A}	87.5 (4.3) ^{A}	

[a] *For pooled averages, different letters indicate statistical difference shown by Tukey's test ($p<0.005$).*

Capítulo II

2.2 Capítulo 2

Influence of modeling liquids on physical properties and adherence analysis of S*treptococcus mutans* on the resin composites

Influence of modeling liquids on the resin composites

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Keywords: Composite resins; Surface properties; Hardness; Wettability; *Streptococcus mutans*

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ABSTRACT

Objectives: To evaluate the effect of modeling liquids on hardness, contact angle, surface roughness and bacteria adhesion in the resin composites.

Methods: Four modeling liquids; control group (CG), Composite Wetting Resin (CWR), Scotchbond Multipurpose (SM), Single Bond Universal (SBU) and two resin composites (Vittra APS and Filtek Z350 XT) were evaluated. The specimens were prepared for determining hardness (n=10) by a microdurometer, hydrophobicity (n=3) by the contact angle, surface roughness (Ra) (n=10) using a profilometer and bacterial adhesion (n=3) were investigated with colony-forming unit counting ($x10^5$ CFU/mL) and by spectrophotometer. The data were analyzed using Shapiro–Wilk, Levene and two-way ANOVA with Tukey's post hoc test ($p < 0.05$).

Results: The highest hardness was found in the CG, and the lowest values were observed in SBU for both resins. SM and SBU had higher contact angles for Vittra and the CG obtained the lowest ($p = 0.003$). SBU and CG had the lowest Ra values and the CWR obtained the highest for both resins ($p = < 0.001$). No statistically significance differences were found for Ra ($p = 0.967$) and *Streptococcus mutans* counts ($p = 0.434$) between the resins. The Z350 XT showed the higher biomass formation ($p = 0.034$).

Conclusions: The tested adhesives showed a reduction in hardness, an increase of the contact angle and no negative influence on the surface roughness and bacterial adhesion of resin composites.

Clinical significance: The application of modeling liquid on the composite did not interfere negatively with its properties of contact angle, surface roughness and bacterial adhesion, with the exception of hardness.

Keywords: Composite resins; Surface properties; Hardness; Wettability; *Streptococcus mutans*

1. Introduction

In the mid-1960s, the resin composite was introduced as restorative material for the reconstruction of the lost tooth structure began. Since then, resin-based materials have constantly improved [1,2]. Resin composite is a restorative material used both for invasive procedures and for more complex aesthetic situations [2]. However, this material has adversities that can affect the longevity of the restoration [1]. These limitations may be inherent to the material [1], or depend on the skill of the operator who performs them and can be reduced or controlled through the correct choice of composition and the techniques used [1,3,4]. The technique directly influences the polymer contraction [1,4,5] and, consequently, the residual contraction stress that can cause undesirable results such as the stress occurrence at the tooth/restoration interface, reduced adhesion, marginal leakage, postoperative sensitivity and/or secondary caries [1,6-7].

The resin composite composition has been changing in the last years, but basically consists of four basic components (1) inorganic filler formed by glass, quartz and/or silica particles, which have the function of increasing strength and decreasing polymerization shrinkage; (2) organic matrix formed by monomers, the part responsible for rigidity, increases polymerization and reduces viscosity; (3) the bonding agent usually is the silane, which adheres fillers with the organic matrix, and (4) initiator-accelerator responsible for the polymerization system [1,7]. However, the high viscosity of some constituent monomers, as the main of them – BisGMA [8], make it difficult to manipulate and insert the resin composite, as well as to obtain the desired sculpture [9,10]. In order to improve this adversity related to the handling of composite, specific liquids for modeling were recently proposed [11]. These low-viscosity materials have in their composition diluent monomers such as triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA). However, many dental dentist's practices "wetting" the composite with adhesives with the aim of reducing the viscosity of the layer surface and facilitating its adaptation to the tooth structure [12,13]. In this sense, another concern arises regarding the effect of hydrophilic monomers for example HEMA and solvents present in adhesives when it is used as a wetting agent [14].

Another important point is the beneficial effect of the low-viscosity materials in reducing defects present in the composite [15,16], but little is yet known about its effect

on bacterial adhesion. The microorganism *Streptococcus mutans (S. mutans)* is considered one of the main etiological agents of tooth decay [17,18]. It adheres to hard surfaces, forming plaques bacterial cariogenic and initiates the production of acids and degrading enzymes that negatively influence the longevity of restorations [18,19]. Studies report that the composition and characteristics of materials influence adhesion, so hydrophilic materials attract more oral biofilm than hydrophobic ones [20], and rough surfaces more than smooth surfaces [18,21]. However, there are controversies that report the insignificance between surface roughness and the amount of bacterial adhesion [22,23].

Given the increasingly frequent use of modeling liquids and different resin composites in the construction of aesthetic restorations, it is important to understand the physical properties that affect the combination of resin composites with wetting agents. Therefore, considering the use of these materials by professionals and the lack of information on the possible influences that allow the practice to be carried out safely, the objectives of this *in vitro* study were to evaluate the hardness, contact angle, surface roughness and bacterial adhesion of *S. mutans* to resin composites subjected to different modeling liquids. Thus, the null hypotheses were tested as follows: (1) The modeling liquids do not affect the hardness, roughness, or contact angle of resin composite surface, and (2) The amount of S. mutans adhesion is not influenced by modeling liquids on resin.

2. Materials and methods

2.1. Specimen preparation

The materials used in this study and their contents are presented in Table 1. A total of 104-disc specimens were prepared by packing of uncured resin composites into custom-made polytetrafluoroethylene moulds, with a diameter of 8 mm and thickness of 1.5 mm. The composite (Vittra APS, FGM, Joinville, SC, Brazil and Filtek Z350 XT, 3M ESPE, St. Paul, MN, USA) was inserted in a single increment and the set was pressed against the glass plate, in a straight motion, aiming to make the surface flat. Therefore, after inserting the resin composite into the matrix, the finalization was done with the nº 4B brush (Kota, Cotia, São Paulo, Brazil) and covered by the modeling liquids. The

specimens were randomly divided into four groups according to the modeling liquids which were performed:

1 Control group (CG): The surface was modeled only with help of the brush, without using any wetting agent.
2 Composite Wetting Resin (CWR): The surface was modeled with brush using Composite Wetting Resin (Ultradent Products Inc, South Jordan, UT, USA).
3 Scotchbond Multipurpose (SM): The surface was modeled with brush using bond of the conventional 3-step Scotchbond Multipurpose adhesive system (3M ESPE, St. Paul, MN, USA).
4 Single Bond Universal (SBU): The surface was modeled with brush using the simplified Single Bond Universal system (3M ESPE, St. Paul, MN, USA).

The brush was applied in six movements in the same direction to avoid air-bubble formation and to obtain a surface like that observed clinically with a minimal amount of a small drop standardized in pilot studies previously. Adhesives were agitated before application and the solvent was evaporated using a gentle air blast for 5 s before light curing. The increment was individually light-cured with the modeling liquids at the same time using a light-curing unit (irradiance 1000 mW/cm^2; VALO, Ultradent Products Inc, South Jordan, UT, USA) for 20 s. The light-curing unit had an irradiance intensity of 1000 mW/cm^2 verified by a light-emitting diode (LED) radiometer (Kondortech Equipamentos Odontológicos Ltda, São Carlos, SP, Brasil). After incubator (Solab, Piracicaba, São Paulo, Brazil) storage in distilled water at 37.7°C for 24 h, the specimens were polished with a series (abrasive medium, fine and extrafine) of aluminum oxide disks (Sof-lex, 3M ESPE, St. Paul, Minnesota, USA) for 20 s per disk by single trained operator. Subsequently, each disk was washed for 20 s, and in the end of the polishing cycle, the specimens were immersed in an ultrasonic bath (Thornton, Vinhedo, São Paulo, Brazil) for 10 min. The final thickness of the specimens was measured using a digital caliper (Absolute AOS Digimatic, Mitutoyo, Tokyo, Japan), and specimens thinner than 1.45 mm or thicker than 1.55 mm were replaced.

2.2. Hardness

To measure the Knoop hardness, a hardness tester was used (HMV 2; Shimadzu, Tokyo, Japan), and an indenter with pyramidal geometry and quadrangular base, obtaining a diamond measurement. The specimen was positioned with the surface to be examined parallel to the horizontal plane of the base of the device. Five indentations of each specimen were performed, starting at the center of the specimen, with a distance between them of 150 μm. The test was performed with controlled force, applying a load of 100 grams (0.98 N) of force in a determined time of 15 s in each indentation. For each specimen, the means resulting from each indentation were recorded and later used for statistical analysis [24].

2.3.Contact angle

To evaluate the wettability of the surfaces and to measure the contact angle, a goniometer (Ramé-Hart, Inc. Model NRL A-100 belonging to FEMEC/UFU) was used. A microsyringe adapted with 0.5 μl of distilled water was deposited on the specimen surface, forming a sessile drop. To standardize the distance, the camera (Canon EOS Rebel T6i, São Paulo, SP, Brazil) was fixed on the table at a distance of approximately 30 cm from the specimen. The capture of image was recorded within 20 s [25] of drop deposition on the specimen. All measurements were made by a single operator indoors and at controlled room temperature (25°C). The contact angle was measured from the contours of the drops using the ImageJ analysis software (National Institute of Mental Health, Bethesda, Maryland, USA).

2.4. Surface roughness

Surface roughness (Ra) was measured with a contact profilometer (Surftest 402, Mitutoyo, Kanagawa, Japan). The measuring length of 1.25 mm and a cut-off of 0.8 mm at a speed of 0.25 mm/s were used [26]. Three measurements were made in the center on each specimen and the arithmetic mean (μm) was obtained.

2.5. Microbiological procedures

All microbiological steps were performed under aseptic conditions and using a laminar flow hood disinfected with 70% alcohol. The entire test for both counting (CFU/mL) and crystal violet staining was performed in triplicate. The disc specimens

were packed separately and then sterilized in an autoclave at 121℃ for 15 min before being tested with bacteria.

2.5.1. Preparation of bacterial suspension

S. mutans strain (ATCC 25175) was planted on blood agar plates with 5% sucrose and allowed to culture at 37°C for 24 hours in an anaerobic condition. Bacteria from the cultures was then transferred into tubes containing 40 ml of BHI (brain-heart infusion; BBL, BD, USA) and incubated at 37°C CO_2 atmosphere for 24 h. After incubation, the bacteria producing tubes were mixed using a centrifuge (Zentrifuge Rotofix 32 Hettich, Germany) for 5 min (Zentrifuge Rotofix 32 Hettich, Germany. Bacterial suspension was prepared at 10^5 CFU/ml from the sediment at the bottom of the tube.

2.5.2. Biofilm formation

Artificial saliva (1 mL) was added and covered to the specimens in the sterilized 48-well plates. The following formula was used to prepare artificial saliva in a compounding pharmacy (Kiropharma, Uberlândia, MG, Brasil): for 1 L of deionize water, 0.1169 $Ca(OH)_2$, 0.1225 KH_2PO_4, 2.4280 tris cap. Specimens were then incubated at 37°C in 5% CO_2 atmosphere for 1 h to stimulate pellicle formation. Next, the specimens were washed with 1 mLof PBS and transferred to new sterilized 48-well plates [27].

Afterwards, 1,5 mL of previously prepared bacterial suspension was added to the surface of each specimen. Discs in BHI pure without inoculum served as the sterility control. Then, for bacterial adhesion, the 48-well plates were placed into an incubator (Thermo/FormaThermo Fisher Scientific CO_2 Water Jacketed Incubator, USA), where they were kept for 24 h at 35°C in 5% CO_2. Following incubation, the plates were gently dip-washed one time in 1,5 mL of sterile BHI to remove the loose bacteria.

2.5.3. Analysis by CFU counting

The specimens for each group were transferred into a sterile tube containing 1 mL physiological saline and then were vortexed for 1 min to harvest the adherent bacteria. The suspensions were sonicated (Ultrasonic Cleaner USC-1450ª- Indaiatuba, São Paulo, Brazil) at 30 W for 15 min to disrupt bacterial aggregates and were then 10-fold serially diluted in sterile physiological saline (x10^5 CFU/ml) and plated into a BHI agar. The

plates were incubated anaerobically for 24 h at 37°C, and the numbers of CFUs were then determined.

2.5.4. Analysis by optical density absorbance

The supernatant from the wells was removed, washed with 1 mL of PBS and confirming the growth of the strain, leaving only the biofilm formed for quantification of the biofilm biomass. After 24 h, 1 mL of 0.2% of crystal violet (Sigma Aldrich) was added to each specimen and incubated for 30 min at room temperature in order to stain the adhered cells. The violet was aspirated and the disc specimens were washed three times in deionized water to remove excess stain, air dried, and destained with 1 mL acetic acid for 20 min. The biofilm was evaluated by optical density (OD) absorbance using 595 nm wavelength light in a spectrophotometer (GloMax Multi, Promega, Madison, WI, USA) [28].

2.6. Statistical analysis

Normal distribution and equal variance of data were analyzed by Shapiro–Wilk and Levene tests, respectively. Two-way ANOVA and Tukey HSD multiple comparison tests were used to evaluate the differences between the groups. The significance level was determined as $\alpha = 0.05$ for all tests.

3. Results

3.1. Hardness

The mean of the surface hardness (KHN) values are shown in Table 2. Both "resin type" ($p = 0.001$) and "modeling liquid" ($p < 0.001$) factors affected hardness, and the interaction between the factors was also significant ($p = 0.005$).

The CG showed the highest hardness to both resins. CWR and SBU had the lowest hardness values to Vittra and Z350 XT resins, respectively. On Vittra surface, SBU and SM provided intermediate values, without differences between CWR.

However, CWR and SM cause intermediate hardness in Z350 XT, while SBU presented the lowest values. In this resin, no differences were shown by CWR and SM. Z350 XT treated with CWR and SM presented higher hardness values than Vittra. There

was no statistical difference between the resin type, Vittra or Z-350 XT, when specimens were subjected to CG or SBU.

3.2. Contact angle

The contact angle values of all materials are presented in Table 3. Only the factor "modeling liquid" ($p = 0.003$) interfered in the results of the contact angle, while "resin type" did not ($p = 0.606$). The interaction between the factors was significant ($p = 0.028$). The highest contact angle values were found in the SM and SBU groups for the Vittra, while the CG had the lowest values. CWR showed an intermediate contact angle value, with no statistical difference from the other treatments. There was no statistical difference between modeling liquids for Z350 XT resin.

Within the CG and the SM, the resin type is important, in which for the CG, Z350 XT resin had a higher contact angle, and for the SM, Vittra resin had a higher contact angle. Within the SBU and CWR, there was no statistical difference between the resins.

3.3. Surface roughness

The surface roughness (Ra) results are shown in Table 4. Two-way ANOVA revealed that Ra values were influenced by the "modeling liquid" factor ($p = < 0.001$), which was not modified by the "resin type" ($p = 0.967$). The interaction between the evaluated factors was also not significant ($p = 0.219$). Regardless of the resin type, the CG and the SBU resulted in the lowest Ra values. CWR had the highest Ra values. Intermediate Ra values were observed for SM, with no difference from the other treatments.

3.4. Bacteria adhesion

Biofilm formation are shown in Table 5. Both factors "resin type" ($p = 0.335$) and "modeling liquid" ($p = 0.434$) did not affect the biofilm formation (CFU/ml) of the specimens, as well as the interaction between the factors was also not significant ($p = 0.412$). Thus, there was no statistical difference between the groups.

An OD_{600} value < 0.1 was considered as the absence of biofilm formation, corresponding to the negative control (no bacteria) in the flow chamber. Only the "resin

type" factor (p = 0.034) affected the biomass (polysaccharides and bacteria), which was not modified by the "modeling liquid" factor (p = 0.925), and the interaction between the factors was not significant (p = 0.208). Regardless of the modeling liquid, specimens of Vittra showed lower total biomass accumulation by optical density when compared to Z350 XT.

4. Discussion

The purpose of this study was to evaluate the effect of modeling liquids on hardness, contact angle, surface roughness and biofilm formation to resin composites. The properties of resin composites have been constantly evaluated in order to investigate the material performance when exposed to different simulations of oral conditions, such as foods, masticatory forces, temperature and pH changes, and wear. In our study, according to the data obtained, the hardness values were different between the modeling liquids, which were significantly lower when compared to resin without modeling. The degree of reduction varied between materials, therefore, the first null hypothesis was rejected. This finding agrees with other studies [29,30]. Although changes in properties of resin composites are more related to the components of the polymer matrix, the size and type of filler can also influence the degradation resistance, mainly in hardness [7]. This justifies the higher hardness values found for the control groups, regardless of the tested resins, since they have a higher surface content of inorganic filler when compared to the other groups.

Although it is expected that the surface layer finished with the modeling liquids can be removed by the finishing and polishing procedure, it may be that they are diffused into deeper layers of the composite. Therefore, it is suggested that there was an increase in the total organic content, when incorporating the modeling liquids over the resin, justifying the lower values for hardness in the presence of them. Thus, when being evaluated, the surface may not only be composed of the material for modeling but also be influenced by the resin composite used. When comparing the hardness values when the composite was treated with CWR and SM, higher values were found for Z350 XT compared to Vittra (~72% filler weight), since Z350 XT (~78% of filler weight) has a higher content of fillers, in terms of weight, according to manufacturer's information. It

is worth remembering that CWR has 45% filler, and also higher viscosity compared to adhesives which gives an uneven surface, which can influence the indentation during the hardness test. On the other hand, the SBU, despite having filler, is not informed which filler type and for having acidic monomer, which leads us to believe that this may reflect in the lower values of hardness when used as a modeling liquid mainly their behavior in the long term. In addition, adhesives have HEMA in their composition, a hydrophilic monomer that causes water absorption [31] and could lead to lower hardness values.

In terms of resisting masticatory forces, microhardness is one of the most important *in vitro* mechanical properties of resin composites. Despite the low hardness values attributed to the groups that used modeling liquids, there are studies in the literature that found a beneficial effect of them, when using the same adhesives used in the present study. Furthermore, they suggested improvement in the cohesion and stability of the material, avoiding rapid hydrolysis and reduction in the occurrence and propagation of cracks under different conditions of mechanical loading and fracture behavior [16,32-33]. More studies are needed, especially under *in situ* conditions and *in vivo* to investigate this controversy and could bias us to use it for anterior teeth.

The highest contact angle values found were when using the Vittra with the respective modeling liquid, SM (90 ± 3.22) and SBU (84.80 ± 2.54), rejecting too the first null hypothesis. These values suggest a more hydrophobic environment, that is, with less affinity for water. SM obtained a higher contact angle values compared to SBU, a relevant factor, as both have the BisGMA monomer, which is a compound with a hydrophobic group (Table 1). However, the percentage of this monomer is considerably higher for SM (60 – 70%), justifying the lower wettability of this composite, which acted as a protective barrier. Another plausible explanation is that these adhesives do not have the TEGDMA diluent [34], which is a hydrophilic monomer (contains hydrophilic ethers), and leads to water sorption by the polymer [35]. However, this added monomer in the composition of the resin composite has a significant role, as which increases polymerization and decreases resin viscosity to facilitate handling and sculpture [34].

Following these same thoughts, the presence of the hydrophobic BisGMA monomer is expected to justify the higher contact angle for the CG of Filtek compared to Vittra. This is because Vittra does not have BisGMA or BisEMA in its composition, but methacrylic monomers such as UDMA and TEGDMA [34]. These monomers have

carboxylic and hydroxyl groups in their molecular structure that are more prone to hydrolysis [35], increasing their wettability. Furthermore, the presence of the APS (Advanced Polymerization System) initiator can indicate a complex nature and influence the material chemistry, although it is a confidential component of the manufacturer. The other groups that used Vittra had higher contact angles, especially because of the characteristics of the modeling liquids used. It was expected that the same groups of modeling liquids would present the same behavior for the Z350 XT. However, some type of chemical reaction or interaction with the resin composite may have taken place to obtain lower contact angle values compared to Vittra.

The surface layer of the restoration has a decisive effect on the esthetics, color stability and surface roughness of the resin composite [36]. A well-polished and smooth restoration reduces plaque accumulation and consequently, decreases the risk of secondary caries and staining [21]. The surface layer of composites can be indirectly affected by the polishing procedure or even by the oral wear that happens over time, therefore modifying the chemical composition of the composites. The findings of this study showed differences in surface roughness between the modeling liquids, which also to reject the first null hypothesis. The CG and SBU presented the lowest Ra values, while the CWR presented the highest, due to the higher viscosity of the material. Differently, the adhesives are less viscous and consequently resulted in lower roughness values. However, in relation to the type of resin composite, there was no statistical difference, justified by the higher amount of small fillers for both. Smaller filler particles provide good mechanical and physical properties for resin composites, for example, lower surface roughness [22,37]. The Z350 XT is nanoparticulate with particles made from a mixture of silica with a size of 20 nm and zirconia with a 4-11 nm and Vittra is nanohybrid with zirconia silicate fillers, with particles of 100-200 nm. All information was considered according to the manufacturer (Table 1).

The amount of S. *mutans* adhesion did not differ between the different types of modeling liquids. Therefore, the second null hypothesis was accepted for the CFU/ml method. There are many *in vitro* and *in vivo* studies that demonstrate the influence of surface characteristics such as surface roughness on bacterial adhesion [21,38,39], as well as others suggest the absence of this effect [23]. However, in this study, there was no statistical difference between the materials for modeling and resin type in bacterial

adhesion by CFU/ml. The presence of pellicle acquired from artificial saliva may have masked the differences between the respective surfaces of the groups tested [40]. Likewise, morphological characteristics, such as filler particle size, shape and distribution of the inorganic matrix composition for both resin composites may have influenced this result, considering that all groups underwent the same polishing procedure [22].

In the current study, the specimens were evaluated in terms of 24-h bacterial biofilm formation, sufficient time for evaluation of S. mutans incubation as seen in other studies [21,27]. On the other hand, although the resin type did not show differences in surface roughness and biofilm formation in CFU/ml of *S. mutans*, Z350 XT showed greater total biomass accumulation. This result suggests that the presence of the BisGMA monomer, which is released into the oral environment, can be influenced by factors such as polymerization, hydrolysis and mechanical degradation [41]. Despite reducing the growth and viability of *S. mutans* [37], curiously, the number of dead cells in the biofilm increased significantly in the presence of BisGMA. This can be explained by the ability of this monomer to decrease resistance to acid stress and consequently increase intracellular polysaccharide accumulation and transport sugar in *S. mutans* on containing environment sucrose. Thus, in this study, the materials composition seemed to influence more on the other surface properties than the surface roughness. Analysis by crystal violet staining also includes the extracellular matrix of polysaccharides (which protects and surrounds bacterial colonies), that is, it identifies a measure of live, dead and fragmented bacteria [42]. In contrast, CFU analysis only quantifies the number of viable bacteria, capable of multiplication and active metabolism [17,43].

The present study is one of the few that tried to identify the effects of modeling liquids on various physical properties of resin composites, when used in the surface layer. Considering the results of the present study, regarding the contact angle, surface roughness and bacterial adhesion, the adhesives did not harm these physical properties of composites, with hardness exception. They also have the advantage of ease of use and availability for clinicians in their dental offices, although there is in the literature disadvantages like the possibility of yellowing due to the presence of camphorquinone. As for resin composite, further studies are needed to evaluate others to detect whether the composition of the monomers really is the most influential factor. In this study, the contact angle and bacterial adhesion of these materials for modeling were pioneers,

contributing to the advancement of studies in this area. Therefore, more studies should be carried out to evaluate the effect of different modeling liquids, in order to estimate other physical and chemical parameters, such as the ideal amount of liquid, pH oscillation present in the oral cavity, color stability, among others. In addition to these possibilities, studies with a variety of bacterial species, mainly under *in situ* and *in vivo* conditions, since in this study only one bacteria strain was used for formation of biofilm, not adequately simulating the oral microbiota.

5. Conclusions

Within the limitations of the current study, the following can be concluded:

1. The adhesives use did not influence negatively the physical properties of contact angle, surface roughness and biofilm formation of resin composites, with the exception of hardness.
2. Z350 XT showed higher hardness values and greater accumulation of total biomass. Vittra and Z350 XT had similar behaviors for contact angle, surface roughness and bacterial adhesion by CFU/ml.

Table 1
Materials used in the study and their contents.

Material	Composition	Filler Content (volume/ weight)	Manufacturer
Filtek Z350 XT	BisGMA, UDMA, TEGDMA, BisEMA, PEGDMA, silica particles (20nm), zirconia (4-11nm), clusters, camphorquinone, photoinitiators, silane.	63,3/ 78,5	3M ESPE, St. Paul, MN, EUA
Vittra APS	UDMA, TEGDMA, zirconia oxide glass nanospheres (70 to 82%), ethyl 4-dimethylaminobenzo, photoinitiator (APS), camphorquinone, stabilizer and silane (100–200 nm).	52-60/ 72-82	FGM, Joinville, SC, Brasil
Composite Wetting Resin	UDMA, TEGDMA (>10-≤25%), DUDMA (>10-≤25%), TMSPM (≤10%), BHT (< 1%).	45%	Ultradent Products Inc, Jordânia do Sul, UT, EUA
Scotchbond Multipurpose Adhesive	BisGMA (60–70%), HEMA (30–40%), triphenyl antimony (< 0.5%), tertiary amines, photoinitiators.	-	3M ESPE, St. Paul, MN, EUA
Single Bond Universal Adhesive	BisGMA (15-25%), phosphate acid monomers (MDP), silane, water, ethanol, HEMA, methacrylate copolymer of polyacrylic acids and polyalkenoic acid, initiators, filler.	-	3M ESPE, St. Paul, MN, EUA

Table 2

Mean ± Standard Deviation (SD) for Hardness (KHN) (n = 10).

Groups	Vittra	Z350 XT
CG	73.74 ± 2.62 Aa	74.55 ± 2.79 Aa
CWR	49.60 ± 3.69 Bc	57.43 ± 3.51 Ab
SM	54.19 ± 3.72 Bb	59.10 ± 3.75 Ab
SBU	52.82 ±3.75 Abc	52.28 ± 3.33 Ac

Different uppercases in the same line indicated the statistical differences between “resin types” ($p<0.05$). Different lowercases in the same column indicated the statistical differences between “modeling liquids” ($p<0.05$).

Table 3

Mean ± Standard Deviation (SD) for Contact Angle (ϕ) (n = 3).

Groups	Vittra	Z350 XT
CG	67.64 ± 0.66 Bb	77.33 ± 1.76 Aa
CWR	76.63 ± 3.3 Aab	74.56 ± 0.88 Aa
SM	90 ± 3.22 Aa	78.73 ± 3.12 Ba
SBU	84.80 ± 2.54 Aa	79.63 ± 2.64 Aa

Different uppercases in the same line indicated the statistical differences between “resin types” ($p<0.05$). Different lowercases in the same column indicated the statistical differences between “modeling liquids” ($p<0.05$).

Table 4

Mean ± Standard Deviation (SD) for Surface Roughness (Ra) (n = 10).

Groups	Vittra	Z350 XT
CG	0.27 ± 0.14 Aa	0.26 ± 0.09 Aa
CWR	0.52 ± 0.22 Ab	0.44 ± 0.09 Ab
SM	0.38 ± 0.30 Aab	0.40 ± 0.07 Aab
SBU	0.24 ± 0.27 Aa	0.39 ± 0.12 Aa

Different uppercases in the same line indicated the statistical differences between “resin types” ($p<0.05$). Different lowercases in the same column indicated the statistical differences between “modeling liquids” ($p<0.05$).

Table 5

Mean ± Standard Deviation (SD) for Bacterial adhesion (n = 3).

	UFC/ml		Optical density	
	Vittra	Z350 XT	Vittra	Z350 XT
CG	1.38 ± 0.31 Aa	1.72 ± 0.25 Aa	3.83 ± 0.35 Aa	4.34 ± 0.42 Ba
CWR	1.75 ± 0.23 Aa	1.78 ± 0.10 Aa	3.94 ± 0.53 Aa	4.36 ± 0.31 Ba

SM	1.60 ± 0.54 Aa	1.92 ± 0.06 Aa	3.91 ± 0.37 Aa	4.89 ± 0.59 Ba
SBU	1.75 ± 0.25 Aa	1.60 ± 0.07 Aa	4.00 ± 0.15 Aa	4.61 ± 0.11 Ba

Different uppercases in the same line indicated the statistical differences between "resin types" ($p<0.05$). Different lowercases in the same column indicated the statistical differences between "modeling liquids" ($p<0.05$).

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Capítulo III

2.3 Capítulo 3

Effect of the resin composite immediate repair using different modeling liquid on the bond strength

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Effect of the resin composite immediate repair using different modeling liquids on the bond strength

Abstract

Objective: The purpose of this study was to evaluate the effect of modeling agents on the bond strength (μSBS) and the failure mode of immediate repair of resin composite.

Materials and methods: Fourty specimens were confectioned with a nanohybrid composite (Forma, Ultradent). The surfaces of the specimens were finished with medium of aluminum oxide disks (Sof-lex, 3M ESPE) with five unidirectional movements and cleaned with air jet by single trained operator. The discs were randomly divided into four groups (n=10) according to the following modeling agents: control group; Composite Wetting Resin (Ultradent Products); Adper Scotchbond Multipurpose adhesive (3M ESPE) or Adper Singlebond Universal adhesive (3M ESPE). Then, cylinders (1.0 mm diameter x 1.5 mm height) with the same composite resin was bonded to the treated surfaces to simulate the immediate repair. After 24 h, the specimens were subjected to the microshear test and the failure area was analyzed under an optical microscope to identify the failure mode. The data were analyzed by one-way analysis of variance and Tukey test (α=0.05).

Results: There is no statistically significantly μSBS between groups, regardless of groups ($p < 0.05$). The failure mode showed predominance of adhesive and cohesive failure in composite resin base.

Conclusions: The modeling agents tested do not influence the bond strength during immediate repair of nanohybrid resin composite.

Clinical significance: In the immediate repair, there is no need to use any modeling agents to improve the resin-resin bond.

KEYWORDS: resin composite, adhesives, shear bond strength

1. INTRODUCTION

Resin composite materials have advanced in their longevity and durability over the last two decades, and has become a preference for patients because it is less expensive and invasive restorative approach.[1] Moreover, aesthetics is another parameter used to

evaluate the quality of the material that is only possible due to the ability of resin composites of biomimicry with the tooth structure.[2-4] The reproduction of polychromy is achieved with varied compositions, colors and effects of composite resins, although it is extremely challenging[5] due to the recommended thickness for each layer.[6]

During the construction of the restoration, it may happen that some of layer are left over in terms of thickness. It may be detected immediately after the filling and if such a situation is encountered, it will need to be removed to be continue the restoration. The ideal solution for such problems is to remove the layer unsatisfactory, make a surface treatment and refill to obtain satisfactory results.[7-9] Considering that there is no contamination by saliva or water during this abrasion, and therefore, without damage to adhesion, the residual monomers and oligomers on surface originated by the oxygen-inhibited layer are not eluted by solventes or degradation.[10,11]

Successful esthetic restorations are only possible with good adhesion. In this sense, one of the objectives of restorative dentistry is to develop adhesive materials that can provide an effective bond at the tooth-restoration interface as well as resin-resin interface.[12] Such an ideal adhesive material can strengthen the composite while preparing the restoration or tooth to be bonded with much more conservative preparations, with minimal wear performed.[13] The surface treatment protocols for repair include the application of phosphoric acid, silane and adhesive, but this requires time to perform the steps and knowledge, since there are several options and adhesive compositions on the market.[14,15] Modeling liquids are low viscosity fluid resins that have monomers in their composition similar to those present in adhesives. In these cases of immediate repair, a doubt arises whether modeling liquids could also be used as a bonding agent, using even when the oxygen layer has been removed or damaged as in the above situation cited.

It is known the tendency of adhesive technology is to reduce steps, application time, and technical sensitivity and facilitate the application of the bonding agent.[12,16] However, to the extent of the authors' knowledge, there is no information regarding the effect of modeling agents to bond to resin when it was necessary adjusments in an immediate layer and if a cleaning procedure only using air and modeling agents can be performed to achieve better bond strength avoiding delamination between the layers and thereby increasing the adhesive strength between them. The purpose of this *in vitro* study was to evaluate the effect of modeling agents on the bond strength of immediate repair of

resin composite. The null hypothesis was that modeling agents does not improve the bond strength, regardless of the material tested.

2. MATERIALS AND METHODS

The characteristics of the materials used and their chemical compositions are described in Table 1.

Specimen preparation

40 cylinder-shaped composite discs' matrix (diameter, 12 mm; height, 2 mm) were prepared in a single increment with the composite (Forma Zirconia Nano-Hybrid Composite, Ultradent do Brasil, Indaiatuba, SP, Brasil). The surface composite was covered with a transparent polyether strip and compressed by 10 s using a glass slide to obtain a flat surface. After the slide was removed, the specimen was photoactivated by 20 s, with the polyether strip in contact with the surface of the layer, using the LED Light unit (Grand Valo, Ultradent, South Jordan, Utah, USA) in standard power mode of approximately 1000 mW/cm² radiant intensity. The abrasion was performed to simulate an intercurrence of excess composite, using the medium (dark orange) disc soflex pop on- (3M ESPE, St. Paul, MN, USA) in a low-speed. It was done 5 movements by a single operator and application air jet during 5 s. After, the composite discs were removed from the matrix and randomized in parallel groups (by Random program- https://www.random.org/). The specimens were divided into a total of four experimental groups:

1 Control group (CG): none bonding agent was used to make the adhesion.
2 Composite Wetting Resin (CWR): after air jet, application of the Composite Wetting resin modeling liquid (Ultradent Products Inc, South Jordan, UT, USA).
3 Scotchbond Multipurpose (SBM): after air jet, application bond of the conventional 3-step Scotchbond Multipurpose adhesive system (3M ESPE, St. Paul, MN, USA).
4 Single Bond Universal (SBU): after air jet, application of the simplified Single Bond Universal system (3M ESPE, St. Paul, MN, USA).

The materials were applied in the same way with the help of brush in six movements in the same direction to avoid air-bubble formation. The bonding agent was individually light-cured with Grand Valo for 20 s.

Specimen preparation: resin composite cylinders

After preparing the disks and the surface treatment, plastic tubes (1.0 x 1.5 mm; diameter x height) were fixed on the surface of the specimen. The resin composite was inserted into the tubes with an insertion spatula. After excesses were removed, photoactivation was carried out for 20 s according to the manufacturer's guidelines. After 1 h at room temperature, the tubes were gently removed using a scalpel blade (N°. 15, Med Goldman Indústria e Comércio Ltda., Santa Catarina, Brazil) carefully by the same operator. In each specimen, four cylinders of the resin composite were obtained. The specimens were stored at 37º C for 24 h in distilled water before the test.

Microshear bond strength test

The microshear bond strength (μSBS) test of specimens was carried out in a machine (Microtensile, OM100, Odeme, Luzema, SC, Brasil) with a load of 50 N at 1.0 mm/min, until the resin composite cylinders ruptured. A caliper (Mitutoyo 530312B10, Tokyo, Japan) was used to measure the dimensions of the composite cylinders. A thin stainless-steel wire (0,2 mm in diameter) was looped around each cylindrical-shaped resin cylinder in contact with the resin base. Force was directly applied to the resin cylinders until the occurrence of failure. The center of the load cell and the wire loop was positioned as straight as possible to ensure the desired orientation for micro-shear test stress. Micro-shear bond strengths were expressed in Megapascale (MPa), as derived from dividing the maximum load (N) at the time of failure by the adhesion area (mm^2).

Failure mode analysis

After the rupture of the cylinders, the surfaces of the disks were observed by three evaluators to determine the failure mode. It was analyzed by stereomicroscope (Mitutoyo Kawasaki, Japan) at 40× magnification. Failure modes were categorized as the following types: I- adhesive interfacial failure between resin composites; II- cohesive failure in base

resin; III- cohesive failure of cylinder resin (of immediate repair); IV- and mixed (adhesive-cohesive) failure.

Statistical analysis

Normal distribution and equal variance of data were analyzed by Shapiro–Wilk and Levene tests, respectively. One-way ANOVA and Tukey HSD multiple comparison tests were used to evaluate the differences between groups. The significance level was determined as p=0.05 for all results.

3. RESULTS

There is no statistically significantly μSBS between groups, regardless of bonding agent ($p < 0.05$) (Table 2). Regarding the failure mode, the predominance was adhesive and base cohesive mode failure, to CG and SBU respectively. CWR and SBM groups had the same behavior ($p < 0.05$), with the same percentage for adhesive, cohesive and mixed failures (Figure 1).

4. DISCUSSION

In the present study, applying the bonding agent did not improve the bond strength for groups tested. Therefore, the null hypothesis should be accepted.

Resin composite is a widely used restorative material and is also considered advantageous because is a suitable material for repair.[17] Although the choice of repair is appropriate, it requires a specific surface treatment procedure with the interface bonding between new layer of resin and the old restoration due to degradation restoration to the oral environment over time.[7,14] Complete removal and remaking of a defective composite restoration is not necessary or desirable in some clinical situations. When dealing with an immediate situation, the casuistry is different, considering the presence of free monomers on the surface. This occurs within the first hours after initial polymerization, thus are still able to be adhered,[10,11] unlike late repair.

In the literature, a positive correlation between the presence of an oxygen inhibition layer and the composite repair strength was suggested, what it means that the remaining active, free radicals is available for reacting with residual monomers being a crucial factor in direct composite repair.[18] Considering that the free radicals have half-

lives that slowly decay over time [19,20] and that the idea of "repair" in this study is immediate, that is, right after the excess removal, new layer is inserted, following the incremental restoration procedure, the adhesion with the surface treated only with the air jet appeared to be satisfactory and not inferior to the tested groups. There are studies that have shown that the presence of an oxygen-inhibited layer does not significantly affect the composite-to-composite bond strength if coupling is performed within the first 14 days.[21,22]

In this study, the finishing with disc produced an irregular surface that probably caused an increase in the contact area, which in turn resulted in rich bonding capability with the new resin. The abrasion was done in a controlled way at low speed in the absence of water. It is worth remembering that the cleaning was simulated only with a constant jet of air and the application of phosphoric acid was not performed in this work due to the absence of saliva, blood or any type of contamination. Therefore, the present study focused on the application of bond agent to assess the possibility of attaining a bond strength comparable to that of the control group. Besides, it is more favorable to carry out a simple procedure rather than a complicated one in clinical settings.

Considering the materials tested, Composite Wetting resin has monomers and is the one with the highest content of inorganic filler, 45% of silica, which could also make it more resistant and in addition, still has less hydrophilicity because it does not present HEMA or other solvents and, therefore, this aspect could be promote less potential to degradation. These inferences are declared by analyzing the contents of the manufacturer's package insert, which it could be favorable factor to be used for this functionality, as the manufacturer recommends. Within this context, the adhesives showed similar behaviors between them, even the SBU that has acid monomer and silane in its composition. It is possible to imply that silane incorporation into universal adhesive does not strongly benefit composite-composite bonding when used to immediate repair. This result leads us to confirm that micromechanical retention proved to be superior to the application of modeling agents tested,[23] showing that composite-composite generates enough bonding, if it has ideal conditions of moisture. In this sense, late evaluation is necessary to assess long-term bond if there really is no difference between modeling agents.

Failure type did not seem to be in agreement with the quantitative values of adhesive strength, as for example the SBU group showed a lower value and predominance of base cohesive failures. So we can speculate that bond strength obtained here can not be extrapolated to other free-zirconia composites, which is in agreement with another study.[14] These failures appeared to be restricted to the tested nanohybrid composite resin, which has a high content of inorganic filler of zirconia and silica.

The limitation of this study was not to carry out the positive control group, which would be the application of phosphoric acid, silane and adhesive to achieve ideal adhesive strength,[15] but for the fact that it was not the main objective, which was to simulate *in vitro* behaviors performed by many professionals. In the present study, the excess removal was prepared with medium discs using a low-speed, yielding a thinner layer of powder than would be the case in others clinical conditions. In order to make better comparisons with the literature, further studies should be done with additional evaluating groups thicker, bur-created layers and composites with different filler contents and monomer compositions. It is known the adhesion of adhesive materials to dentin is highly sensitive and can be easily affected by contamination of the dentin surface.[24] Nevertheless, saliva or thermocycling conditions were not performed in this study to simulate an immediate repair in the face of an intercurrence still during the construction of the restoration, however, a late evaluation of this immediate repair is also necessary. Moreover, adding drying-only group will contribute to the literature and comparability of results, which seems to be advantageous, since it reduces steps and consequently procedure time to achieve long term success.

CONCLUSION

Within the limitations of this current study, it was concluded that:

Composite excess removal should be avoided during clinical procedures of restoration construction, but if such a situation is encountered, only air jet cleaning procedure can be performed to bond immediate repair of resin composite.

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DISCLOSURE

The authors declare that they do not have any financial interest in the companies whose materials are included in this article.

DATA AVAILABILITY STATEMENT

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

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TABLE 1: Material compositions obtained from manufacturer's information and safety data sheets

Material	Manufactures	Type/Color	Composition	Batch Number
Forma	UltraDent, Indaiatuba, SP, Brasil	Nano-hybrid / A1 Enamel	BisGMA, TEGDMA, BisEMA e (UDMA), zircônia/silica e glass of barium fillers	D0G05
Sof-Lex pop-on disks	3M ESPE, Seefeld, Germany	Abrasive disks/ Serie Orange 4931 M	Polyester film coated with aluminum oxide abrasive and metallic center.	2202800636
Adper ™ Scotchbond Multipurpose	3M ESPE, St. Paul, MN, USA	-	BisGMA, HEMA, polyalkenoic acid copolymer, silanized silicium, alcohol, water, photo-initiator	2212400452
Single Bond Universal	3M ESPE, St. Paul, MN, USA	-	MDP, dimethacrylate resins, HEMA, polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	2202500081
Composite Wetting Resin	Ultradent, South Jordan, Utah, EUA	-	TEGDMA, DUDMA	D0F81

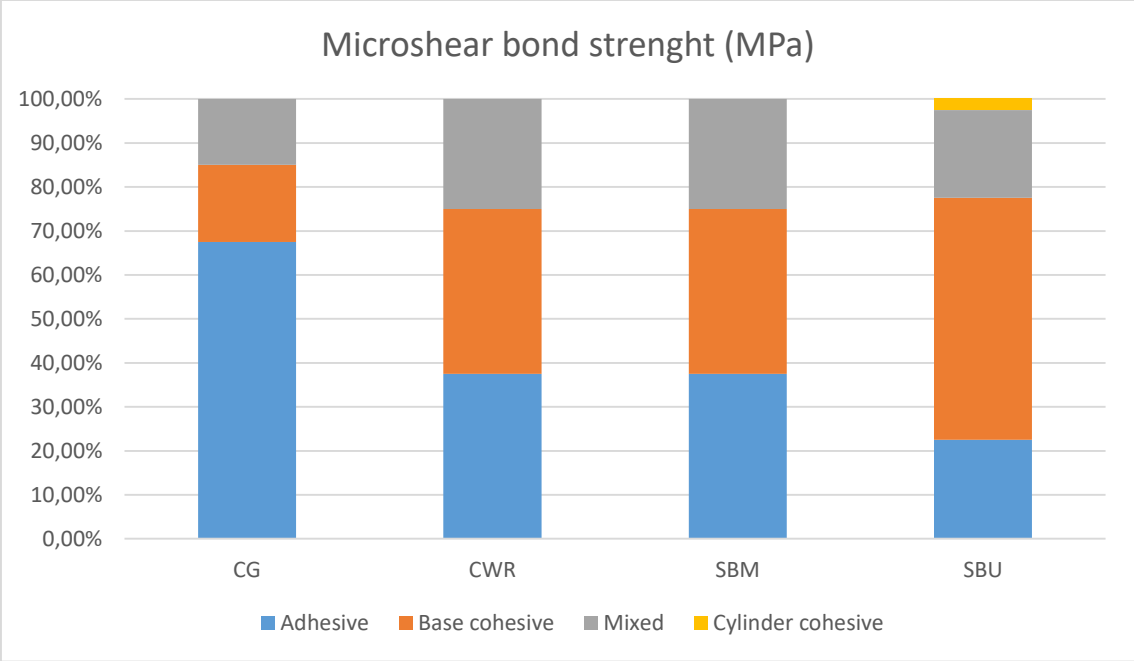
Abbreviations: Bis-GMA: bisphenol A glycidylmethacrylate; TEGDMA: triethylenglycol dimethacrylate; BIESMA: ethoxylated Bisphenol-A Diglycidyl Dimethacrylate; UDMA: urethane dimethacrylate; DUDMA: diurethane dimethacrylate; TMSPM: HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

TABLE 2. Mean microshear bond strengths (μSBS) for each subgroup (MPa ± SD)

Modeling agents	Bond strenght
CG	14.19 ± 3.19 A
CWR	15.62 ± 4.76 A
SBM	12.83 ± 3.59 A
SBU	11.68 ± 2.77 A

Note: Values identified using similar letters are not significantly different ($p < 0,05$).

FIGURE 1. Failure mode (%) of resin composite treated by bonding agent.



Considerações Gerais

3. CONSIDERAÇÕES GERAIS

A partir dos resultados obtidos e considerando as limitações metodológicas deste estudo, conclui-se que:

- O Composite Wetting Resin apresentou a maior rugosidade superficial e potencial de manchamento. Todos os grupos apresentaram alguma degradação, resultando em superfícies irregulares, arranhões superficiais e áreas de descolamento, após manchamento e escovação.

- A escovação reduziu as alterações de cor (ΔE_{00}) produzidas pelo manchamento do vinho, exceto para a resina líquida. O manchamento causou redução do IB (índice de brancura), enquanto os ciclos de escovação não aumentaram o IB. Os menores valores de opacidade foram observados no estágio inicial, após o polimento.

- Ambos os adesivos foram benéficos como líquido para modelagem: promovendo menor rugosidade superficial, melhor estabilidade de cor, maior IB e menores valores de opacidade.

- O uso dos adesivos como líquidos para modelagem não prejudicaram as propriedades de ângulo de contato, rugosidade superficial e adesão bacteriana de Steptococcus mutans, com exceção da dureza.

- Z350 XT apresentou maiores valores de dureza e maior acúmulo de biomassa total. Vittra e Z350 XT tiveram comportamentos semelhantes para ângulo de contato, rugosidade superficial e adesão bacteriana por UFC/ml.

- Apenas o procedimento de limpeza com jato de ar pode ser realizado para alcançar uma resistência de união aceitável no reparo imediato da resina composta.

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