Faculdade de Odontologia de Araçatuba - UNESP
Departamento de Materiais Odontológicos e Prótese
Programa de Pós-Graduação em Odontologia – Prótese Dentária

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EFFECT OF ANTIOXIDANT AGENTS AFTER DENTAL BLEACHING ON LUTING OF CERAMIC LAMINATES

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Orientador: Prof. Associado Paulo Henrique dos Santos

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Dedicatória

A Deus

" Conheço teu medo, a tua felicidade e os teus sonhos. Conheço tua estrada e sei exatamente o teu destino. Conheço-te por dentro…

E sem que tu tenhas que me pedir, eu entendo o que tu queres. Conheço o teu sorriso, e sei tudo que esta dentro do teu coração. Conheço e te reconheço em qualquer lugar…

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Conheço-te e sei que és muito especial, como é especial cada filho meu, mas cada um com as suas diferenças, ainda assim o meu amor é incondicional, e ele é o maior amor do mundo. Conheço-te, porque eu te criei…

(Salmo 46: 10)

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Um irmão é uma continuação de nós mesmos. Você é minha continuação do meu jeito de ser, de viver e da minha maneira de olhar o mundo.

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"A gratidão é a virtude das almas nobres"

Esopo

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Epígrafe

“Felicidade é a certeza de que a nossa vida

não está se passando inutilmente.”

Érico Veríssimo

Resumo Geral

Sahyon HBS. Efeito de agentes antioxidantes pós-clareamento na cimentação de laminados cerâmicos [tese]. Araçatuba: Universidade Estadual Paulista "Júlio de Mesquita Filho" (UNESP), Faculdade de Odontologia; 2021.

RESUMO GERAL

O objetivo deste estudo *in vitro* foi avaliar o efeito da aplicação de agentes antioxidantes após o clareamento dentário e previamente à cimentação de laminados cerâmicos na estabilidade cromática do conjunto restaurador, assim como nas propriedades mecânicas de nanodureza (HIT), módulo de elasticidade (Eit*), grau de conversão, resistência de união e morfologia da interface adesiva. Ademais, a neutralização de peróxido de hidrogênio e a caracterização superficial do esmalte, como ângulo de contato, energia de superfície, energia livre total de interação, microscopia eletrônica de varredura e espectroscopia de energia dispersiva do substrato submetido à ação das soluções antioxidantes e do agente clareador também foram analisados. Duzentos e quarenta e dois blocos de esmalte dentário (7x8x0,6mm) foram utilizados para o processo de cimentação e distribuídos em grupos experimentais de acordo com os métodos de procedimentos (grupo não clareado, grupo clareado com Whiteness HP Maxx 35%), tipos de antioxidantes adotados (controle; ácido ascórbico 10% e α-tocoferol 10%) e períodos de cimentação (após 24 horas e 14 dias do processo de cimentação) (n = 22). Foi utilizado o sistema adesivo Tetric N Bond Universal e o cimento resinoso Variolink Esthetic LC (Ivoclar Vivadent) como agentes cimentantes. Os dados foram submetidos a testes estatísticos de normalidade e analisados por ANOVA e teste de Tukey (α = 0,05). Os dados da morfologia da interface adesiva, obtidas pela microscopia confocal a laser, foram submetidas ao teste Kappa inter-examinadores e os dados foram submetidos aos testes Kruskal-Wallis e Dunn (α = 0,05). Os resultados mostraram que, de modo geral, a utilização da solução antioxidante α-tocoferol 10% pós-clareamento no período mediato promoveu resultados satisfatórios com relação à estabilidade cromática do conjunto restaurador, assim como para as propriedades mecânicas, grau de conversão, resistência de união e morfologia da interface adesiva comparado ao grupo clareado sem associação dos agentes antioxidantes, tanto para o período mediato, quanto para o período de 14 dias ($p<0,05$). A solução de α-tocoferol 10% apresentou maiores valores de neutralização do peróxido de hidrogênio e maiores valores de molhabilidade do esmalte em relação aos grupos controle e ácido ascórbico ($p<0,05$). A energia de superfície e energia livre total de interação do esmalte dentário foi significativamente influenciada pelo tratamento clareador ($p<0,05$). Dessa forma,

conclui-se que o emprego da solução antioxidante α-tocoferol 10% promoveu resultados promissores, sugerindo que o mesmo poderia ser utilizado mediatamente após o clareamento dental na cimentação de laminados cerâmicos.

Palavras-chave: Cerâmica. Cimentos de Resina. Adesivos. Cor. Dureza. Módulo de Elasticidade. Propriedades de Superfície.

Abstract

Sahyon HBS. Effect of antioxidant agents after dental bleaching on luting of ceramic laminates [thesis]. Araçatuba: UNESP – São Paulo State University; 2021.

ABSTRACT

The aim of this *in vitro* study was to evaluate the effect of antioxidant agents application after tooth bleaching and prior to luting of ceramic veneers on color stability of the restorative set, as well as on mechanical properties of nanohardness (HIT), elastic modulus (Eit*), degree of conversion, bond strength and morphology of the adhesive interface. Furthermore, the hydrogen peroxide neutralization and surface characterization of enamel, such as contact angle, surface energy, total free energy of interaction, scanning electron microscopy and energy-dispersive spectroscopy of the substrate submitted to the action of antioxidant solutions and bleaching agent were also analyzed. Two hundred forty two dental enamel blocks (7 x 8 x 0.6 mm) were used for the luting process and distributed into experimental groups according to the procedure methods (unbleached group, bleached group with Whiteness HP Maxx 35%), types of antioxidants adopted (control; 10% ascorbic acid and 10% α-tocopherol) and the luting periods (24 hours and 14 days after the luting process) (n = 22). Tetric N Bond Universal adhesive system and Variolink Esthetic LC resin cement (Ivoclar Vivadent) were used as luting agents. Data were submitted to statistical tests of normality and analyzed by ANOVA and Tukey tests (α = 0.05). The adhesive interface morphology data, obtained by confocal laser microscopy, were submitted to the inter-examiner Kappa test and the data were submitted to Kruskal-Wallis and Dunn´s tests (α = 0.05). The results showed that, in general, the use of 10% α-tocopherol antioxidant solution after bleaching in mediate period promoted satisfactory results regarding the color stability of the restorative set, as well as for the mechanical properties, degree of conversion, shear bond strength and adhesive interface morphology compared to the bleached group without antioxidant agents association, both for the mediate and 14-day period ($P < 0.05$). The 10% α-tocopherol solution showed higher hydrogen peroxide neutralization values and higher enamel wettability values compared to the control and ascorbic acid groups ($P < 0.05$). Surface energy and total free energy of interaction of tooth enamel were significantly influenced by the bleaching treatment ($P < 0.05$). Thus, it is concluded that the use of 10% α-tocopherol antioxidant solution promoted promising results, suggesting that it could be used mediately after tooth bleaching on luting of ceramic laminates.

Keywords: Ceramics. Resin Cements. Adhesives. Color. Hardness. Elastic Modulus. Surface Properties.

Listas e Sumário

LISTA DE FIGURAS

Chapter 1

Chapter 2

LISTA DE TABELAS

Chapter 1

Chapter 2

LISTA DE ABREVIATURAS, SÍMBOLOS E SIGLAS

α Alpha

> Maior que

< Menor que

≥ Maior ou igual

≤ Menor ou igual

^{o}C Grau Celsius

^{o} Angle

± Mais ou menos

≅ Aproximadamente igual

% Percentagem

μm Micrometro

μN Micro Newton

μg Micro grama

μ Micro litro

γ^{AB} Polar component

γ^{LW} Nonpolar component

γ+ Receptor component

γ- Donor component

γ^{S} Surface energy

ΔG_{sws}^{Total} Total free interaction energy

λ Comprimento de onda

υi Possion ratio of the indenter

υs Posion ratio of the material

ΔE Variação de estabilidade de cor

A Área

Adj. Adjunto

Ass. Assistente

ANOVA Análise da variância

ASTM – G53 American Society for Testing Materials – Norma 53

Au Ouro

Bis-GMA Bisfenol A glicidil dimetacrilato

BS Bond strength

C Carbono

Ca Cálcio

CAD/CAM Computer Aided Design/Computer Aided Manufacture

CAPES Coordenação de Aperfeiçoamento de Pessoal de Nível Superior

CF Calibration factor

CIE Comission Internacionale de I´Eclairage

cm Centímetro

Corp. Corporation

D3MA Decandiol dimethadrylate

DC Degree of conversion

Dr. Doutor

Dra. Doutora

EDS Energy dispersive X-ray spectroscopy

Eit* Módulo de elasticidade

E_i Elasticity modulus of the diamond

E_r Reduced modulus

et al. E colaboradores

FAPESP Fundação de Amparo à Pesquisa do Estado de São Paulo

F_{max} Força máxima

FOA Faculdade de Odontologia de Araçatuba

g Grama

GPa GigaPascal

h Hora

HEMA Hidroxil-etil-metacrilato

HIT Dureza Bercovich

HT High Translucency

IL Illinois

J Joule

LC Light-cured

LED Light Emitting Diode

m Metro

MCAP Methacrylated carboxylic acid polymer

MDP Metacriloxidecil di-hidrogênio fosfato

Mg Magnésio

min Minute

mL Mililitro (unidade de medida equivalente a 10^{-3} l)

mm Milímetro (unidade de medida equivalente a 10^{-3} m)

mN Mili Newton

MN Minessota

MO Missouri

MPa Mega Pascal

N^{o} Número

N Newton

Na Sódio

NiCr Níquel-cromo

nm Nanômetro

NY New York

O Oxigênio

OH Hidroxyl Group

P Fósforo

PG Pós-graduação

pH Potencial hidrogeniônico

PR Paraná

Prof. Professor

Prof^{a}. Professora

PVC Polyvinyl chloride

s Seconds

Si Silício

SC Santa Catarina

SEM Scanning electron microscopy

SP São Paulo

TEGDMA Dimetacrilato de trietilenoglicol

UDMA Uretano Dimetacrilato

UK United Kingdon

UNESP Universidade Estadual Paulista “Júlio de Mesquita Filho”

UFABC Universidade Federal do ABC

USA United Sates of America

UT Utah

UV radiação ultravioleta

W Watt

SUMÁRIO

Introdução Geral

1 INTRODUÇÃO GERAL*

Com o desenvolvimento dos materiais odontológicos, o cirurgião dentista possui como opções restauradoras para os elementos dentais várias modalidades de tratamento, incluindo as resinas compostas e as cerâmicas odontológicas. Com o aumento da demanda por restaurações estéticas, o correto diagnóstico de qual é o material mais indicado para cada caso, muitas vezes é um desafio para o clínico.

O conceito atual da Odontologia Restauradora preconiza que, para qualquer tipo de procedimento, o profissional deva sempre optar pelo tratamento mais conservador, evitando o desgaste desnecessário do elemento dental.[1] Porém, é preciso considerar que, em muitos casos, a utilização de restaurações indiretas, que requerem um desgaste planejado e controlado, poderia ser mais efetiva, em termos de estética e durabilidade comparado às restaurações diretas.[2,3] Nesse sentido, os laminados cerâmicos associados aos procedimentos adesivos de cimentação resinosa são uma das escolhas mais satisfatória ao se tratar do quesito estético, uma vez que concede o mínimo ou até mesmo nenhum desgaste no elemento dental,[2,3] permitindo uma mimetização da cor, textura e forma, além de proporcionar o restabelecimento do tamanho e posição dos dentes, conferindo naturalidade e harmonia ao sorriso. As propriedades ópticas dos laminados cerâmicos reproduzem a policromia natural, translucidez e opacidade, possibilitando a obtenção de caracterizações semelhantes aos dentes adjacentes.[4,5]

A evolução tecnológica proveniente das diversas áreas vem sendo gradativamente incorporada na Odontologia, em especial, na área da prótese. Dentro deste contexto, podemos exemplificar a tecnologia CAD/CAM, modificando paradigmas e provocando uma revolução no planejamento e confecção das restaurações protéticas, possibilitado métodos de tratamento que aliam resultados estéticos e duradouros com a facilidade de execução e economia de tempo das sessões clínicas, tanto para o profissional quanto para o paciente,[6-8] permitindo, dessa forma, a confecção de restaurações indiretas em apenas uma sessão. As cerâmicas reforçadas por dissilicato de lítio têm sido amplamente utilizadas nestas situações, pois proporcionam uma estética satisfatória[2,3] devido a sua alta translucidez e resistência,[9] fazendo com que a combinação cerâmica/cimento resinoso possa mimetizar a estrutura dental com ampla reprodução de detalhes.[10] Entretanto, estes procedimentos podem muitas vezes

* Normalização segundo as Normas de Vancouver. Referências (ANEXO B)

necessitar de modificação prévia de cor do substrato dentário, envolvendo nestes casos a associação de agentes clareadores.[11]

Tendo em vista a valorização de procedimentos cada vez menos invasivos, a técnica de clareamento dental associado à cimentação de laminados cerâmicos torna-se uma alternativa bastante conservadora,[12,13] possibilitando uma escolha satisfatória aos procedimentos invasivos com desgastes da estrutura dentária associados a restaurações mais espessas a fim de solucionar as alterações cromáticas. Na Odontologia, existem diversas técnicas para clarear os elementos dentários. Dentre elas, podemos citar o clareamento caseiro e o clareamento em consultório, ambas caracterizadas pela aplicação de agentes clareadores à base de peróxido de carbamida e hidrogênio, em diferentes concentrações, tempos de atuação, com ou sem ativação por luz.[14] Nas técnicas de clareamento, há uma variação no tipo de agente clareador e na velocidade do processo, no entanto as técnicas têm em comum a liberação de oxigênio como ponto principal da reação.[15,16]

O processo de clareamento é caracterizado pela reação de óxido-redução provocada pelo peróxido de hidrogênio, promovendo a liberação de radicais livres que devido ao seu baixo peso molecular penetram através das porosidades dos cristais de hidroxiapatita do esmalte dentário atingindo a dentina.[17] Através de um processo químico, os radicais livres permitem a conversão de substâncias complexas em substâncias abertas simplificadas e mais claras, em consequência da abertura de seus anéis de carbono, convertendo-as eventualmente em dióxido de carbono e água, liberados juntamente com as bolhas de oxigênio.[17]

Apesar da eficiência clareadora destes agentes, um efeito adverso relevante de importância clínica significante deve ser levado em consideração. Estudos na literatura têm mostrado um redução considerável na resistência adesiva de restaurações resinosas ao esmalte, quando o procedimento reabilitador é realizado imediatamente após o clareamento dental, independente da concentração dos peróxidos ou do tipo de técnica.[18,19] A causa deste efeito adverso se deve ao oxigênio residual acumulado na superfície dentária ou até mesmo no interior da estrutura, dificultando a penetração dos monômeros resinosos,[19,20] assim como a inibição da polimerização dos mesmos,[21] prejudicando o selamento marginal e promovendo o início do processo de microinfiltração e na redução da resistência adesiva da interface resinosa.[22-24]

De acordo com a literatura, a redução da resistência adesiva após o procedimento clareador é tempo dependente, sendo menor a resistência de união quando os testes são

realizados imediatamente após o uso do agente clareador. Todavia, não existe um consenso na literatura sobre o período específico de espera a fim de que ocorra a liberação do oxigênio residual e radicais livres. Segundo alguns autores, pode-se variar de 24 horas até 4 semanas.[25-27]

Dessa forma, visando otimizar o tempo clínico e reduzir o período de espera da realização do procedimento reabilitador, algumas técnicas têm sido sugeridas, dentre elas a utilização de substâncias redutoras e antioxidantes, tais como o ácido ascórbico,[19,28-30] α-tocoferol[31] e associação de agentes antioxidantes, possibilitando a neutralização dos efeitos oxidativos dos agentes clareadores, restabelecendo a resistência adesiva do conjunto cimentante. Alguns autores têm demonstrado o efeito satisfatório da substância α-tocoferol na proteção contra a citotoxidade[32] causada pela atuação do peróxido de hidrogênio sobre a atuação celular.[33-35] Porém, até o presente momento, são escassas pesquisas consistentes analisando a adesividade de sistemas adesivos e cimentos resinosos fotoativados empregados na cimentação de laminados cerâmicos posteriormente a aplicação destes antioxidantes pós-clareamento, assim como a interação destes com o tratamento clareador nas propriedades superficiais do esmalte dentário. Tal fato se torna ainda mais relevante pelo número cada vez maior de restaurações cerâmicas confeccionadas pelo sistema CAD-CAM, que, em alguns casos, permite que o processo de confecção e cimentação da peça cerâmica seja realizado na mesma sessão clínica.

Diante do exposto, é de grande relevância clínica e científica avaliar o efeito do clareamento previamente à cimentação de restaurações indiretas, bem como a atuação de diferentes antioxidantes nas propriedades da interface adesiva de laminados cerâmicos, assim como nas propriedades superficiais do esmalte dentário. Os objetivos deste estudo foram: 1) avaliar o efeito de soluções antioxidantes pós-clareamento dental na estabilidade de cor de laminados cerâmicos, bem como avaliar as propriedades mecânicas de nanodureza, módulo de elasticidade e grau de conversão dos componentes da interface adesiva, e 2) avaliar a resistência de união, morfologia da interface adesiva, e propriedades superficiais do esmalte dentário submetido à ação de agentes antioxidantes após o tratamento clareador.

Chapter 1

2 CHAPTER 1 - EFFECT OF ANTIOXIDANT AGENTS AFTER DENTAL BLEACHING ON COLOR STABILITY AND MECHANICAL PROPERTIES OF BONDING INTERFACE COMPONENTS IN CERAMIC LAMINATE VENEER LUTING[†]

Short Title: Effect of antioxidants on adhesive interface after dental bleaching

2.1 Abstract

Purpose: Studies evaluating the influence of antioxidant agents on the optical and mechanical properties of ceramic laminate veneers after dental bleaching are scarce. Thus, this *in vitro* study aimed to evaluate the influence of antioxidant agents on the color stability and mechanical properties, such as the nanohardness (HIT), elastic modulus (Eit*), and degree of conversion (DC) of the bonding interface components after dental bleaching in ceramic laminate veneer luting.

Material and Methods: A total of 143 bovine teeth were distributed into experimental groups, according to the procedure method (unbleached or bleached with Whiteness HP Maxx 35%), antioxidant type (control, 10% ascorbic acid, or 10% α-tocopherol), and luting period (24 hours or 14 days) (n = 13). Tetric N-Bond Universal adhesive system and Variolink Esthetic LC resin cement were used as luting agents to lute IPS e.max ceramic restorations (0.6 mm in thickness) to enamel. An ultraviolet–visible spectrophotometer (UV–VIS) was used to assess the color stability prior to and after UV-B artificial accelerated aging for 252, 504, and 756 h (n = 8). The HIT and Eit* of the adhesive and resin cement were measured using a nanohardness tester under a load of 1000 μN, and the DC was measured using a microRaman spectrometer (n = 5). The color stability and mechanical properties were measured and evaluated by 2-way and 1-way ANOVA respectively, and Tukey's test (α = .05).

Results: Distinct aging periods exerted significant changes on the color stability of the restorations luted in the enamel associated with ascorbic acid, bleached and unbleached, and the bleached enamel under no antioxidant agent action, for the experimental groups evaluated

[†] Standardization according to Operative Dentistry Guidelines https://jopdent.com/author-review-for-journal/instructions-to-authors

after 14 days ($P < .05$). The use of the α-tocopherol antioxidant solution after the bleaching process in 24 hours period did not alter the optical and mechanical properties of the adhesive interface of the laminate restorations, compared to those of the control group ($P > .05$).

Conclusion: The use of a 10% α-tocopherol antioxidant solution promoted promising results, suggesting that it could be mediately used after tooth bleaching to lute ceramic laminate veneers.

Keywords: Ceramic; Color; Elastic Modulus; Hardness; Polymerization; Tooth Bleaching.

Clinical Relevance: Alpha-tocopherol antioxidant can reverse the oxidizing effects of dental bleaching procedures, thereby reducing the waiting time to lute ceramic restorations.

2.2 Introduction

An emphasis on minimally invasive principles and luting materials has led to the development of alternatives for restorations, including ceramic laminates.[1,2] Computer-aided design and computer-aided manufacturing (CAD-CAM) technology has provided an efficient way to combine these principles with high aesthetics, strength, predictability, and longevity for dental restorations,[3] in addition to simplifying and reducing costs and manufacturing time.[4] Lithium disilicate ceramics have been widely used in oral rehabilitations because of their optical properties, natural aesthetics,[5] chemical durability, and resistance;[6] its combination with resin cement allows mimicry of shape, texture, and color, improving the naturalness of a smile.[7,8] However, these procedures typically demand prior modification of the tooth substrate color, as a dental bleaching, since ultrathinness ceramic restorations could be required.[9,10]

Regarding minimally invasive conceits, the tooth bleaching technique associated with ceramic laminate luting has become a satisfactory alternative.[11] During dental bleaching, the home-use and in-office bleaching procedures vary according to the bleaching agent type, concentration, action time, and light association.[12] However, oxygen release is common to both techniques as the main product of the reaction.[13,14] The bleaching process is characterized by an oxide reduction reaction caused by hydrogen peroxide, which releases free radicals because of its low molecular weight, enabling the hydroxyapatite crystals to

penetrate the enamel pores and reach the dentin, thereby inducing oxygen bubble formation.[15,16]

Despite the efficiency of bleaching agents, a relevant adverse effect of clinical importance must be considered. Previous studies confirm a considerable reduction in the bond strength of resin restorations to enamel, when the restorative procedure is performed immediately after tooth bleaching, regardless of the peroxide concentration or the technique used.[17,18] This is because the residual oxygen accumulated on the tooth surface or inside the structure hinders the penetration of resinous monomers,[17,19] and inhibits polymerization,[20] impairing the marginal sealing and reducing the adhesive interface strength.[21] Furthermore, the bond strength after the bleaching procedure is time-dependent; there is no specific waiting period of time for the release of residual oxygen and free radicals, and it could vary from 24 h to 4 weeks.[15,22]

Certain methods have been suggested to optimize the clinical time and reduce the waiting period of ceramic restoration luting, such as the use of reducing substances and antioxidants,[20] including ascorbic acid,[23,24] α-tocopherol,[25] and antioxidant agents that neutralize the oxidative effects of bleaching agents. Previous studies have demonstrated the protective effect of α-tocopherol in the cytotoxicity,[26] caused by the action of hydrogen peroxide on cell performance.[26-28] However, there is insufficient information regarding the effect of this agent on the color stability and mechanical properties of the luting interface components of ceramic laminates bonded to a bleached enamel surface. This has become relevant because of the increasing number of ceramic restorations made by the CAD-CAM system, in which, in some cases, the process of designing, milling, and luting the ceramic restorations is conducted in the same clinical session.[3,4]

Therefore, this *in vitro* study evaluated the effect of antioxidant agents on the color stability of ceramic laminates subjected to ultraviolet (UV) aging after dental bleaching and assessed the mechanical properties, such as the nanohardness (HIT), elastic modulus (Eit*), and degree of conversion (DC) of the adhesive system and resin cement in ceramic laminate veneer luting. Two null hypotheses were tested: 1) the bleaching process and antioxidant agents would not cause changes in the color stability of the restorations or in the mechanical properties of the dental adhesive and resin cement, and 2) different aging periods would not result in differences in the color stability of the ceramic restorations.

2.3 Material and Methods

Specimen Preparation

This research was conducted after approval by the Local Institutional - Ethics Committee on the Use of Animals (#01058-17). The materials investigated in this study are listed in Table 1.

A total of one hundred forty three lithium disilicate veneers (7×8×0.6 mm) were fabricated from highly translucent lithium disilicate blocks, (shade A1, IPS e.max CAD; Ivoclar Vivadent, Schaan, Liechtenstein) using a low-speed diamond saw coupled to a cutter machine (Isomet 1000; Buehler, Lake Bluff, IL, USA) under constant water cooling. The ceramic veneers were sintered in the manufacturer's furnace (Programat EP 5000; Ivoclar Vivadent, Schaan, Liechtenstein) for 1h at 780°C, according to the manufacturer's recommendations.[8]

One hundred forty three bovine incisors, from cattle aged 29–35 months, were obtained; teeth that exhibited stains, excessive incisal wear, morphological crown changes, and apparent cracks/fractures in the enamel substrate were substituted.[29] The teeth were mechanically cleaned using periodontal curettes and underwent prophylaxis with pumice stone and water. Thereafter, the teeth were evaluated using a magnifying glass at a magnification of ×4 (Bio-Art Equipamentos Odontológicas Ltda, São Carlos, SP, Brazil) to identify fractures and cracks induced by the extraction.[8]

Anatomic crowns were sectioned from the roots 1.0 mm above the cementum–enamel junction through a transversal section using a low-speed diamond saw under water irrigation, coupled to an Isomet 1000 (Buehler, Lake Bluff, IL, USA) to obtain tooth specimens of 7×8×4 mm.[7] The buccal specimen surfaces were flattened with #600 grit silicon carbide paper (Buehler, Lake Bluff, IL, USA), and distributed into 11 experimental groups (n=13), listed in Table 2.

Experimental Groups

In the control group, the enamel surface was etched with 37% phosphoric acid (FGM, Joinville, SC, Brazil) for 30s, followed by rinsing with deionized water and drying with air jets. The Tetric N-Bond Universal adhesive system (Ivoclar Vivadent, Schaan, Liechtenstein)

was actively applied on the etched surface for 20s, an air jet was applied for 5s, and curing was performed for 10s using a polywave light-curing unit (VALO; Ultradent, South Jordan, UT, USA) in standard mode (1000 mW/cm^2).[8] The inner lithium disilicate surface was etched with 5% hydrofluoric acid (FGM, Joinville, SC, Brazil) for 20s; the resulting residues were removed with an air/water spray, and the substrate was dried with an air jet. The Monobond Plus (Ivoclar Vivadent, Schaan, Liechtenstein) silane agent was actively applied to the etched ceramic surface for 60s and air-dried for 5s. Subsequently, a layer of the Tetric N-Bond Universal adhesive system was applied, and the solvent was evaporated using an air jet for 20s, without polymerization. The neutral shade Variolink Esthetic LC resin cement (Ivoclar Vivadent, Schaan, Liechtenstein) was directly dispensed on the inner surface of the ceramic laminate to avoid bubble formation, and the ceramic was positioned on the enamel surface. Prior to the restoration activation, a load of 4.9N was positioned on the restoration to standardize the thickness of the resin cement and removed thereafter. The excess resin cement was removed using a microbrush. The assembly was light-activated using a Valo polywave curing light (standard mode) for 30s in an opaque black box to prevent external light interference, and the distance between the light source and the sample was standardized to 1 mm.[8]

In the AA-24H group, specimens were treated as described for the control group. However, 24 h prior to the luting process, the treatment with 20 μL of 10% ascorbic acid (Apothicário, Araçatuba, SP, Brazil) antioxidant solution was conducted using a calibrated syringe.[25] The solution was maintained in contact with an enamel surface for 15 min, followed by rinsing with deionized water and drying with air jets.[25] The specimen was stored in containers containing artificial saliva (pH = 7.0; Apothicário, Araçatuba, SP, Brazil) at 37 °C. The AA-14D group specimens were treated as described for the AA-24H group; however, the ceramic laminate veneer luting was performed 14 days after the antioxidant action. The αT-24H and αT-14D group specimens were treated as described for the AA-24H and AA-14D groups, respectively; however, 20 μL of the 10% α-tocopherol (Apothicário, Araçatuba, SP, Brazil) antioxidant solution was applied.

The 35% hydrogen peroxide bleaching product (Whiteness HP Maxx; FGM, Joinville, SC, Brazil) was administered to the BLE-24H group specimens. Three drops of peroxide were mixed with one drop of a thickener. Thereafter, 40 μL of the final product was applied to the buccal enamel using a calibrating syringe; the bleaching agent was maintained in contact with the surface for 15 min and removed using sterile cotton and absorbent paper.

This application was performed thrice, totaling 45 min of exposure to the bleaching product. The product was removed with air jets/water and air-dried. This procedure was repeated three times with an interval of one week between sessions, during which the specimens were stored in artificial saliva (pH=7.0) at 37°C. At the end of the bleaching treatment, the samples were washed with deionized water and air-dried. The ceramic laminate veneer luting process was performed 24 h after the bleaching process.

The BLE-14D group specimens were treated as described for the BLE-24H group; however, the ceramic laminate veneer luting was performed 14 days after the bleaching treatment. The BLE-AA-24H, BLE-AA-14D, BLE-αT-24H, and BLE-αT-14D group specimens were treated as described for AA-24H, AA-14D, αT-24H, and αT-14D, respectively; however, the specimens were subjected to the bleaching treatment prior to the antioxidant solution application.

All samples were stored in Hanks solution (Sigma Chemical Co., St. Louis, MO, USA) at 37°C for 24 h in light-protected recipients.[1] For the color stability analysis, 88 specimens were used (n=8), and 55 specimens were designated for a mechanical property assessment (HIT, Eit*, and DC) (n=5).

Color Stability Analysis

A total of 88 specimens (n=8) were subjected to an initial chromatic analysis, using a UV–visible (VIS) spectrophotometer (model UV-2450; Shimadzu, Kyoto, Japan) using the Commission Internationale de I'Eclairage (CIE) L*a*b* System. A demarcation was made on the posterior portion of each sample to standardize the insertion into the color analysis device. Five measurements were made for each sample, and the arithmetic mean was calculated.[7,8]

After the initial readings, the specimens were subjected to an accelerated artificial aging process. The samples were fixed on a metal plate and aged using an UV-accelerated aging chamber (EQUV; Equilam, Diadema, SP, Brazil) coupled with eight fluorescent lamps (40 W each; emission peak of 313 nm), according to the ASTM G53 (American Society for Testing Materials, Standard 53). The artificial aging process was conducted under alternate periods of UV light and condensation with oxygen-saturated distilled water under conditions of heat and 100% relative humidity. Each aging cycle was run for 12 h. During the first 8 h, UV light was irradiated at 60 ± 3°C; in the following 4 h, a condensation period without light

was used at 45 ± 3°C. This test was performed for three aging periods: 252, 504, and 756 h, simulating the natural aging process in the oral cavity. Color readings were evaluated after each aging cycle. Each aging period was composed of 21 cycles (168 h of UV-B irradiation exposure and 84 h of condensation). The color change (ΔE) was determined by the baseline color coordinates and after the UV-B aging procedure and was calculated using the following equation:[7,8]

$$\Delta E = [(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2]^{1/2},$$

where the color change is represented by ΔE, ΔL ∗ is $L_{after\ aging} - L_{baseline}$, Δa ∗ is $a_{after\ aging} - a_{baseline}$, and Δb ∗ is $b_{after\ aging} - b_{baseline}$. The specimens were stored in Hanks solution (Sigma Chemical Co., St. Louis, Missouri, USA) at 37 °C until the color analysis was performed.[7,8]

HIT and Eit Mechanical Properties*

A total of 55 specimens (n=5) were sectioned perpendicular to the occluso-apical direction using a diamond saw coupled with a metallographic cutter machine (Isomet 1000; Buehler, Lake Bluff, IL, USA) (Figure 1A) under continuous water cooling. Slices of 3 mm were obtained from the specimens' central portions (Figure 1B). These slices were included in acrylic resin (Clássico, São Paulo, SP, Brazil), manually finished using #320, #600, #800, and #1200 grit silicon carbide papers (Buehler, Lake Bluff, IL, USA), and polished with #6, #3, #1, and #0.25 diamond pastes (Buehler, Lake Bluff, IL, USA) with 3 min for each paste (Figure 1C). The samples were cleaned in an ultrasonic unit (Cristofoli, Campo Mourão, PR, Brazil) with deionized water for 8 min between each finishing and polishing step and after the procedure. Subsequently, the specimens were preserved in a Hanks balanced salt solution to maintain the pH of the restorative set, and to prevent cell degradation and ion loss (Figure 1D).[7,8]

The HIT and Eit* of the adhesive interface were assessed using a nanoindenter (NHT2; Anton Paar, Graz, Austria) under a load action of 1000 μN, and a standard trapezoidal loading function of 5-2-5 seconds (Figure 1E).[7,8] Twelve measurements were made for each sample in two different substrates of the adhesive interface using the Berkovich diamond tip: six indentations in the dental adhesive and six indentations in the resin cement layer (Figure 2). The HIT and Eit* values were calculated according to the load–displacement curve and the following equation:[7,8]

$$HIT = \frac{F_{MAX}}{A},$$

where F_{MAX} is the total force applied, and A is the contact area between the diamond tip and the sample under the maximum load.

$$Eit *= E * (1 - vs^2)$$

$$E * = 1/\left[\left(\frac{1}{Er}\right) - \frac{1 - vi2}{Ei}\right],$$

where E_r is the reduced modulus, E_i is the elasticity modulus of the diamond tip, υ_i is the Poisson ratio of the indenter, and υ_s is the Poisson ratio of the assessed material. The mechanical property values were automatically calculated using the software installed with the nanohardness machine.

Degree of Conversion

The degree of conversion analysis of the adhesive system and resin cement was conducted using the same samples prepared for the HIT and Eit* mechanical property analysis (n=5). It was performed using a confocal Raman microscope (In Via; Renishaw, New Mills, Gloucestershire, UK) with a diffraction grating of 1800 grooves/mm, coupled to an optical microscope (Leica TCS SPE@, Leica, Mannheim, Germany) with a spatial resolution of 1.0 μm that selectively recorded the Raman spectrum of the interest region of the sample, using the He–Ne laser (λ = 633 nm). The Fourier transform (FT)-Raman spectra were analyzed by selecting a spectral region from 1550 to 1700 cm^{-1}.[30,31]

Raman readings were performed using a ×50 objective, and each spectrum was recorded with an integration time of 10s (Figure 1F). Five spectra of the dental adhesive and the resin cement layer were obtained for each specimen. During the experiment, the light in the room was turned off to prevent interference with the Raman reading. The room temperature was maintained at 23 ± 1°C, with a controlled relative humidity of ± 34%.[30,31]

To calculate the DC, the height ratio of the peaks at 1607 cm^{-1} (aromatic) and 1637 cm^{-1} (aliphatic) of the cured and uncured adhesive system and resin cement were assessed. The uncured materials were placed onto glass microscope slides, and the materials were read using the microRaman equipment. The DC values of the adhesive system and resin cement were obtained through specific software installed in the Raman machine, and calculated as follows:[30,31]

$$DC\ (\%) = 100 \times [1 - (R\ polymerized / R\ nonpolymerized)],$$

where R represents the ratio between the peaks of the cured and uncured luting materials.

Statistical Analysis

The color stability, HIT, Eit*, and DC data were subjected to normality (Shapiro-Wilk; Bioestat 2.0 Program) and homogeneity tests (Bartlett; Bioestat 2.0 Program). The color stability was analyzed using a two-way repeated measure analysis of variance (ANOVA) and a one-way ANOVA (5.0 Statview Program; Version 5.0.1) was used to analyze the mechanical properties. Tukey's protected least significant difference test ($\alpha = .05$) was performed for both analyses.

2.4 Results

Color Stability

The color stability values (ΔE) are listed in Table 3. The influence of enamel bleaching on color stability was observed for the first aging period (252 h); the control group presented lower color change values, compared to those of the BLE-24H, BLE-14D, BLE-AA-24H, and BLE-AA-14D groups ($P < .05$). However, there was no difference between control and BLE-αT-24H and BLE-αT-14D groups ($P > .05$). Furthermore, there were no differences among the control and experimental groups in which the unbleached enamel was subjected to the antioxidant solution application , regardless of the luting period ($P > .05$) (Table 3). For the second and third aging periods (504 and 756 h, respectively), in general, there was no significant difference among the experimental groups, except for the second aging cycle in which the AA-24H group showed lower chromatic alteration compared to those of the AA-14D group ($P = .0206$) (Table 3).

Regarding the aging periods, the BLE-14D and BLE-AA-14D groups showed higher chromatic change values in the first cycling period, compared to second and third aging periods ($P < .05$). However, for the AA-14D group, in the first aging period, the color stability was higher compared to the other cycling periods ($P < .05$). For the other experimental groups, there was no significant difference among the aging periods ($P > .05$) (Table 3).

HIT and Eit Mechanical Properties*

Dental Adhesive

The HIT and Eit* of the dental adhesive and resin cement are listed in Tables 4 and 5, respectively. The lowest HIT values of the dental adhesive were observed for the AA-14D, BLE-24H, and BLE-αT-14D groups (Table 4), compared to αT-14D group ($P < .05$). In the Eit* analysis of the dental adhesive, it was observed that the control group exhibited higher values than the AA-24H, AA-14D, and BLE-24H groups ($P < .05$). There was no statistical difference between the control group and the enamel surface treated with the α-tocopherol antioxidant solution, regardless of the bleaching process and luting period ($P > .05$) (Table 4).

Resin Cement

Regarding the resin cement, the BLE-24H group exhibited lower HIT values, compared to the other experimental groups ($P < .05$) (Table 5). The control group showed no significant difference in HIT compared to the other groups ($P > .05$). Regarding the Eit*, the control group showed higher values than the AA-14D, αT-24H, BLE-24H, and BLE-AA-24H groups ($P < .05$). (Table 5).

Degree of Conversion

Dental Adhesive

The DC for the dental adhesive and resin cement layers are listed in Table 6. For the dental adhesive, the less value was found for the group in which the enamel surface was bleached and luted mediately after the bleaching treatment (BLE-24H group), compared to the other groups ($P < .01$) (Table 6). No difference were found among other experimental groups ($P > .05$).

Resin Cement

For the DC of the resin cement, the control group showed a significant difference, compared to the BLE-24H group ($P < .05$). The αT-24H group showed a higher DC, compared to the BLE-24H and BLE-14D groups ($P < .05$) (Table 6).

2.5 Discussion

The bleaching process and antioxidant agents influenced the color stability of the restorative assembly and the mechanical properties of the adhesive system and resin cement; therefore, the first null hypothesis was rejected. The assessment of the influence of aging periods on the color stability of ceramic laminates led to the rejection of the second null hypothesis.

The artificial accelerated aging method allows the evaluation of the physicochemical behavior of the luting agents applied during luting procedures and simulates the conditions of the oral environment.[1] This methodology has been used to assess the color stability of dental materials and ceramic restorations.[7,8] Chromatic changes are directly related to intrinsic factors (type and concentration of the compounds inserted in the resin matrix and its hydrolytic degradation);[32] extrinsic factors (consumption of beverages and foods containing pigments in their composition, smoking, humidity, heat, and UV irradiation);[33] or iatrogenic factors (inefficient polymerization of the restoration assembly).[34]

The aging process promoted clinically perceptible chromatic changes ($\Delta E > 3.3$) for all experimental groups, regardless of the aging period (Table 3); these changes were perceptible to the human eye.[7,8] For the antioxidant agents, the ascorbic acid action applied after the dental bleaching procedure (BLE-AA-24H and BLE-AA-14D groups) resulted in similar color changes, compared to the bleached and nonantioxidant-treated enamels (BLE-24H and BLE-14D), in contrast to the α-tocopherol solution, which showed lower color alterations values for the first aging cycle (Table 3). The bleaching mechanism is based on an oxidation process that releases oxygen free radicals, promoting the breaking of organic molecules and converting them to carbon dioxide and water, which is released with singlet oxygen in the long term.[25] According to Naidu *et al.*[35] ascorbic acid is a hydrophilic compound with high solubility in aqueous media, and its effectiveness is directly proportional to the concentration of water and oxygen, and the surface pH level.[25,35] It can be speculated that the hydrophilic components of the ascorbic acid absorbed the water derivate from the bleaching reaction, which could have neutralized the antioxidant solution, corroborating the results of this study (Table 3).

In general, there was a decrease (nonsignificant difference) in the chromatic alterations between the aging periods of 756 and 252 h, except for the AA-14D experimental group (Table 3). These results corresponded with those of previous studies in which the color

change was evaluated in the first 300 h of aging; after this period, the color alteration was stabilized.[7,8,36,37] Strazzi-Sahyon *et al*.[8] stated that after 300 h of artificial accelerated aging, a decrease in water incorporation in the resin matrix occurs, stabilizing the polymer chains.[8] This performance could be attributed to the composition of the luting agents. Variolink Esthetic resin cement contains urethane dimethacrylate (UDMA) as a monomer (Table 1), which is characterized as a hydrophobic component that minimizes water sorption into the resin matrix.[7]

Furthermore, the Tetric N-Bond Universal adhesive system contains the methacrylated carboxylic acid polymer (MCAP) and decandiol dimethadrylate (D3MA) as monomers (Table 1). The MCAP reacts and adheres to hydroxyapatite crystals, and the adhesion of this component to the dental substrate is proportional to the concentration of carboxylic acid groups along the polymeric chain.[38,39] D3MA monomer, classified as a hydrophobic monomer, prevents water sorption into the resin matrix and promotes the adhesive's reaction with less polar monomers included in the resin cement, yielding a strong and durable interface.[38,39] In the present study, the adhesive system contained on the enamel was previously photoactivated to the luting process, since a previous study reported more satisfactory color stability and mechanical properties of the adhesive interface of ceramic laminate restorations when this protocol is adopted.[8] The Tetric N-Bond Universal adhesive system presents ethanol and water as solvents (Table 1). The water present in the adhesive system could have been incorporated by hydrophilic components present in the resin cement that could promote a hydrolytic degradation of the interface, yielding a high color alteration of the restorative assembly.[8] Thus, the previous activation of the adhesive system may facilitate the volatilization of the solvents by the gradual temperature increase from the light-curing unit.[1] All these factors corroborate and justify the tendency of the nondifference in chromatic alterations through the aging periods (Table 3).

The assessment of the mechanical properties of the luting agents applied in ceramic laminate veneer restorations is extremely important, as the luting interface is exposed to the oral cavity, possibly jeopardizing the material properties by the dissolution or leaching of unpolymerized resin monomers.[7,40] The alteration of the physicochemical and mechanical properties of the resin interface can result in microleakage, adhesion of biofilm and bacteria, recurrent caries, postoperative sensitivity, color alteration, decrease in wear resistance, gingival inflammation, and consequently, ceramic restoration failure.[41,42] The mechanical

properties (nanohardness, elastic modulus, and DC) of the adhesive and resin cement layers were influenced by the bleaching treatment and the antioxidant solutions (Tables 4–6).

The group of the ceramic restorations luted onto the enamel surface 24 hours after the bleaching procedure (BLE-24H group) exhibited a lower HIT, Eit*, and DC of the adhesive system and resin cement, compared to the control group (Tables 4–6). Freire *et al*.[21] stated that the effects of bleaching are the presence of residual hydrogen peroxide in the interprismatic area of the enamel and the oxygen bubbles released in long term.[21] These effects prevent the infiltration of monomers into dental structures and inhibit polymerization.[21] Furthermore, the propagation of vinyl free radicals during light activation is interfered by the presence of residual free radicals from hydrogen peroxide, resulting in premature polymer chain termination and inadequate polymerization.[21] These factors corroborate and justify the inferior mechanical properties of the adhesive interface of the BLE-24H group (Tables 4–6).

Thus, studies have suggested that the waiting period prior to performing the restorative procedure should vary from 1 to 4 weeks, to allow time for the elimination of the residual oxygen from the dental substrate.[10,21,31] In this study, a waiting period of 14 days was investigated. Although the mechanical properties of the adhesive interface components in the BLE-14D group were numerically lower than those of the control group, there were no statistical differences among these groups (Tables 4-6). Thus, it was observed that the deleterious effects of tooth whitening tend to be neutralized after 14 days. These findings are in agreement with the results reported by Metz *et al*.[43] and Wilson *et al*.[44]

Some studies have suggested the application of alcohol on bleached dental substrates to decrease the deleterious effect of dental bleaching on the shear bond strength of resin materials.[25,45-47] In this context, Sung *et al*.[48] suggested the use of adhesive systems containing organic solvents, such as ethanol and acetone.[48] Organic solvents easily carry resinous monomers onto the conditioned dental surface, which is its water chaser function that displaces water from the surface.[49] The incorporation of ethanol in the α-tocopherol solution may have contributed to the satisfactory color stability of the ceramic restoration assembly and the mechanical properties of the adhesive interface.[25] Furthermore, α-tocopherol is characterized as a hydrophobic component,[50] implying a decreased interaction with water, which promotes less water incorporation into the resinous matrix. The hydrophobicity of the antioxidant agent and the ethanol incorporation in the formulation could have potentialized

the adhesive interface integrity,[25] justifying the satisfactory properties of the BLE-αT-24H group in this study (Tables 3–6).

Several studies report that the use of antioxidant solutions after dental bleaching can assuage the side effects of bleaching on the adhesion of restorations; however, it does not completely reverse it.[15,17,51,52] Notably, the 10% α-tocopherol antioxidant solution was able to reverse the adverse bleaching effects on the chromatic stability of ceramic restorations and mechanical properties of the adhesive interface; the use of this antioxidant solution 24 h after the bleaching procedure promoted similar results to control group. Many studies have evaluated the influence of the bleaching treatment on dentin substrates; however,[15,17,51,52] studies assessing the influence of hydrogen peroxide on the enamel substrate are scarce. The difference among the results obtained in this study and those that evaluated dentin substrates may be related to the substrate morphology and characteristics because dentin contains a higher concentration of water, organic matrix, collagen fibers, and dentinal fluid and tubules that can act as peroxides and oxygen reservoirs.[25,53] Thus, the use of the 10% α-tocopherol antioxidant agent mediately after enamel bleaching is effective and is a promising approach during ceramic laminate veneer luting.

Certain limiting factors in this study include the sensitivity of the nanoindentation test, use of only one resin cement and adhesive system, concentrations of the antioxidant agents, and use of a single hydrogen peroxide concentration. Furthermore, this study is characterized as *in vitro*; therefore, the application of the laboratory results to clinical conditions must be conducted with caution, as an *in vitro* evaluation cannot replicate conditions similar to those in the oral cavity. Further in-depth longitudinal and clinical analyses should be conducted to investigate the influence of distinct antioxidant types, concentrations, and application times to increase the long-term durability of the adhesive interface of ceramic laminate veneers luted onto bleached enamel surfaces using different concentration agents and bleaching techniques. Moreover, additional variables relating the adhesion of ceramic laminates to bleaching procedures are warranted, such as marginal adaptation and permeability analyses, and the influence of the use of antioxidant agents after dental bleaching on the collagen fiber behavior.

2.6 Conclusion

Based on the methodology and findings of this *in vitro* study, the following conclusions were drawn:

1. Color stability, HIT, Eit*, and DC were influenced by the bleaching treatment and antioxidant agents.

2. The 10% α-tocopherol is a suitable antioxidant solution for reversing bleaching effects on dental enamel, as it does not alter the color stability and mechanical properties of the components of resin interface and could be implemented after enamel bleaching during ceramic laminate veneer luting.

3. Color stability was influenced by aging periods.

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Table 1: Materials, classification, chemical compositions, and batch numbers of materials investigated

Material	Classification	Chemical Composition	Batch Number
Tetric N Bond Universal (Ivoclar Vivadent)	Adhesive System	HEMA, 10-MDP, Bis-GMA, MCAP, D3MA, ethanol, water, highly dispersed silicon dioxide, photoinitiator.	X25012
Variolink Esthetic LC (Ivoclar Vivadent)	Light-Cured Resin Cement	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, boroaluminofluorosilicate glass, spheroidal mixed oxide, benzoylperoxide, stabilizers, pigments, ivocerin as initiator Volume of inorganic fillers: 38%, 60-68%.	X43425
Monobond N (Ivoclar Vivadent)	Silane Agent	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-MDP, disulfide acrylate	X17917
Whiteness HP Maxx 35% (FGM)	Bleaching Agent	Thickeners, dye mixture, glycol, inorganic filler, deionized water, 35% hydrogen peroxide.	031218
Ascorbic Acid – Solution (Apothicário)	Vitamin C	Ascorbic acid at 10% and 2.5% alcohol, adjustment of pH=7.0 with triethanolamine.	_
α – Tocopherol – Solution (Apothicário)	Vitamin E	α-tocopherol at 10% and 2.5% alcohol, adjustment of pH=7.0 with triethanolamine.	_

Abbreviations: HEMA, 2-hydroxyethylmethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-A glycidyl methacrylate; MCAP, methacrylated carboxylic acid polymer; D3MA, decandiol dimethadrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

Table 2: Division of the experimental groups

Groups Abbreviation	Group Treatment Detail
Control	Luting of ceramic laminate veneers onto enamel surface unbleached and non-treated with antioxidant agents
AA-24H	Luting of ceramic laminate veneers onto unbleached enamel surface after 24 hours of 10% ascorbic acid antioxidant application
AA-14D	Luting of ceramic laminate veneers onto unbleached enamel surface after 14 days of 10% ascorbic acid antioxidant application
αT-24H	Luting of ceramic laminate veneers onto unbleached enamel surface after 24 hours of 10% α-tocopherol antioxidant application
αT-14D	Luting of ceramic laminate veneers onto unbleached enamel surface after 14 days of 10% α-tocopherol antioxidant application
BLE-24H	Luting of ceramic laminate veneers onto bleached enamel surface after 24 hours associated with no antioxidant agents
BLE-14D	Luting of ceramic laminate veneers onto bleached enamel surface after 14 days associated with no antioxidant agents
BLE-AA-24H	Luting of ceramic laminate veneers onto bleached enamel surface after 24 hours of 10% ascorbic acid antioxidant application
BLE-AA-14D	Luting of ceramic laminate veneers onto bleached enamel surface after 14 days of 10% ascorbic acid antioxidant application
BLE-αT-24H	Luting of ceramic laminate veneers onto bleached enamel surface after 24 hours of 10% α-tocopherol antioxidant application
BLE-αT-14D	Luting of ceramic laminate veneers onto bleached enamel surface after 14 days of 10% α-tocopherol antioxidant application

Table 3: Mean color change values (ΔE ± standard deviation) as function of the experimental groups and aging periods

	252 Hours	**504 Hours**	**756 Hours**
Control	3.49 ± 2.31 D a	5.33 ± 3.88 AB a	6.52 ± 5.24 A a
AA-24H	7.12 ± 2.72 BCD a	3.60 ± 1.69 B a	6.91 ± 5.91 A a
AA-14D	5.73 ± 1.70 CD b	10.55 ± 4.18 A a	9.17 ± 3.86 A a
αT-24H	5.91 ± 3.27 BCD a	6.23 ± 3.47 AB a	9.94 ± 5.13 A a
αT-14D	7.02 ± 2.95 BCD a	5.63 ± 2.97 AB a	4.45 ± 2.45 A a
BLE-24H	12.09 ± 7.71 ABC a	8.35 ± 3.76 AB a	9.54 ± 5.61 A a
BLE-14D	15.19 ± 1.07 A a	6.20 ± 1.70 AB b	6.90 ± 3.14 A b
BLE-AA-24H	11.32 ± 5.59 ABC a	8.33 ± 4.05 AB a	9.96 ± 4.34 A a
BLE-AA-14D	12.20 ± 4.37 AB a	6.56 ± 3.15 AB b	6.16 ± 3.45 A b
BLE-αT-24H	7.34 ± 2.00 BCD a	7.94 ± 5.08 AB a	8.77 ± 3.95 A a
BLE-αT-14D	9.08 ± 3.35 ABCD a	7.09 ± 2.75 AB a	8.69 ± 1.13 A a

Different letters, uppercase in column and lowercase in row, indicate statistically significant differences ($P<.05$).

Table 4: Mean ± standard deviation values of the nanohardness (HIT) (MPa) and elastic modulus (Eit*) (GPa) of the dental adhesive as function of the experimental groups

	HIT	**Eit***
Control	264.58 ± 25.03 AB	5.22 ± 0.10 A
AA-24H	208.25 ± 48.08 AB	3.82 ± 1.06 C
AA-14D	199.28 ± 32.17 B	3.56 ± 0.42 C
αT-24H	242.73 ± 15.07 AB	4.54 ± 0.64 ABC
αT-14D	289.38 ± 27.59 A	5.05 ± 0.41 AB
BLE-24H	197.56 ± 56.76 B	3.99 ± 0.92 BC
BLE-14D	234.70 ± 47.37 AB	4.39 ± 0.43 ABC
BLE-AA-24H	247.99 ± 27.94 AB	4.20 ± 0.55 ABC
BLE-AA-14D	243.27 ± 39.34 AB	4.11 ± 0.21 ABC
BLE-αT-24H	275.54 ± 42.32 AB	4.26 ± 0.26 ABC
BLE-αT-14D	206.77 ± 12.16 B	4.26 ± 0.41 ABC

Different letters in column indicate statistically significant differences for each mechanical property analyzed (P<.05).

Table 5: Mean ± standard deviation values of the nanohardness (HIT) (MPa) and elastic modulus (Eit*) (GPa) of the resin cement as function of the experimental groups

	HIT	**Eit***
Control	714.33 ± 43.22 A	11.38 ± 0.51 A
AA-24H	718.31 ± 38.28 A	10.85 ± 0.81 AB
AA-14D	622.49 ± 24.93 A	9.73 ± 0.23 BC
αT-24H	640.40 ± 51.57 A	9.91 ± 0.93 BC
αT-14D	646.10 ± 31.70 A	10.53 ± 0.69 AB
BLE-24H	505.49 ± 54.37 B	8.82 ± 0.96 C
BLE-14D	641.65 ± 68.07 A	10.49 ± 0.85 AB
BLE-AA-24H	628.22 ± 30.36 A	9.98 ± 0.39 BC
BLE-AA-14D	686.64 ± 89.18 A	11.38 ± 0.32 A
BLE-αT-24H	696.36 ± 23.84 A	10.79 ± 0.45 AB
BLE-αT-14D	653.25 ± 35.25 A	10.60 ± 0.49 AB

Different letters in column indicate statistically significant differences for each mechanical property analyzed (P<.05).

Table 6: Mean ± standard deviation values of the degree of conversion (DC) (%) of the dental adhesive and resin cement as function of the experimental groups

	Adhesive System	**Resin Cement**
Control	97.53 ± 0.83 A	85.28 ± 2.20 AB
AA-24H	95.82 ± 0.95 A	85.65 ± 1.12 AB
AA-14D	96.05 ± 1.77 A	85.72 ± 1.06 AB
αT-24H	96.67 ± 0.92 A	86.40 ± 1.92 A
αT-14D	97.05 ± 0.81 A	83.97 ± 0.99 ABC
BLE-24H	89.97 ± 3.01 B	79.54 ± 1.04 C
BLE-14D	95.44 ± 1.08 A	80.90 ± 4.74 BC
BLE-AA-24H	97.11 ± 1.29 A	84.36 ± 2.50 ABC
BLE-AA-14D	98.12 ± 0.38 A	84.05 ± 2.67 ABC
BLE-αT-24H	97.26 ± 0.78 A	84.75 ± 2.27 ABC
BLE-αT-14D	97.60 ± 0.41 A	84.15 ± 3.28 ABC

Different letters in column indicate statistically significant differences for each material analyzed (P<.05).

Figure 1. Specimen preparation for mechanical properties analyses. A. Specimens sectioned perpendicularly to the adhesive interface before obtaining a slice of the central portion of the restoration assembly. B. Slice, approximately 3.0 mm in thickness. C. Specimens embedded in acrylic resin, finished with #320, #600, #800, and #1200 grit silicon carbide papers and polished with #6, #3, #1, and #0.25 µm diamond pastes. D. Specimens immersed in Hanks balanced salt solution to prevent ion loss and cell degradation. E. Assessment of hardness (HIT) and elastic modulus (Eit*) of the adhesive system and resin cement layers under a load of 1000 µN using a nanoindenter machine (Anton Paar). F. Assessment of the degree of conversion (DC) of the adhesive system and resin cement layers using a confocal Raman microscope (Renishaw).

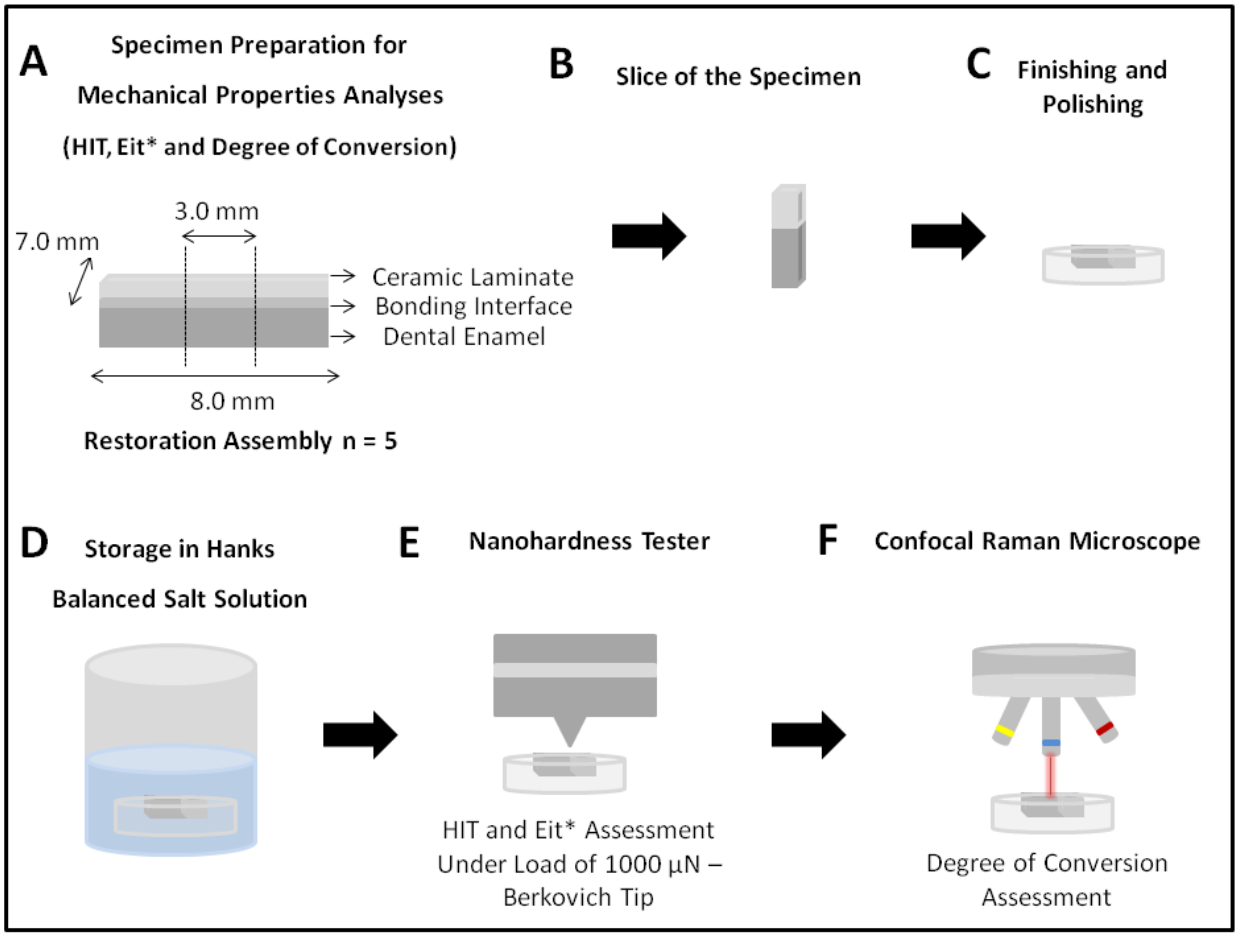
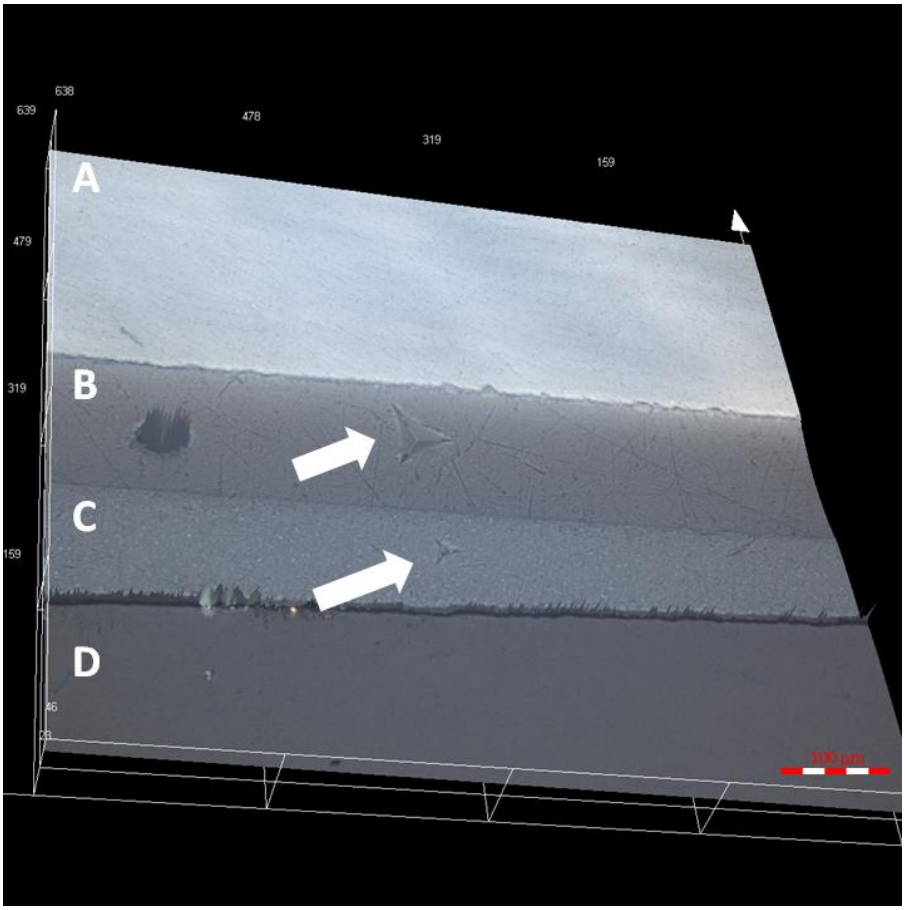


Figure 2. Laser scanning confocal micrograph of the representative image of the adhesive interface. A. Enamel substrate B. Adhesive system layer; arrow: nanoindentation in the adhesive system substrate C. Resin cement layer; arrow: nanoindentation in the resin cement substrate D. Ceramic substrate



Chapter 2

3 CHAPTER 2 - IN VITRO STUDY ON HOW ANTIOXIDANT SOLUTIONS AFFECT ENAMEL SURFACE CHARACTERISTICS AND BOND INTERFACE OF CERAMIC LAMINATE VENEERS LUTING AFTER DENTAL BLEACHING[‡]

Short Title: Reversal of oxidizing effect by antioxidants on bleached enamel

3.1 Abstract

Objectives: This *in vitro* study aimed to determine the effect of antioxidant solutions used after dental bleaching on the shear bond strength and adhesive interface morphology of ceramic laminate veneer luting. Additionally, effects on the enamel surface characteristics of hydrogen peroxide neutralization, wettability, surface energy, total free interaction energy, morphology, and chemical composition of enamel were assessed.

Methods: Total 137 bovine incisors were divided into experimental groups, according to the surface treatment (unbleached and bleached enamel), antioxidant types (control; 10% ascorbic acid and 10% α-tocopherol), and periods of luting of ceramic laminates (24 h and after 14 days). Shear bond strength was assessed using microtensile test before and after thermal cycling (5760 cycles, 5–55°C) (n=6). The morphology of the adhesive interface was assessed using a confocal laser scanning microscope (n=3). Hydrogen peroxide neutralization analysis was performed using a spectrophotometer (n=5). The contact angle (n=10), surface energy, and total free interaction energy (n=10) were measured using an automatic goniometer, while enamel morphology and chemical composition were assessed by scanning eletron microscopy (n=3). Shear bond strength and enamel surface properties data were subjected to ANOVA followed by Tukey´s test (α=.05). Adhesive interface micrographs were evaluated by the inter-examiner Kappa test and subjected to Kruskal-Wallis and Dunn´s tests (α=.05).

Results: In general, thermal aging decreased the shear bond strength values of the luting agents to enamel (P<.05). The α-tocopherol solution was able to reverse the oxidizing effect from dental bleaching, increasing the shear bond strength values and preserving the integrity of the adhesive interface morphology (P<.05). Moreover, the α-tocopherol antioxidant agent promoted higher hydrogen peroxide neutralization and wettability values after dental

[‡] Standardization according to Journal of Dentistry Guidelines
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bleaching (P<.05). Dental bleaching influenced the enamel surface, decreasing the surface energy and total free interaction energy values (P<.05).

Conclusion: α-tocopherol was able to reverse the oxidizing effects of dental bleaching, improving the enamel surface properties, as well as the adhesion and interface morphology of ceramic laminate veneer restorations.

Clinical Relevance: The waiting time of ceramic laminate veneer luting can be optimized after enamel bleaching by applying the α-tocopherol antioxidant.

Keywords: Ceramics; Enamel; Hydrogen Peroxide; Shear Strength; Surface Properties; Tooth Bleaching.

3.2 Introduction

In previous years, new techniques and adhesive restorative materials have been introduced in the dental market, including new ceramic materials that are fabricated chairside using CAD/CAM technology (computer-aided design and computer-aided manufacturing), allowing the professional to design, mill, and lute the indirect restorations in one session [1,2]. Lithium disilicate ceramics have been amply used in indirect restorations due to their optical properties, aesthetic nature [3], chemical durability, high translucency, and biocompatibility [4]. Furthermore, lithium disilicate reinforced ceramics present great mechanical and resistance properties, providing minimally invasive procedures with laminate veneers luted to the dental structure with minimal or no dental wear [5,6]. Their combination with luting agents allows mimicry of the texture, shade, and color, providing a natural and harmonious smile [5-7]. However, depending on the chroma intensity of the underlying dental substrate, and as a result of the thinness of the veneers, just the use of luting agents would not mimic the restorative assembly, affecting the final result [8-9]. In order to neutralize the effect of the dental substrate, dental bleaching before luting the ceramic laminate veneers could be recommended [8,9].

In-office and at-home tooth bleaching techniques differ in terms of the bleaching agent type, concentrations, time of action, and association with irradiation light [10]. However, both techniques have in common the release of oxygen as the main product of the reaction, which

remains on tooth surfaces [11,12], hindering the penetration of resinous monomers [12], as well as inhibiting polymerization [13]. Several studies reported an immediate adverse effect on bond strength of adhesive restorations to tooth substrates, suggesting a delay of approximately 7–28 days to overcome this problem [14,15].

Despite the undesirable effects, such as a decrease in microhardness, changes in tooth structure, and bond strength reduction of resin restorations to dental tissues, hydrogen peroxide presents oxidant intracellular action during cell activities [16,17]. The reactive oxygen species resulting from its degradation are greatly unstable and reactive, interacting with cellular components, such as nucleic acids, proteins, and lipids [16,17]. Higher hydrogen peroxide concentrations have been commonly used in dental offices due to their ability to oxidize quickly the organic compounds that are present in dental structures that promote tooth darkening [10]. The free radicals resulting from the oxide-reduction reaction have a low molecular weight that yields higher diffusion though the dentinal tubules, promoting oxidative stress, which leads to pulp cells, post-operative sensitivity, and in severe cases, acute inflammatory conditions and pulp necrosis [16-18].

Vargas et al. [16] endorsed the α-tocopherol efficacy of protection and prevention of oxidative stress diffusion, enhancing the cell viability during the bleaching treatment [16,17]. Moreover, some previous studies have suggested that the use of antioxidant agents after dental bleaching, such as ascorbic acid and sodium ascorbate [12,13,19,20] can reverse the adhesion reduction between bonding materials and dental tissues [12,19,21,22]. Because of the lack of in-depth information regarding the effect of α-tocopherol solution on the shear bond strength of luting materials and on the adhesive interface morphology after dental bleaching, there is no consensus on how the antioxidant solutions influences the enamel surface properties and the effectiveness of these solutions on hydrogen peroxide neutralization. If the antioxidant effectiveness of α-tocopherol in reversing the hydrogen peroxide oxidative effect on the bond strength is proven, concomitantly with its cellular protective effect against hydrogen peroxide toxicity [16,17], an effective protocol could be established.

Thus, the purpose of this *in vitro* study was to determine the effect of antioxidant agents after dental bleaching on the shear bond strength of ceramic laminate restorations subjected to thermal aging, as well as its effects on the adhesive interface morphology, hydrogen peroxide neutralization, and enamel surface properties, such as wettability, surface

energy, total free interaction energy, morphology, and chemical composition of antioxidant-treated enamel. The null hypotheses tested were: 1) the bleaching treatment and antioxidant solutions would not result in differences in the shear bond strength and adhesive interface morphology of ceramic laminate veneers luting, as well as in the hydrogen peroxide neutralization and in the enamel surface properties; 2) thermal aging would not promote differences in the shear bond strength of ceramic laminate veneers; 3) the distinct evaluation times would not cause differences in hydrogen peroxide neutralization.

3.3 Material and Methods

Specimen Preparation

This research was approved by the Local Institutional Ethics Committee on the Use of Animals (#01058-17). Table 1 lists the details of the materials assessed in this study.

One hundred thirty-seven bovine teeth, from cattle aged 2.5–3.0 years old, were used [23]. Teeth containing stains, morphological crown alteration, excessive incisal loss, cracks, and fractures on the enamel surface were discarded. Periodontal curettes were used to mechanically clean the teeth, followed by prophylaxis with pumice stone and water. To identify possible cracks and/or fractures, a magnifying glass with a magnification of ×4 (Bio-Art Equipamentos Odontológicos Ltda, São Carlos, SP, Brazil) was used to verify the integrity of the teeth [6].

Anatomic crowns were separated from the radicular portion 1.0 mm above the cementum-enamel joint trough a transversal section, using a low-speed diamond saw under water cooling connected to a metallographic cutter machine (Isomet 1000; Buehler, Lake Bluff, IL, USA). Subsequently, four cuts were made on the anatomic crown, two in the mesio-distal direction at a distance of 7 mm, and another two cuts in the cervico-incisal direction, 8-mm apart, obtaining an enamel fragment of 7 × 8 × 4 mm. The buccal surfaces of the enamel samples were abraded and flattened using #600 grit silicon carbide papers (Buehler, Lake Bluff, IL, USA) [5]. Ninety nine enamel specimens were randomly divided into 11 groups for the analysis of shear bond strength (n=6) and adhesive interface morphology (n=3), according to the bleaching treatment (bleached and unbleached enamel), antioxidant solutions (control, 10% ascorbic acid, and 10% α-tocopherol), and luting periods of the ceramic laminates (24 h and after 14 days). Thirty-eight specimens were used to assess the influence of bleaching

treatment and antioxidant agents on enamel surface properties: ten specimens for contact angle analysis, ten specimens for surface energy and total free interaction energy analyses, and 18 samples for scanning electron microscopy and energy dispersive spectroscopy. The enamel sample distribution for the analyses is shown in Figure 1.

Shear Bond Strength

Table 2 presents the details of the experimental groups. For shear bond strength analysis, sixty-six enamel blocks were included in PVC cylinders with acrylic resin (Clássico, São Paulo, SP, Brazil), abraded and flattened manually using #320 and #600 grit silicon carbide papers (Buehler, Lake Bluff, IL, USA) to expose the enamel surface under water cooling (Figure 2 – A). The samples were cleaned in an ultrasonic unit (Cristofoli, Campo Mourão, PR, Brazil) with deionized water for 8 min after using the abrasive sandpaper. The enamel samples were then randomly divided into 11 experimental groups.

In the control group, the enamel surface was etched with 37% phosphoric acid (FGM, Joinville, SC, Brazil) for 30 s, followed by rinsing with deionized water and drying with air jets. The adhesive system (Tetric N Bond Universal; Ivoclar Vivadent, Schaan, Liechtenstein) was actively applied to the surface for 20 s (Figure 2 – B). An air jet was used for 5 s to evaporate the solvent, and then the adhesive was polymerized using a polywave LED light curing unit (VALO; Ultradent, South Jordan, UT, USA), in standard mode (1000 mW/cm^2) for a period of 10 s [6].

The resin cement cylinders were prepared using a condensation silicone mold (7 x 8 x 1 mm) (Oranwash, Zhemarck, Rovigo, Italy) containing eight orifices with an internal diameter of 1.0 mm and height of 1 .0 mm, each [24]. The silicone mold was positioned on the properly treated enamel surface (Figure 2 – C), and the orifices were filled with resin cement (neutral shade Variolink Esthetic LC; Ivoclar Vivadent, Schaan, Liechtenstein) using an applicator tip and an explorer probe. The silicone matrix was covered with a Mylar strip, and then the cement cylinders were cured for 30 s using the VALO light (Ultradent, South Jordaan, UT, USA) in standard mode (Figure 2 – D). After polymerization of the resin cylinders, the silicone mold was carefully removed using a scalpel blade to obtain eight cylinders bonded to the dental enamel.

AA_{24H} group samples were prepared as described for the control group; however, 24 h prior to the luting procedure, treatment with 20 μL of 10% ascorbic acid antioxidant agent (Apothicário, Araçatuba, SP, Brazil) was performed using a calibrated syringe [22]. The solution remained in contact with the enamel surface for 15 min, followed by rinsing with deionized water and drying with air jets [22]. The samples were kept in recipients with artificial saliva (pH=7.0; Apothicário, Araçatuba, SP, Brazil) at 37°C. The AA_{14D} group samples were treated as described for the AA_{24H} group; however, the luting procedure of the ceramic restorations was performed after 14 days of the antioxidant solution application. The αT_{24H} and αT_{14D} group samples were treated as described for AA_{24H} and AA_{14D} groups, respectively; however, 20 μL of 10% α-tocopherol antioxidant agent (Apothicário, Araçatuba, SP, Brazil) was applied [22].

Enamel samples of the BLE_{24H} group were bleached with 35% hydrogen peroxide (Whiteness HP Maxx; FGM, Joinville, SC, Brazil), and three drops of peroxide were mixed with one drop of thickener. Using a calibrating syringe, 40 μL of the mixed product was applied to the enamel surface that remained in contact with the substrate for 15 min. Subsequently, the bleaching agent was removed using sterile cotton and absorbent papers, and the peroxide product was applied, for 3 total applications of 15 min each, totaling 45 min of exposure to the bleaching agent. The product was removed with deionized water, and the enamel surface was dried with an air jet. The bleaching treatment was repeated for three sessions, with an interval of one week between each application. During the interval between each session, the samples were stored in artificial saliva (pH7.0) at 37°C. At the end of the bleaching treatment, the specimens were rinsed with deionized water and dried with an air jet. Then, 24h post dental bleaching, the luting procedure of the ceramic laminate veneers was carried out as described above.

The samples of the BLE_{14D} group were treated as described for the BLE_{24H} group; however, the luting process of the ceramic restoration was carried out after 14 days of bleaching treatment. BLE_{AA24H}, BLE_{AA14D}, $BLE_{\alpha T24H}$, and $BLE_{\alpha T14D}$ group samples were prepared as described for AA_{24H}, AA_{14D}, αT_{24H}, and αT_{14D}, respectively; however, the enamel surfaces were bleached beforehand by the action of antioxidant agents.

Four cylinders were selected for the mediated test (24 hours after luting), and the other four cylinders were evaluated after aging in thermal-cycling (5760 cycles, 5°C and 55°C, with a permanence time of 28 seconds in each solution and transfer time of 2 seconds) using an

MSCT-3 Plus thermal cycle simulation machine (ElQuip, São Carlos, SP, Brazil) [25]. For shear bond strength test, the samples were positioned and fixed in a metallic device of the mechanical testing machine (Microtensile OM100; Odeme, Luzerna, SC, Brazil). The samples were positioned so that the resin cement cylinders were aligned in the direction of the force application. A 0.2 mm diameter orthodontic wire (NiCr; Morelli, Sorocaba, SP, Brazil) was used to apply the load on the cylinders at a speed of 0.7 mm/min to assess the bond strength (Figure 2 – E). The bond strength (BS) of each resin cylinder was calculated in MPa according to the following formula [24,26]:

$$BS = F/A,$$

where F is the force required for rupture (N), and A is the adhesive area of the resin cylinders (mm^2).

After the bonding test, the specimen failure modes were analyzed using a stereoscopic microscope (Stemi SV11; Zeiss, NY, USA) at magnifications of × 6 and × 66. The failure types were divided into 1) adhesive failure, 2) mixed failure, 3) cohesive failure in resin cement, and 4) cohesive failure in enamel. Representative samples of the specimens with the predominant failure type for each experimental group were coated with gold using a sputter machine (Balzers SCD-050 sputter coater, Germany), and the fracture pattern was qualitatively evaluated using a scanning electron microscope (SEM-JSM5600LV, LEOL, Tokyo, Japan) to obtain micrographs with original magnification × 100 and × 500 [26].

Adhesive Interface Morphology

Thirty-three enamel blocks (n=3) were submersed into distilled water with fluorescein diacetate dye (0.1%) (FDA, Sigma Chemical Co., St. Louis, MO, USA) for 4 h to promote dye infiltration into the enamel hydroxyapatite crystals (Figure 2 – F), and the samples were then dried with air jets [25]. For adhesive interface morphology analysis, rhodamine B dye was incorporated into the resin cement (16 μg/g) and into the adhesive system (26.5 μg/ml) (Figure 2 – G) [25]. The enamel samples were divided randomly into 11 groups and treated similarly according to the experimental groups previously reported.

Thirty-three lithium disilicate veneers (7 × 8 × 0.6 mm) were made from high-translucency lithium disilicate blocks (shade A1-IPS e.max CAD; Ivoclar Vivadent, Schaan, Liechtenstein) using a low-speed diamond saw installed in an Isomet 1000 (Buehler, Lake Bluff, IL, USA) under constant water irrigation. The ceramic restorations were sintered in the

manufacturer's furnace (Programat EP 5000; Ivoclar Vivadent, Schaan, Liechtenstein) for 1 h at 780^{o}C, following the manufacturer´s instructions [7].

The ceramic surface was etched with 5% hydrofluoric acid (FGM, Joinville, SC, Brazil) for 20 s. Residues from the ceramic etching were removed with air/water spray and dried with an air jet. Onto the conditioned ceramic, the silane agent (Monobond Plus; Ivoclar Vivadent, Schaan, Liechtenstein) was actively applied for 60 s and air-dried for 5 s. Then, a layer of adhesive (Tetric N Bond Universal; Ivoclar Vivadent, Schaan, Liechtenstein) was applied, and an air jet was used for 20 s to evaporate the solvent without polymerization [5]. Light-cured resin cement (neutral shade, Variolink Esthetic LC; Ivoclar Vivadent, Schaan, Liechtenstein) was directly applied to the inner ceramic surface to avoid bubble formation, and the ceramic laminate was positioned on the enamel surface previously treated according to the respective experimental groups. Prior to the restoration assembly polymerization, a mass of 4.9 N was placed on the ceramic restoration to standardize the resin cement thickness, and then removed [5]. The overflow luting agent was removed with a microbrush, and the assembly was light-cured using a polywave light source (VALO - standard mode; Ultradent, South Jordaan, UT, USA) for 30s. The assembly restoration was polymerized inside an opaque black box to avoid external light interference, and the distance was standardized to 1 mm between the light tip and the specimen [5]. After the bonding procedure, all specimens were kept in Hanks' solution at 37°C for 24 h.

Each specimen was submitted to an Isomet 1000 to obtain three middle slices of 2.0 mm thickness each (Figure 2 – H). The slices were stored in Hanks' solution to conserve the pH and preserve the ion loss of the restoration [25]. The analysis analyzed using a confocal laser scanning microscope (Leica TCS SP2; Leica Microsystems, Heidelberg, Germany), under argon (488 nm) and He-Ne (453 nm) lasers (Figure 2 – I). The confocal micrographs were registered in the fluorescent mode using an oil immersion objective lens (40×, numerical aperture 1.25) [25,27]. Three regions along the adhesive interface of each specimen were assessed; one record was prepared for each edge of the restoration specimen, and another for the evaluation in the middle region. The micrographs were obtained with a 1 μm z-step to optically section the samples to a depth of up to 20 μm below the surface. The images were analyzed using two previously calibrated evaluators [27]. The evaluation was carried out using the double-blind method. In cases where there was disagreement between the two examiners, the images were evaluated again until a final consensus was reached.

The following criteria were evaluated [27,28]:

1) Quality of the enamel/luting adhesive interface:

Score 0 - no cracks, clear adhesive interface, well organized;

Score 1 – partial presence of cracks, partially organized adhesive interface, present in less than 50% of the interface;

Score 2 – presence of cracks with disorganized interface, present in more than 50% of the interface.

2) Tags Formation into enamel:

Score 0 – not detectable;

Score 1 – few visible tags;

Score 2 – uniform tags formation with few lateral branches;

Score 3 – formation of long tags with many lateral branches.

3) Tag penetration depth:

Score 0 - no tags

Score 1 – tags ≤ 3 μm on average;

Score 2 – tags = 3 to 6 μm on average.

Hydrogen Peroxide Neutralization

To confirm the effectiveness of the antioxidant agents in neutralizing hydrogen peroxide, a standard curve (absorbance vs. concentration) was plotted out [18,29-31]. To prepare the absorbance curve, a mixture of deionized water, leucocrystal, acetate buffer, hydrogen peroxide, and peroxidase solutions was used [18,29-31]. In this analysis, six experimental groups (n=5) were evaluated: unbleached with no antioxidant agent, unbleached + ascorbic acid, unbleached + α-tocopherol, bleached with no antioxidant agent, bleached + ascorbic acid, and bleached + α-tocopherol. In the bleached groups, a drop of peroxide from the bleaching product was added to glass test tubes containing 100 μL of acetate buffer solution. In the bleached and unbleached groups associated with the use of antioxidant agents,

20 μL of ascorbic acid and α-tocopherol was individually incorporated into different test tubes containing the buffer solution. After the incorporation of antioxidant solutions and hydrogen peroxide for each group´s protocol, 25 μL of this mixture was incorporated with 2750 μL of deionized water, 100 μL of violet leucocrystal (0.5 mg/mL; Sigma Chemical Co., St. Louis, MO, USA), and 50 μL of peroxidase enzyme (1 mg/mL; Sigma Chemical Co., St. Louis, MO, USA) (Figure 2 – J). This mixed solution was diluted to a final volume of 3 mL of deionized water [18,29-31].

This method is based on the reaction of hydrogen hydroxide with the violet leucocrystal dye, catalyzed by the peroxidase enzyme [18,29-31]. The solution color varies according to the peroxide concentration; thus, the absorbance curve is proportional to the concentration of hydrogen peroxide [18,29-31]. The hydrogen peroxide neutralization effectiveness of antioxidant solutions was calculated using the absorbance × concentration curve using a visible reflection spectrophotometer (model UV-2450; Shimadzu, Kyoto, Japan) (Figure 2 – K). The calibration factor (CF) equivalent to the ratio of the standard hydrogen peroxide solution for the absorbance curve was calculated using the following formula [29,31]:

$$CF = \text{Specimen Solution} / \text{Absorbance}$$

The analysis was evaluated in five different periods: 15 min, 30 min, 1 h, 2 h, and 4 h after the preparation of the final solution. The absorbance curve analysis was performed 15 min after this period for the antioxidant agents' performance, as reported previously.

Contact Angle

Ten enamel blocks (7 × 8 × 4 mm) were used to analyze the contact angle of the antioxidant solutions on the enamel surface before and after bleaching treatment using an automatic goniometer (DSA 100S; Krüss, Hamburg, Germany). A volume of 0.3 μL of each solution (distilled water [control], ascorbic acid, and α-tocopherol) was automatically dispensed into different quadrants of the unbleached enamel surface using a glass syringe (500 μL) and a 0.5 caliber needle (Figure 2 – L) [32]. After 1 s of dripping, the shape of the drop was recorded using a camera to capture the images, and the contact angles (right and left) were automatically measured using the tangent method (Drop Shape Analysis DSA4 Software, version 2.0-01; Krüss, Hamburg, Germany) [32]. Each drop was measured 5 times for 5 s, and an arithmetic mean was obtained for each solution and for each specimen.

Subsequently, the bleaching procedure was performed as previously reported, and a new contact angle measurement was performed [32].

Surface Energy and Total Free Interaction Energy

Ten enamel samples (7 × 8 × 4 mm) were used to evaluate the effect of the bleaching procedure on surface energy and total free interaction energy of the enamel (Figures 1 and 2). All samples were stored in Hank´s solution until measurements were taken. The enamel free surface energy (γs) and its nonpolar (γ^{LW}: Lifshiz van der Waals) and polar (γ^{AB}: acid/base) components were calculated by contact angle measurements on enamel using an automatic goniometer (DSA 100S; Krüss, Hamburg, Germany) [32]. Three specific solutions with established surface energy parameters were used: water (polar), methylene iodide (apolar), and ethylene glycol (polar with acid/base component) [32].

On unbleached enamel surface, 0.3 μL of each solution was automatically dropped on three specific regions, previously determined for each solution, using a glass syringe (500 μL) and 0.5 mm needle of caliber (Figure 2 – M) [32]. The contact angle was determined by the drop image captured by the software (Drop Shape Analysis DSA4 Software, version 2.0-01; Krüss, Hamburg, Germany) installed in the goniometer, and then measured by the tangent method [32]. Each drop was measured five times during 5 seconds at 20°C and relative humidity of 44% ± 6, and an arithmetic mean was calculated [32]. The parameters, such as Lifshiz van der Waals (γ^{LW}, nonpolar component), Lewis acid-base (γ^{AB}, polar component), acid component (γ+; receptor component), and base component (γ-; donor component) of free surface energy (mN/m) were calculated to determinate the free energy of substrate interaction according to the following equation [33]:

$$(1 + \cos\theta)\,\gamma_s = -2\,(\sqrt{\gamma_s}^{LW} - \sqrt{\gamma_L}^{LW}) + (\sqrt{\gamma_s^+\gamma_L^-} + \sqrt{\gamma_s^-\gamma_L^+})$$

The total free interaction energy (ΔG_{sws}^{Total}) between the enamel and water was measured to determine the hydrophilicity/hydrophobicity of enamel surface, according to the following formula:

$$\Delta G_{sws}^{Total} = -2\,(\sqrt{\gamma_s}^{LW} - \sqrt{\gamma_w}^{LW})^2 - 4\,(\sqrt{\gamma_s^+\gamma_s^-} + \sqrt{\gamma_w^+\gamma_w^-} - \sqrt{\gamma_s^+\gamma_w^-} - \sqrt{\gamma_s^-\gamma_w^+}),$$

where $\Delta G_{sws}^{Total} > 0$ characterizes the surface as a hydrophilic surface and $\Delta G_{sws}^{Total} < 0$ as a hydrophobic surface. Subsequently, the bleaching procedure was performed as previously reported, and measurement was carried out anew.

Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS)

For the SEM and EDS analyses, six experimental groups were evaluated (n=3): unbleached and no antioxidant-treated enamel, bleached and no antioxidant-treated enamel, unbleached and ascorbic acid-treated enamel, bleached and ascorbic acid-treated enamel, unbleached and α-tocopherol-treated enamel, and bleached and α-tocopherol-treated enamel (Figure 1). The specimens were sputter-coated with gold (Baltec SCD 050; Balzers, Liechtenstein, Austria) to determine the morphology and chemical composition of the enamel surface. Enamel morphology micrographs were obtained using scanning electron microscopy under ×250 magnification, and the substrate composition was evaluated by energy-dispersive X-ray spectroscopy coupled to the SEM equipment (JSM5600LV; JEOL, Tokyo, Japan). Data on oxygen (O), carbon (C), calcium (Ca), phosphorus (P), sodium (Na), magnesium (Mg), silicon (Si), and gold (Au) were collected from the SEM – EDS analysis under ×250 magnification [34].

Statistical Analysis

Data were submitted to normality (Shapiro-Wilk; Bioestat 2.0 Program) and homogeneity tests (Bartlett; Bioestat 2.0 Program). Shear bond strength and contact angle were analyzed using two-way repeated measures analysis of variance (ANOVA; 5.0 Statview Program; Version 5.0.1). Hydrogen peroxide neutralization was assessed using three-way repeated measures ANOVA (5.0 Statview Program; Version 5.0.1), and surface energy and total free interaction energy were evaluated by one-way repeated measures ANOVA (5.0 Statview Program; Version 5.0.1). The Tukey protected least significant difference test (α=.05) was also performed for all the analyses mentioned above. Adhesive interface morphology was evaluated by the inter-examiner Kappa test and submitted to Kruskal-Wallis and Dunn´s tests (α=.05) (SigmaPlot; Version 12.0).

3.4 Results

Shear Bond Strength

The shear bond strength values are listed in Table 3. The bond strength values of the BLE_{24H} group were lower than those of the αT_{24H}, αT_{14D}, and $BLE_{\alpha T24H}$ groups before thermal cycling (P<.05) (Table 3). Evaluating the post-cycling period, the αT_{24H}, BLE_{AA24H}, and $BLE_{\alpha T24H}$ groups yielded higher shear bond strength values than the BLE_{24H} group (P<.05). Assessing the cycling periods, in general, thermal aging influenced the shear bond strength of luting agents on the enamel substrate, and decreased the bonding values before and after cycling (P<.05) (Table 3). However, there was no significant difference between the cycling periods for the AA_{24H}, AA_{14D}, and αT_{14D} groups (P>.05).

Figures 3 and 4 show the predominance of mixed failures for all experimental groups, regardless of the bleaching treatment, antioxidant agents, luting periods, and thermal-aging times evaluated. Illustrative scanning electron micrographs qualitatively represent the failure type prevalence of the experimental groups, where the luting agent residuals remained on the enamel surface (Figure 5 A–V).

Adhesive Interface Morphology

The adhesive interface morphology scores and representative confocal scanning laser micrographs are presented in Table 4 and Figure 6, respectively. A kappa inter-examiner test was performed and a value of 0.71, 0.73, and 0.72 was obtained to interface quality, tags formation, and tag penetration depth parameters respectively. Regarding the adhesive interface quality, there were no significant differences among the experimental groups (P>.05) (Table 4), with a score of 0 prevalence, once the integrity and well-organized bond interface was observed (Figure 6). Regarding the resin tag formation, the control and $BLE_{\alpha T24H}$ groups presented significant difference to BLE_{24H} group (P<.05) (Table 4). In relation to the depth of tags, there were no significant differences among the experimental groups (P>.05) (Table 4).

Hydrogen Peroxide Neutralization

The hydrogen peroxide neutralization values are listed in Table 5. In general, there was no significant difference in hydrogen peroxide neutralization between the control group and antioxidant solutions for the unbleached group, regardless of the period evaluated (P>.05),

except for the 30 minutes period, in which the α-tocopherol antioxidant solution presented higher values compared to the control and ascorbic acid groups (P<.05) (Table 5). For the bleached group, the α-tocopherol solution promoted greater hydrogen peroxide neutralization, compared to the control group, regardless of the period assessed (P<.05). The same condition can be observed in the comparison between the antioxidant solutions of α-tocopherol and ascorbic acid, in which the first one presented higher peroxide neutralization values for the experimental times of 15 min, 1 h, and 4 h (P<.05). For the periods of 30 min and 2 h, there was no significant difference between the antioxidant solutions for the bleached group (P>.05) (Table 5).

Comparing the periods for the unbleached groups, no significant difference was observed in the peroxide neutralization values for the control group (P=.4404) and ascorbic acid (P=.1401). However, for the α-tocopherol group, the values for periods of 30 min, 1 h, and 2 h were higher than those at 15 min and 4 h (P<.0001) (Table 5). For the bleached groups, the control, ascorbic acid, and α-tocopherol groups under the experimental time of 4 h presented higher hydrogen peroxide concentration values in relation to the other periods analyzed (P<.0001).

Comparing the bleaching procedure, it can be noted that the bleached groups promoted higher hydrogen peroxide concentration values compared to the unbleached groups under the same experimental conditions, regardless of the period and experimental groups investigated (P<.0001) (Table 5).

Contact Angle

The contact angle values are listed in Table 6. Comparing the solutions, α-tocopherol antioxidant solution showed higher wettability values compared to distilled water (control) and ascorbic acid solutions before and after enamel bleaching treatment (P<.05) (Table 6). Assessing the bleaching treatment period, there was no significant difference in the contact angle values for distilled water solution (P=.1728). However, the bleaching treatment period was significantly influenced by ascorbic acid and α-tocopherol antioxidant agents (P<.05). For the ascorbic acid solution, the post-bleaching procedure yielded higher contact angle values compared to the pre-bleaching period (P=.0013), in contrast to the α-tocopherol antioxidant agent, for which the post-bleaching procedure promoted lower contact angle values compared to the pre-bleaching period (P=.0247), increasing the solution wettability on the enamel surface (Table 6).

Surface Energy and Total Free Interaction Energy

The surface energy and total free interaction energy values are listed in Table 7. The bleached enamel presented lower surface energy (P=.0045) and total free interaction energy (P=.0032) values compared to the unbleached enamel surface (Table 7).

Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS)

Scanning electron micrographs and energy dispersive X-ray spectra showed qualitative similarity in the enamel morphology and chemical composition of all evaluated groups (Figures 7 and 8).

3.5 Discussion

The bleaching treatment and antioxidant solutions influenced the shear bond strength of luting agents to the enamel surface, adhesive interface morphology, hydrogen peroxide neutralization, and enamel surface properties; thus, the first null hypothesis was rejected. The thermal aging process resulted in differences in the shear bond strength values; thus, the second null hypothesis was rejected. Finally, analysis of the influence of distinct periods on hydrogen peroxide neutralization led to a rejection of the third null hypothesis.

The success of laminate veneer restorations is directly related to the adhesion efficacy between luting agents to dental and ceramic substrate [7]. The most common causes for restoration failures using ceramic laminates are the marginal defects, substrate fractures, and the ineffective hybridization of the dental and ceramic substrate promoting the debonding of the restorative assembly [35]. Micro shear bond strength test allows evaluation of a small dental area using different material substrates, and the preparation of multiples specimens from the same tooth without involving a section or trim procedure that could induce early microcracking or some interference in the bonding interface [36]. In addition, thermocycling is a universally effective method for simulating the aging of dental materials evaluating the longevity by thermal stress [37,38]. Thus, these methodologies were adopted in the present study to assess the influence of antioxidant agents post dental bleaching on the ceramic laminate veneers luting.

The thermal aging process promoted significant differences in shear bond strength for all experimental groups, except for the AA_{24H}, AA_{14D}, and αT_{14D} groups (Table 3). The thermal

stress caused by cycle oscillation in water between 5 and 55°C weakens the bonding interface between the filler content and the resinous matrix [38]. According Strazzi-Sahyon et al., [6] the water sorption into resinous compounds directly affects the clinical efficacy of dental materials, promoting irreversible damages, such as micro-cracks, fissures, and hydrolytic degradation of the chemical components of the resinous matrix [6]. The non-cured monomers could be released due to their higher solubility in aqueous medium, resulting in porosities in the resin matrix, yielding lower mechanical properties, shear bond strength, and higher occurrence of fractures, debonding, and color alteration of the restorative assembly [5,6]. This could justify the increase in adhesive failures of the experimental groups compared to the post-aging period to the pre-cycling (Figures 3 and 4).

The luting of ceramic laminates onto the bleached and no antioxidant-treated enamel after 24 hours (BLE_{24H} group) promoted significantly lower shear bond strength compared to the αT_{24H} and $BLE_{\alpha T24H}$ groups, regardless of the aging period evaluated (Table 3). The release of oxygen bubbles and residual hydrogen peroxide imprisoned in the enamel structure obstructed the infiltration of the resinous monomers into the interprismatic area [39]. Moreover, the inhibition of monomer polymerization occurs once the free radicals from hydrogen peroxide interfere with the propagation of vinyl free radicals, promoting immature polymer chain termination and ineffective polymerization [39]. Lower conversion of monomers into polymers provides degradation of the resin matrix, decrease in mechanical properties, poor wear resistance under stomatognathic system action, and marginal gap formation [40]. These factors could have corroborated the lower shear bond strength of the BLE_{24H} group (Table 3).

All the experimental groups presented a higher incidence of mixed failures regardless of the aging period (Figure 3 and 4). The mixed failure type is characterized by the presence of residual luting agents on the dental surface after the shear strength test, meaning that the hybridization and bonding process was effective between the luting materials and the tooth surface (Figure 5) [41]. This performance may be correlated to the composition of the luting agents. Variolink Esthetic LC contains the hydrophobic monomer urethane dimethacrylate (UDMA) in its composition (Table1) that allows less hygroscopy in the resinous matrix [5]. Moreover, Tetric N Bond Universal adhesive system includes the methacrylated carboxylic acid polymer (MCAP) and decandiol dimethadrylate (D3MA) (Table 1). The MCAP monomer reacts and bonds with the hydroxyapatite components, and the monomer bonding efficacy to the dental substrate is proportional to the concentration of carboxylic acid groups

along the polymeric chain [42,43]. The D3MA monomer reacts with less polar components present in the resin cement, promoting a strong and well-structured interface [42,43]. In addition, it is characterized as a hydrophobic monomer that minimizes water sorption into the resin matrix, enhancing the integrity of the adhesive interface [42,43]. The Tetric N Bond Universal adhesive system included water and ethanol as solvents in its formulation (Table 1). The hydrophilic compounds incorporated in the resin cement could absorb the water from the adhesive system, promoting the hydrolytic degradation of the resin interface [6]. In this sense, the prior photoactivation of the adhesive system on the enamel surface was performed, once the gradual temperature increase of the curing light could provide advantages regarding solvent volatilization [7]. Strazzi-Sahyon et al. [6] reported that the prior photoactivation of the adhesive system on the enamel substrate yields more satisfactory mechanical properties regarding the bonding components of the adhesive interface and better color stability of the ceramic laminate restorations [6]. All these aspects could influence the integrity of the bonding interface, promoting a higher incidence of mixed failures in the present study (Figures 3 – 5).

The micromechanical interactions of the bonding interface were assessed using confocal laser scanning microscopy. This method allows the investigation of the micropermeability and sealing capacity of the resinous tags on the tooth substrate, offering qualitative details of the components that constitute the bonding interface, with less artifact formation compared with scanning electron microscopy [27]. According to the confocal micrographs and scores of the confocal analysis, there was no difference in the quality of the adhesive interface between enamel and resin cement for all the experimental groups (Table 4; Figure 6). However, regarding the parameter of tag formation, the BLE_{24H} group showed significant differences compared to the control and $BLE_{\alpha T24H}$ groups (Table 4). As described previously, the oxygen bubbles and residual peroxide interfered with the resin monomer infiltration [39], promoting formation of a few short tags (Table 4; Figure 6 – F). This performance corroborates the lower bond strength values shown by the experimental group (Table 3).

Upon evaluation of the efficacy of antioxidant agents, it was seen that, in general, α-tocopherol provided more satisfactory results compared to ascorbic acid in terms of shear bond strength and adhesive interface morphology (Tables 3 and 4). Ascorbic acid is characterized as a hydrophilic component with high solubility in aqueous medium. According to Naidu et al., [44] the effectiveness of ascorbic acid is influenced by the water and oxygen

concentration, as well as the pH of the substrate [22,44]. It can be hypothesized that the hydrophilic properties of antioxidants promote water absorption as a sub product generated from the bleaching process, neutralizing the ascorbic acid efficacy.

The use of adhesive systems, including organic solvents, such as acetone and ethanol, has been suggested after dental bleaching [45], as the application of these components to bleached tooth surfaces can decrease the effect of hydrogen peroxide on the shear bond strength of resin materials [22,45-47]. Such solvents allow the infiltration of resinous monomers easily into the hydroxyapatite crystals due to their water chaser property, displacing the water from the tooth surface [48]. Moreover, α-tocopherol is considered a hydrophobic compound [49] that prevents the hygroscopy of the resin matrix. The presence of α-tocopherol associated with ethanol and its hydrophobicity may have corroborated the satisfactory performance of this antioxidant agent on shear bond strength (Table 3; Figure 5 – S and T) and the integrity of the adhesive interface morphology (Table 4; Figure 6 – J).

The methodology used to evaluate the antioxidants´ effectiveness on neutralizing the hydrogen peroxide is extremely sensitive, and can detect low peroxide concentrations [18,29-31]. The hydrogen peroxide reacts with leucocrystal violet, catalyzed by peroxidase, changing the color of the solution according to the peroxide concentration [30]. Thus, since the signal of the absorbance curve is directly proportional to the hydrogen peroxide concentration, it is possible to verify the efficacy of the antioxidant agents in neutralizing the hydrogen peroxide [30]. Vitamin E is formed by tocopherols and tocotrienols components containing the structures of α, β, γ, and δ for both compounds [50]. The α-tocopherol is shown to be the most effective of all tocopherols due to its broad spectrum of action, reacting with various reactive oxidants, such as alkoxyl radicals, singlet oxygen, nitrogen dioxide, peroxinitrite, ozone, and superoxide [50,51]. In contrast to other antioxidants types, vitamin E efficacy is enhanced by it regeneration from its oxidized products [50]. In other words, α-tocopherol can prevent free radicals by donating one of its electrons to the free radical; however, after the donation, the antioxidant does not become a new free radical, and remains stable, regenerating its function [17,50]. All these characteristics could corroborate with more satisfactory performance of the α-tocopherol antioxidant agent in neutralizing the hydrogen peroxide (Table 5) yielding higher shear bond strength values (Table 3) and a better adhesive interface morphology (Tale 4) comparing to the ascorbic acid.

According to Cakir et al., [34] the mineral concentration in the tooth substrate is a good indication of the demineralization and remineralization process. The use of scanning electron microscopy and energy dispersive X-ray spectroscopy analyses allows the investigation of the accurate mineral content by electron bombardment using a high-voltage beam, providing wavelength emission for each mineral [34]. In the present study, SEM and EDS analyses revealed no qualitative difference in the surface morphology and mineral contents of the enamel treated with antioxidant solutions after dental bleaching (Figures 7 and 8). These findings are in agreement with those of previous studies that evaluated the enamel topography and mineral concentration after bleaching treatment [52,53], but contrary to others that observed changes in enamel surface and mineral content [34,54]. The pH of the bleaching and antioxidant agents, around 6.5, and 7.0, respectively, could explain why no changes in enamel surface and mineral content were observed. These pHs are considered practically neutral, with no capacity for surface conditioning and substrate modification [52]. The controversial results in the literature may be justified by distinct bleaching protocols with different numbers of sessions, times of agent action, and diverse bleaching types having different concentrations and pH [52].

Despite no changes in the enamel morphology and mineral content, the bleaching treatment was able to modify the surface energy and total free interaction energy of the enamel substrate (Table 7). Bleaching treatment generates radical species that are able to modify the dental surface [34]. In the present study, the hydrogen peroxide decreased the surface energy and total free interaction energy, increasing the enamel hydrophobicity due to the interaction of the hydrocarbon to the bleaching agent (Table 7). As previously reported, ascorbic acid is characterized as a hydrophilic component and a polar molecule presenting four hydroxyl groups (OH) that promote higher solubility in water. However, the α-tocopherol antioxidant is described as a hydrophobic compound and an apolar molecule presenting only one hydroxyl group, making solubility in hydrophilic media, such as water, difficult [49]. The characteristics of the enamel surface associated with the properties of the antioxidant could justify the higher wettability of the α-tocopherol solution on the enamel surface (Table 6).

Antioxidant solutions are indicated to assuage the effects of hydrogen peroxide after dental bleaching. However, several studies claim that they cannot reverse it completely [15,55,56]. In the present study, it can be noted that 10% α-tocopherol antioxidant agent was able to completely reverse the adverse effects of hydrogen peroxide on shear bond strength, yielding satisfactory integrity of the adhesive interface morphology, as the use of this solution

after 24 hours of the bleaching treatment yielded similar performance to the control group. Thus, the use of 10% α-tocopherol antioxidant agent mediately post enamel bleaching is an effective and promising alternative approach to ceramic laminate veneers luting.

The evaluation of only one concentration of bleaching agent and antioxidant solutions, as well as the use of one resin cement and adhesive system, could be considered as a limitation of the present study. Furthermore, the applicability of laboratory data to clinical conditions must be extrapolated with caution, as this *in vitro* study was not able to replicate oral conditions, such as the interference of enzymes, microorganisms, temperature, and occlusal loads. Further investigations are required to assess the influence of antioxidants on collagen fibers, permeability, marginal adaptation, and other physical-mechanical properties aimed at improving the luting protocols and clinical longevity of ceramic laminates.

3.6 Conclusion

Based on the methodology and findings of this *in vitro* study, the following conclusions were drawn:

1. Shear bond strength, adhesive interface morphology, hydrogen peroxide neutralization, wettability, and enamel surface characteristics such as surface energy and total free interaction energy, were influenced by bleaching treatment and antioxidant agents.

2. Bleaching treatment and antioxidant agents did not qualitatively alter the morphology and chemical composition of the enamel substrate.

3. α-Tocopherol is a suitable antioxidant agent to be applied after enamel bleaching, as it reverses the deleterious effect of bleaching on shear bond strength, promoting an intact adhesive interface, and is effective in neutralizing hydrogen peroxide, yielding satisfactory enamel surface properties.

4. Shear bond strength and hydrogen peroxide neutralization were influenced by the evaluation time.

3.7 References

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Table 1 Investigated materials information regarding their composition and batch number.

Material	Composition	Batch
Tetric N Bond Universal Adhesive System (Ivoclar Vivadent)	HEMA, 10-MDP, Bis-GMA, MCAP, D3MA, ethanol, water, highly dispersed silicon dioxide, photoinitiator.	X25012
Variolink Esthetic LC Light-Cured Resin Cement (Ivoclar Vivadent)	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, boroaluminofluorosilicate glass, spheroidal mixed oxide, benzoylperoxide, stabilizers, pigments	X43425
Monobond N Silane Agent (Ivoclar Vivadent)	Ethanol, 3-trimethoxysilylpropyl methacrylate, 10-MDP, disulfide acrylate	X17917
Whiteness HP Maxx 35% Bleaching Agent (FGM)	Thickeners, dye mixture, glycol, inorganic filler, deionized water, 35% hydrogen peroxide.	031218
Ascorbid Acid Solution Vitamin C (Apothicário)	Ascorbic acid at 10% and 2.5% alcohol, adjustment of pH=7.0 with triethanolamine.	_
α – Tocoferol Solution Vitamin E (Apothicário)	α-tocopherol at 10% and 2.5% alcohol, adjustment of pH=7.0 with triethanolamine.	_

Abbreviations: HEMA, 2-hydroxyethylmethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-A glycidyl methacrylate; MCAP, methacrylated carboxylic acid polymer; D3MA, decandiol dimethadrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.

Table 2 Abbreviation and treatment details of the experimental groups.

Groups Abbreviation	Group Treatment Detail
Control	Unbleached and no antioxidant-treated enamel luted with ceramic laminate veneers
AA_{24H}	Unbleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action
AA_{14D}	Unbleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action
αT_{24H}	Unbleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action
αT_{14D}	Unbleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action
BLE_{24H}	Bleached and no antioxidant-treated enamel luted with ceramic laminate veneers after 24 hours of the bleaching procedure
BLE_{14D}	Bleached and no antioxidant-treated enamel luted with ceramic laminate veneers after 14 days of the bleaching procedure
BLE_{AA24H}	Bleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action
BLE_{AA14D}	Bleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action
$BLE_{\alpha T24H}$	Bleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action
$BLE_{\alpha T14D}$	Bleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action

Table 3 Mean ± standard deviation values of shear bond strength (MPa) of resin cement to enamel surface as a function of the experimental groups and cycling periods.

	Pre-Cycling	**Post-Cycling**
Control	8.36 ± 5.82 ABC a	4.56 ± 3.29 AB b
AA_{24H}	4.53 ± 1.17 BC a	3.48 ± 1.72 AB a
AA_{14D}	8.67 ± 4.24 ABC a	4.12 ± 1.38 AB a
αT_{24H}	11.42 ± 3.46 A a	5.24 ± 1.58 A b
αT_{14D}	10.14 ± 5.31 AB a	5.09 ± 2.32 AB a
BLE_{24H}	3.29 ± 0.82 C a	1.33 ± 0.44 B b
BLE_{14D}	4.75 ± 1.43 BC a	2.13 ± 0.22 AB b
BLE_{AA24H}	8.77 ± 2.60 ABC a	5.39 ± 2.15 A b
BLE_{AA14D}	6.90 ± 2.16 ABC a	4.53 ± 1.64 AB b
$BLE_{\alpha T24H}$	11.44 ± 1.24 A a	5.51 ± 1.60 A b
$BLE_{\alpha T14D}$	8.29 ± 1.16 ABC a	4.13 ± 2.95 AB b

Different letters, uppercase in column and lowercase in row, indicate statistically significant differences ($P<.05$).

Table 4 Scores of the adhesive interface morphology as a function of the experimental groups.

Parameters	Adhesive Interface Quality				Tags Formation in Enamel					Tags Penetration Depth			
Scores / Groups	0	1	2		0	1	2	3		0	1	2	
Control	9	-	-	A	-	2	7	-	A	-	8	1	A
AA_{24H}	9	-	-	A	-	5	4	-	AB	-	9	-	A
AA_{14D}	9	-	-	A	-	7	2	-	AB	-	9	-	A
αT_{24H}	9	-	-	A	2	3	4	-	AB	1	8	-	A
αT_{14D}	9	-	-	A	1	4	4	-	AB	1	8	-	A
BLE_{24H}	7	2	-	A	5	4	-	-	B	5	4	-	A
BLE_{14D}	7	2	-	A	3	6	-	-	AB	3	6	-	A
BLE_{AA24H}	9	-	-	A	1	7	1	-	AB	1	8	-	A
BLE_{AA14D}	9	-	-	A	1	7	1	-	AB	1	8	-	A
$BLE_{\alpha T24H}$	9	-	-	A	-	2	7	-	A	-	8	1	A
$BLE_{\alpha T14D}$	9	-	-	A	2	6	1	-	AB	2	7	-	A

Different letters in column indicate statistically significant differences for each adhesive interface parameter evaluated ($P<.05$).

Table 5 Mean ± standard deviation values of hydrogen peroxide neutralization as a function of the evaluation time, antioxidant solutions, and bleaching procedure.

	Control	**Ascorbic Acid**	**Alfa-Tocopherol**
Unbleached			
15 minutes	(-) 0.012 ± 0.227 Aa^{B}	(-) 0.053 ± 0.038 Aa^{B}	(-) 0.101 ± 0.030 Ca^{B}
30 minutes	(-) 0.051 ± 0.048 Ab^{B}	(-) 0.107 ± 0.068 Ab^{B}	0.052 ± 0.037 Aa^{B}
1 hour	0.088 ± 0.018 Aa^{B}	0.060 ± 0.059 Aa^{B}	0.057 ± 0.043 Aa^{B}
2 hours	0.038 ± 0.071 Aa^{B}	0.016 ± 0.058 Aa^{B}	0.022 ± 0.021 Aa^{B}
4 hours	(-) 0.015 ± 0.109 Aa^{B}	0.007 ± 0.192 Aa^{B}	(-) 0.037 ± 0.035 Ba^{B}
Bleached			
15 minutes	3.201 ± 0.129 Ba^{A}	3.069 ± 0.214 Ba^{A}	2.600 ± 0.187 Bb^{A}
30 minutes	2.977 ± 0.173 Ba^{A}	2.420 ± 0.160 Db^{A}	2.177 ± 0.087 Cb^{A}
1 hour	3.155 ± 0.308 Ba^{A}	2.720 ± 0.022 Cb^{A}	2.255 ± 0.126 Cc^{A}
2 hours	3.127 ± 0.185 Ba^{A}	2.817 ± 0.149 $BCab^{A}$	2.634 ± 0.271 Bb^{A}
4 hours	4.045 ± 0.174 Aa^{A}	3.784 ± 0.251 Aa^{A}	3.403 ± 0.091 Ab^{A}

Different letters (uppercase in column among experimental times, lowercase in row, and superscript uppercase for comparison between the bleaching treatment for the same experimental time indicate statistically significant differences ($P<.05$).

Table 6 Mean ± standard deviation values of contact angle (º) of tooth enamel surface as a function of the antioxidant solutions and bleaching procedure.

	Control	**Ascorbic Acid**	**Alfa-Tocopherol**
Before Bleaching	63.77 ± 9.47 A a	56.25 ± 9.37 B a	21.84 ± 4.10 A b
After Bleaching	71.64 ± 11.39 A a	74.33 ± 5.04 A a	16.23 ± 3.81 B b

Different letters, uppercase in column and lowercase in row, indicate statistically significant differences (P<.05).

Table 7 Mean ± standard deviation values of surface energy γs (mN/m) and total free interaction energy - Delta G (mJ/m^2) of bleached and unbleached enamel surface.

	Surface Energy	**Total Free Interaction Energy**
Unbleached Enamel	35.47 ± 0.92 A	- 3.33 ± 1.59 A
Bleached Enamel	28.27 ± 7.71 B	-15.74 ± 12.64 B

Different letters in column indicate statistically significant differences for each surface property analyzed ($P<.05$).

Fig. 1. Flowchart of the experimental design.

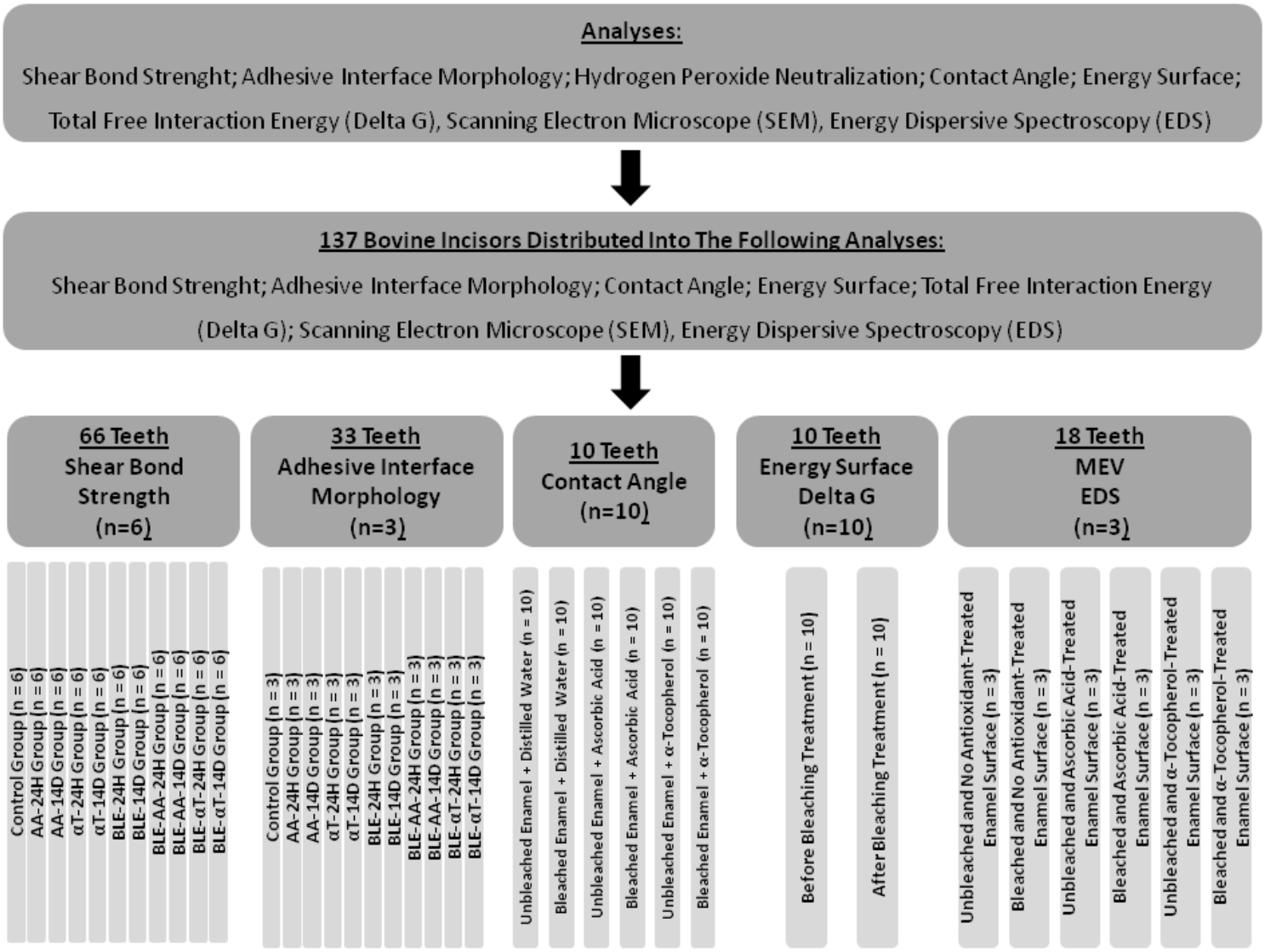


Fig. 2. Specimens treatment and experimental analyses. A, Inclusion of enamel blocks (7 x 8 x 4 mm) in PVC cylinders. B, Tetric N Bond Universal adhesive system was applied on the etched enamel surface and light-cured. C, Accommodation of the silicone mold to prepare the resin cement cylinders usingVariolink Esthetic LC resin cement. Four resin cylinders were destined for the shear strength test pre thermal-cycling, and four cylinders for post aging-cycling. D, Polymerization of the resin cement cylinders using the VALO polywave light curing unit. E, Use of orthodontic wire to apply the load on the resin cylinders assessing the bond strength measurement. F, Enamel specimens were kept in distilled water containing fluorescein during four hours. G, Rhodamine B dye was inserted into de adhesive system and resin cement. H, Specimens were sectioned perpendicularly to adhesive interface. I, Confocal laser scanning microscopy. J, Solution preparation for hydrogen peroxide neutralization analysis. K, Absorbance vs concentration standard curve was carried out using a reflection spectrophotometer to assess the antioxidants effectiveness in neutralizing hydrogen peroxide. L, Distilled water, ascorbic acid, and α-tocopherol were dispensed into specific enamel regions. M, Distilled water, methylene iodide, and ethylene glycol were dispensed to assess the energy surface and total free interaction energy of the bleached and unbleached enamel substrate.

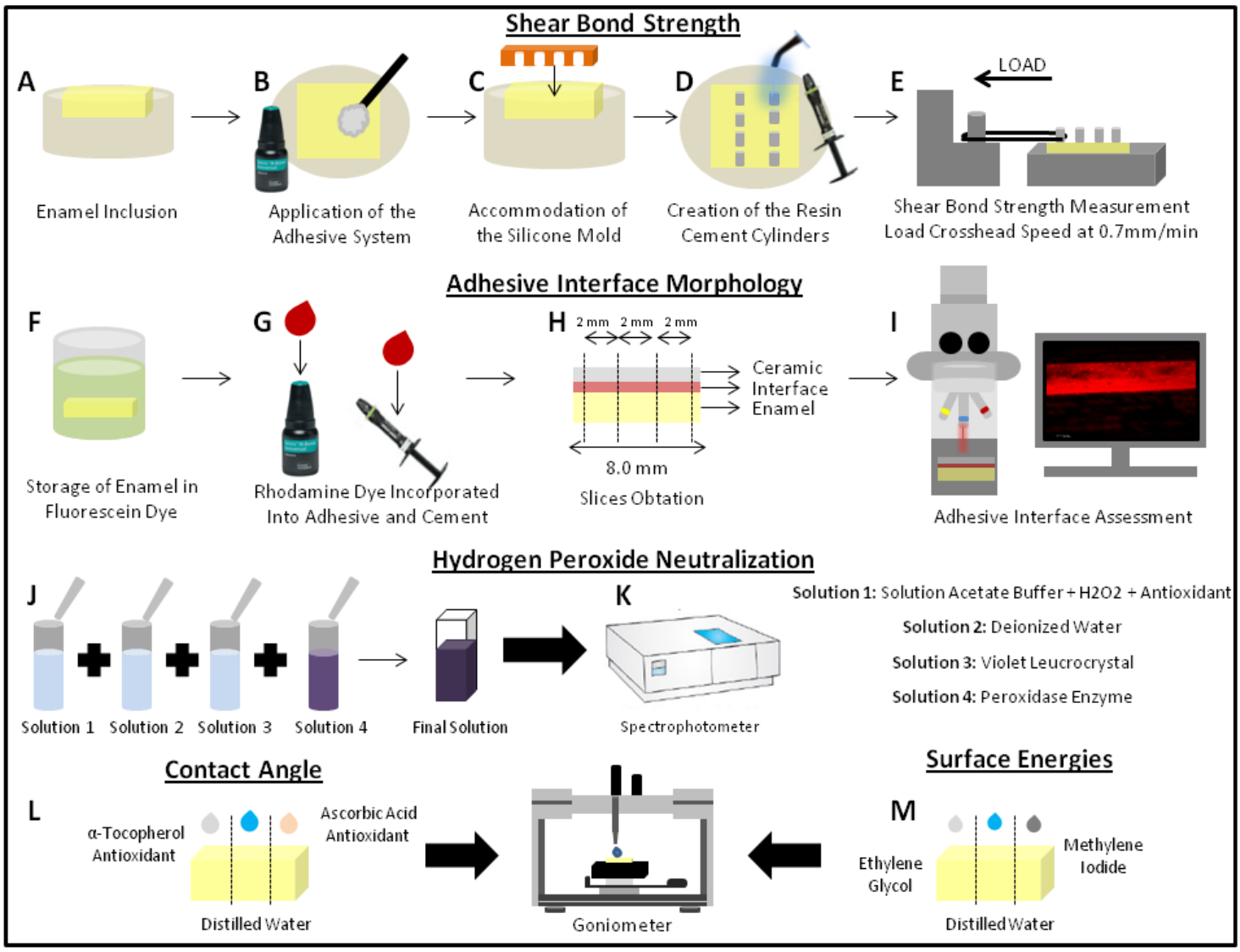

Shear Bond Strength
A
Enamel Inclusion
B
Application of the Adhesive System
C
Accommodation of the Silicone Mold
D
Creation of the Resin Cement Cylinders
E
LOAD
Shear Bond Strength Measurement
Load Crosshead Speed at 0.7mm/min
Adhesive Interface Morphology
F
Storage of Enamel in Fluorescein Dye
G
Rhodamine Dye Incorporated Into Adhesive and Cement
H
2 mm 2 mm 2 mm
Ceramic
Interface
Enamel
8.0 mm
Slices Obtation
I
Adhesive Interface Assessment
Hydrogen Peroxide Neutralization
J
Solution 1
Solution 2
Solution 3
Solution 4
Final Solution
K
Spectrophotometer
Solution 1: Solution Acetate Buffer + H2O2 + Antioxidant
Solution 2: Deionized Water
Solution 3: Violet Leucrocrystal
Solution 4: Peroxidase Enzyme
Contact Angle
L
α-Tocopherol Antioxidant
Ascorbic Acid Antioxidant
Distilled Water
Goniometer
Surface Energies
M
Ethylene Glycol
Methylene Iodide
Distilled Water

Fig. 3. Incidence of fracture patterns (percentage) according to the pre-aging failure type.

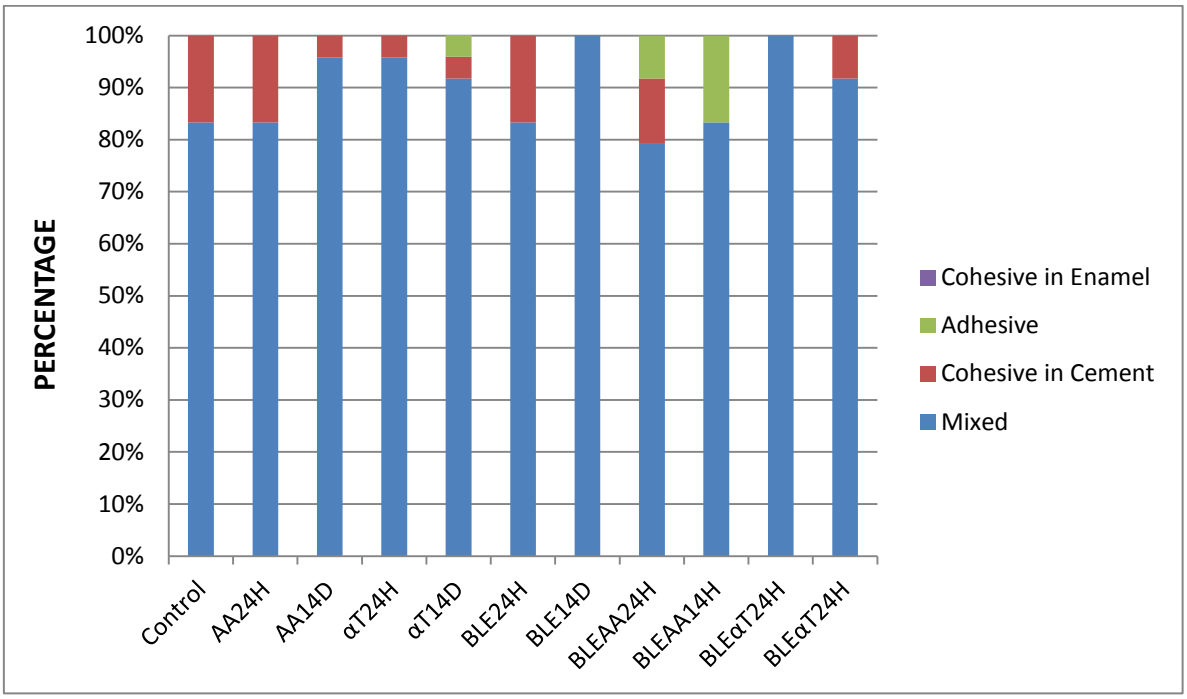


Fig. 4. Incidence of fracture patterns (percentage) according to the post-aging failure type.

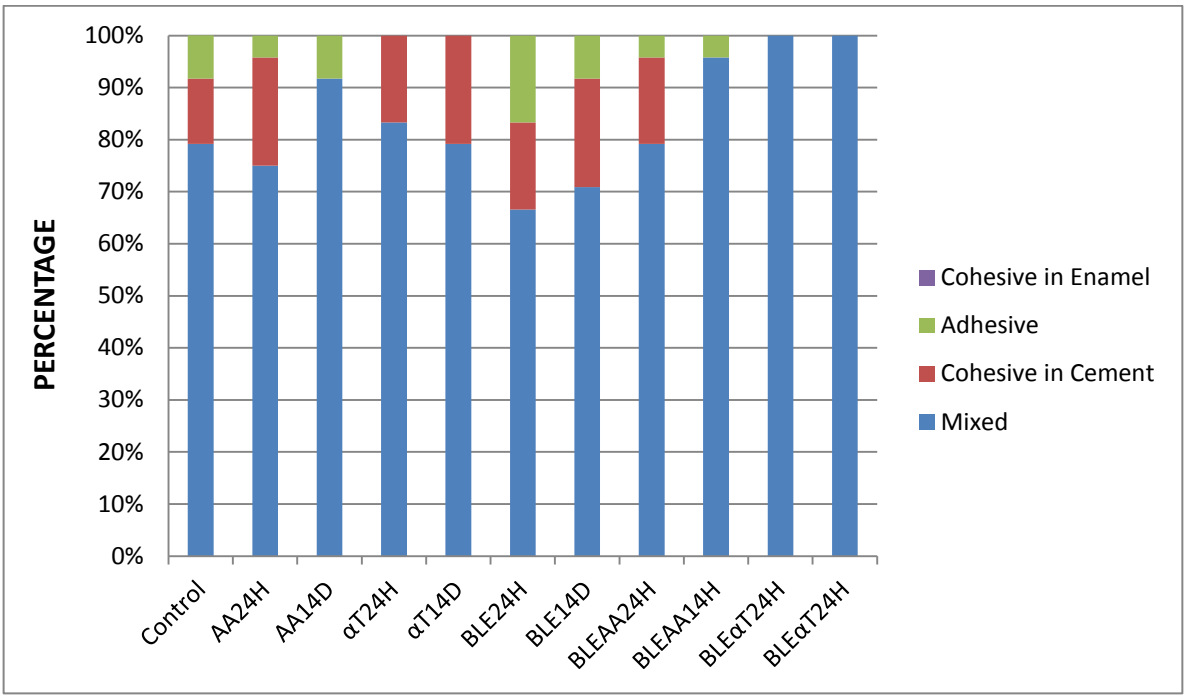
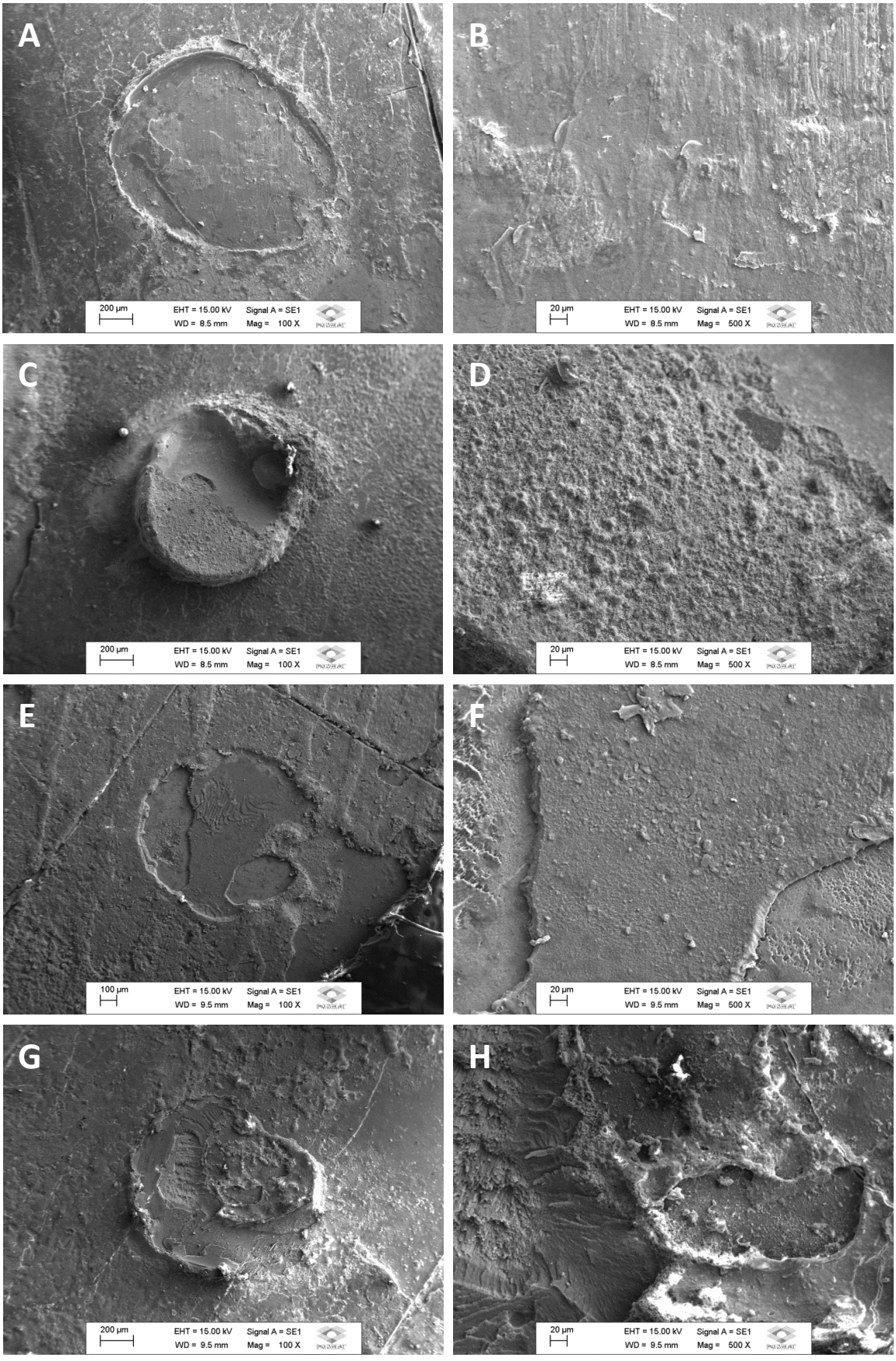


Fig. 5. Scanning electron micrographs of representative failure types according to the experimental groups (original magnification ×100 and ×500). A, B – Mixed failure of unbleached and no antioxidant-treated enamel luted with ceramic laminate veneers (control group - × 100 e × 500). C, D – Mixed failure of unbleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action (AA_{24H} group - × 100 e × 500). E, F – Mixed failure of unbleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action (AA_{14D} group - × 100 e × 500). G, H – Mixed failure of unbleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action (αT_{24H} group - × 100 e × 500). I, J – Mixed failure of unbleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action (αT_{14D} group - × 100 e × 500). K, L – Mixed failure of bleached and no antioxidant-treated enamel luted with ceramic laminate veneers after 24 hours of the bleaching procedure (BLE_{24H} group - × 100 e × 500). M, N – Mixed failure of bleached and no antioxidant-treated enamel luted with ceramic laminate veneers after 14 days of the bleaching procedure (BLE_{14D} group - × 100 e × 500). O, P – Mixed failure of bleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action (BLE_{AA24H} group - × 100 e × 500). Q, R – Mixed failure of bleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action (BLE_{AA14D} group - × 100 e × 500). S, T – Mixed failure of bleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action ($BLE_{\alpha T24H}$ group - × 100 e × 500). U, V - Mixed failure of bleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action ($BLE_{\alpha T14D}$ group - × 100 e × 500).


A
200 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 100 X
B
20 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 500 X
C
200 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 100 X
D
20 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 500 X
E
100 µm
EHT = 15.00 kV
WD = 9.5 mm
Signal A = SE1
Mag = 100 X
F
20 µm
EHT = 15.00 kV
WD = 9.5 mm
Signal A = SE1
Mag = 500 X
G
200 µm
EHT = 15.00 kV
WD = 9.5 mm
Signal A = SE1
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H
20 µm
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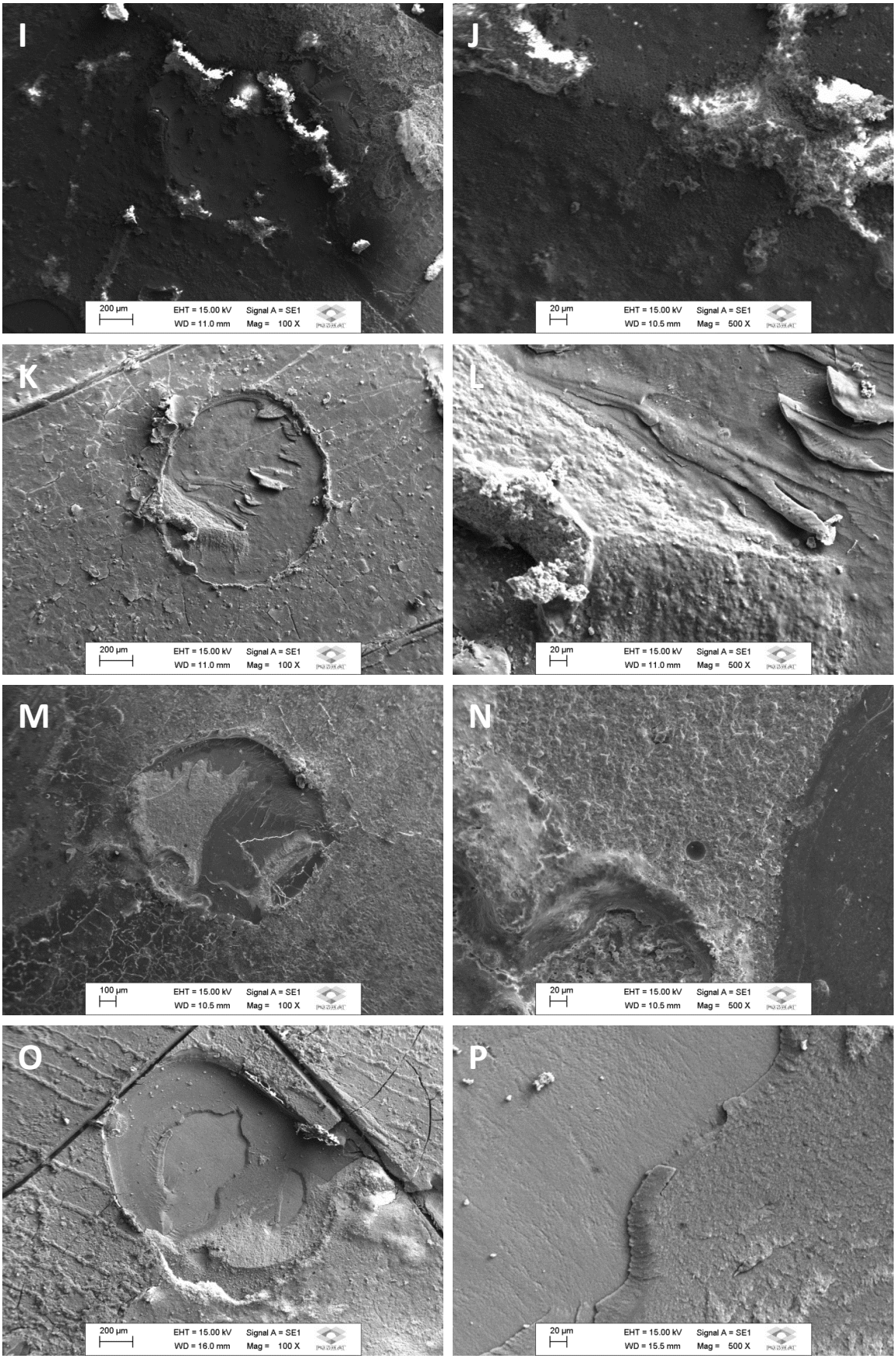
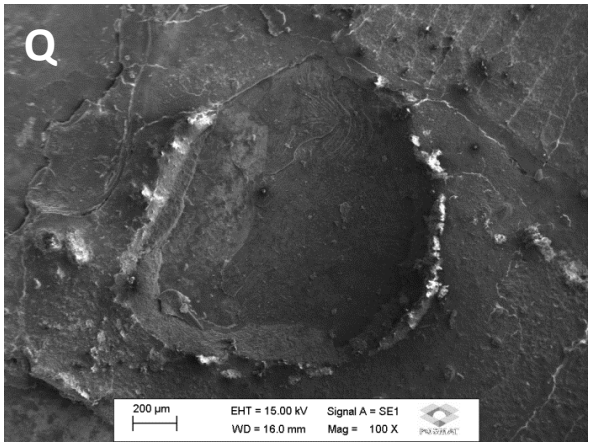
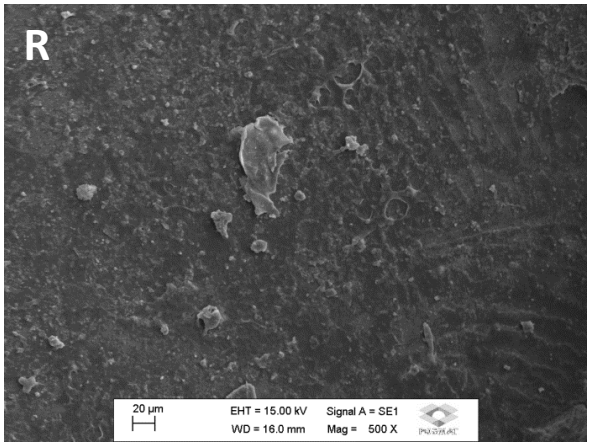
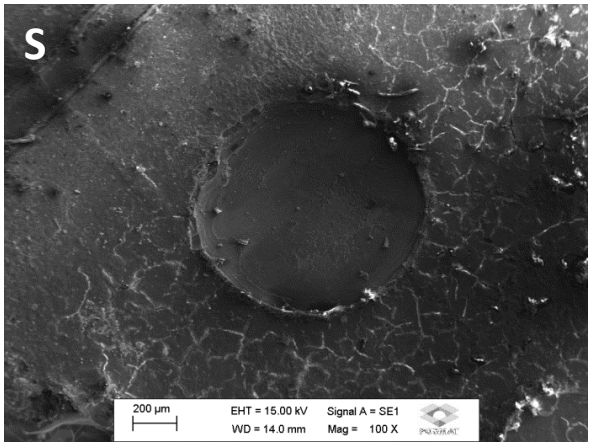
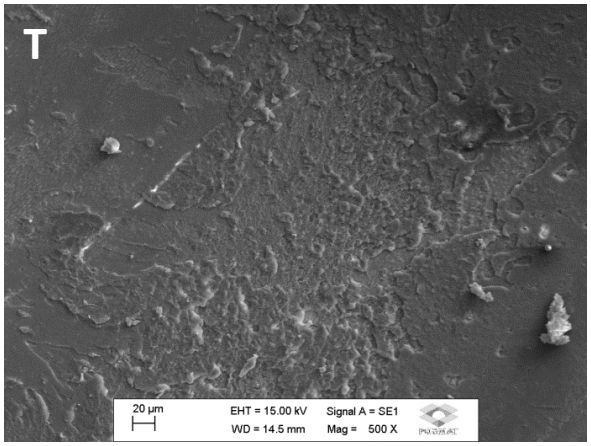
Signal A = SE1
Mag = 500 X

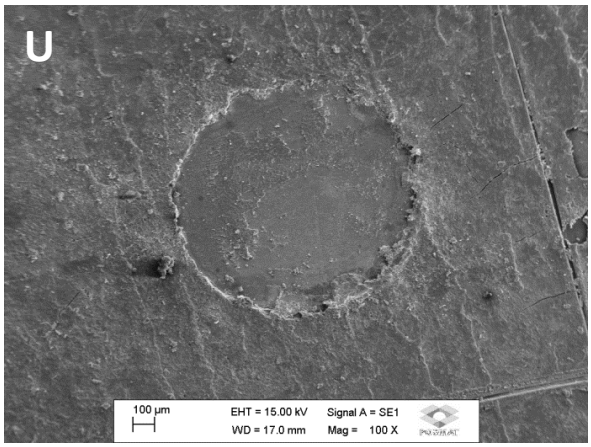

I
200 µm
EHT = 15.00 kV
WD = 11.0 mm
Signal A = SE1
Mag = 100 X
J
20 µm
EHT = 15.00 kV
WD = 10.5 mm
Signal A = SE1
Mag = 500 X
K
200 µm
EHT = 15.00 kV
WD = 11.0 mm
Signal A = SE1
Mag = 100 X
L
20 µm
EHT = 15.00 kV
WD = 11.0 mm
Signal A = SE1
Mag = 500 X
M
100 µm
EHT = 15.00 kV
WD = 10.5 mm
Signal A = SE1
Mag = 100 X
N
20 µm
EHT = 15.00 kV
WD = 10.5 mm
Signal A = SE1
Mag = 500 X
O
200 µm
EHT = 15.00 kV
WD = 16.0 mm
Signal A = SE1
Mag = 100 X
P
20 µm
EHT = 15.00 kV
WD = 15.5 mm
Signal A = SE1
Mag = 500 X


Q
200 µm
EHT = 15.00 kV
WD = 16.0 mm
Signal A = SE1
Mag = 100 X


R
20 µm
EHT = 15.00 kV
WD = 16.0 mm
Signal A = SE1
Mag = 500 X


S
200 µm
EHT = 15.00 kV
WD = 14.0 mm
Signal A = SE1
Mag = 100 X


T
20 µm
EHT = 15.00 kV
WD = 14.5 mm
Signal A = SE1
Mag = 500 X


U
100 µm
EHT = 15.00 kV
WD = 17.0 mm
Signal A = SE1
Mag = 100 X

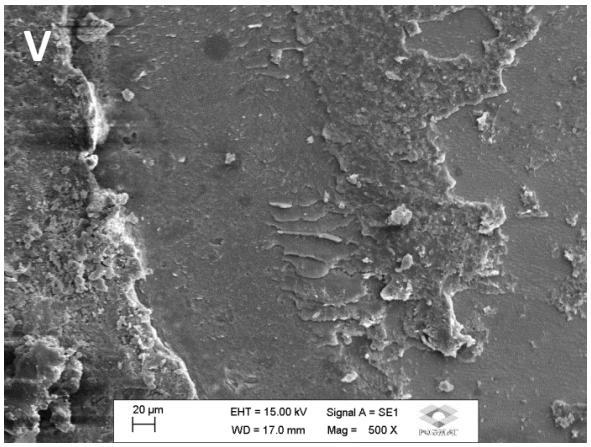
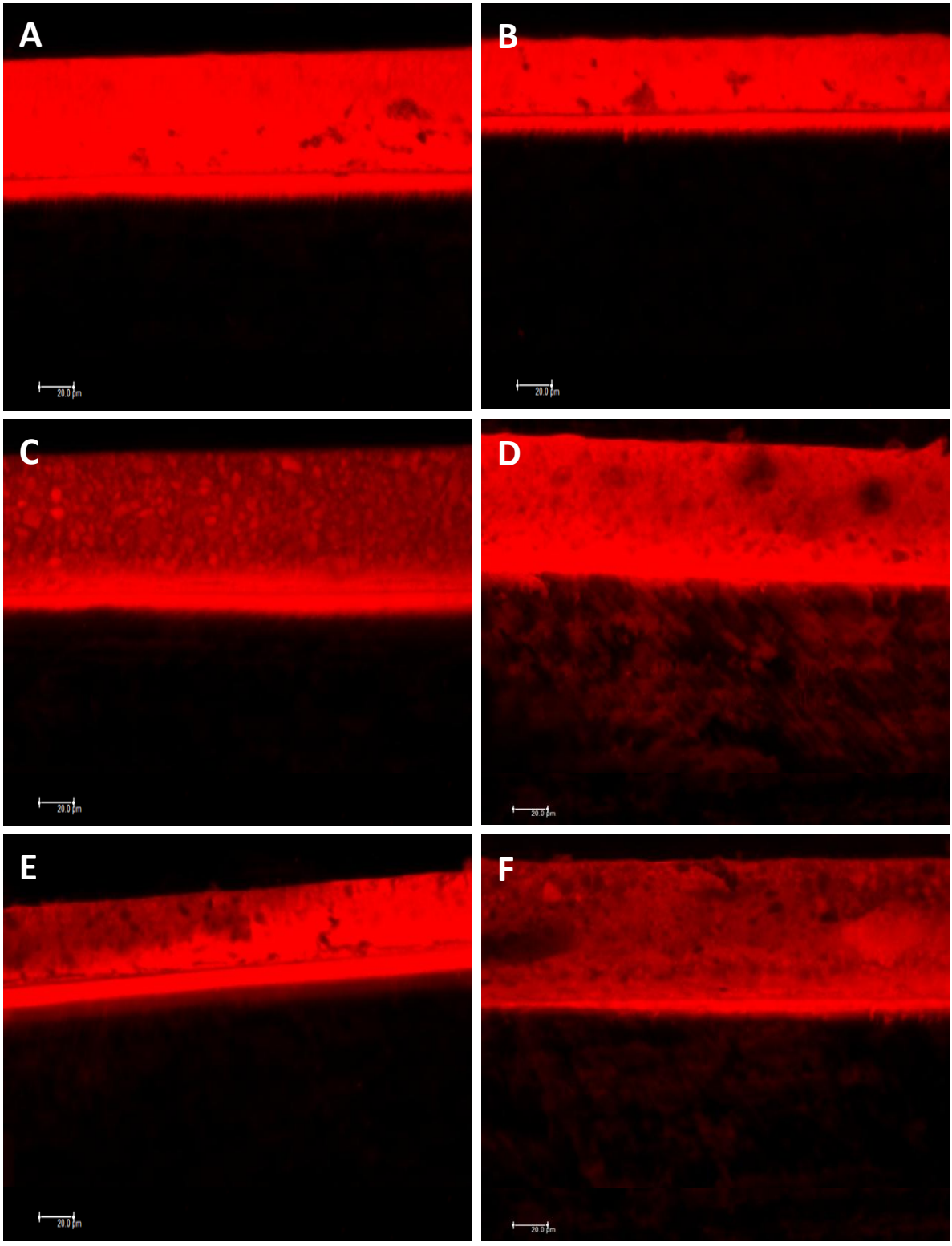

V
20 µm
EHT = 15.00 kV
WD = 17.0 mm
Signal A = SE1
Mag = 500 X

Fig. 6. Confocal laser scanning micrographs of adhesive interface morphology according to the experimental groups. A, Adhesive interface of unbleached and no antioxidant-treated enamel luted with ceramic laminate veneers (control group). B, Adhesive interface of unbleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action (AA_{24H} group). C, Adhesive interface of unbleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action (AA_{14D} group). D, Adhesive interface of unbleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action (αT_{24H} group). E, Adhesive interface of unbleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action (αT_{14D} group). F, Adhesive interface of bleached and no antioxidant-treated enamel luted with ceramic laminate veneers after 24 hours of the bleaching procedure (BLE_{24H} group). G, Adhesive interface of bleached and no antioxidant-treated enamel luted with ceramic laminate veneers after 14 days of the bleaching procedure (BLE_{14D} group). H, Adhesive interface of bleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action (BLE_{AA24H} group). I, Adhesive interface of bleached and 10% ascorbic acid-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action (BLE_{AA14D} group). J, Adhesive interface of bleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 24 hours of the antioxidant action ($BLE_{\alpha T24H}$ group). K, Adhesive interface of bleached and 10% α-tocopherol-treated enamel luted with ceramic laminate veneers after 14 days of the antioxidant action ($BLE_{\alpha T14D}$ group).


A
B
C
D
E
F

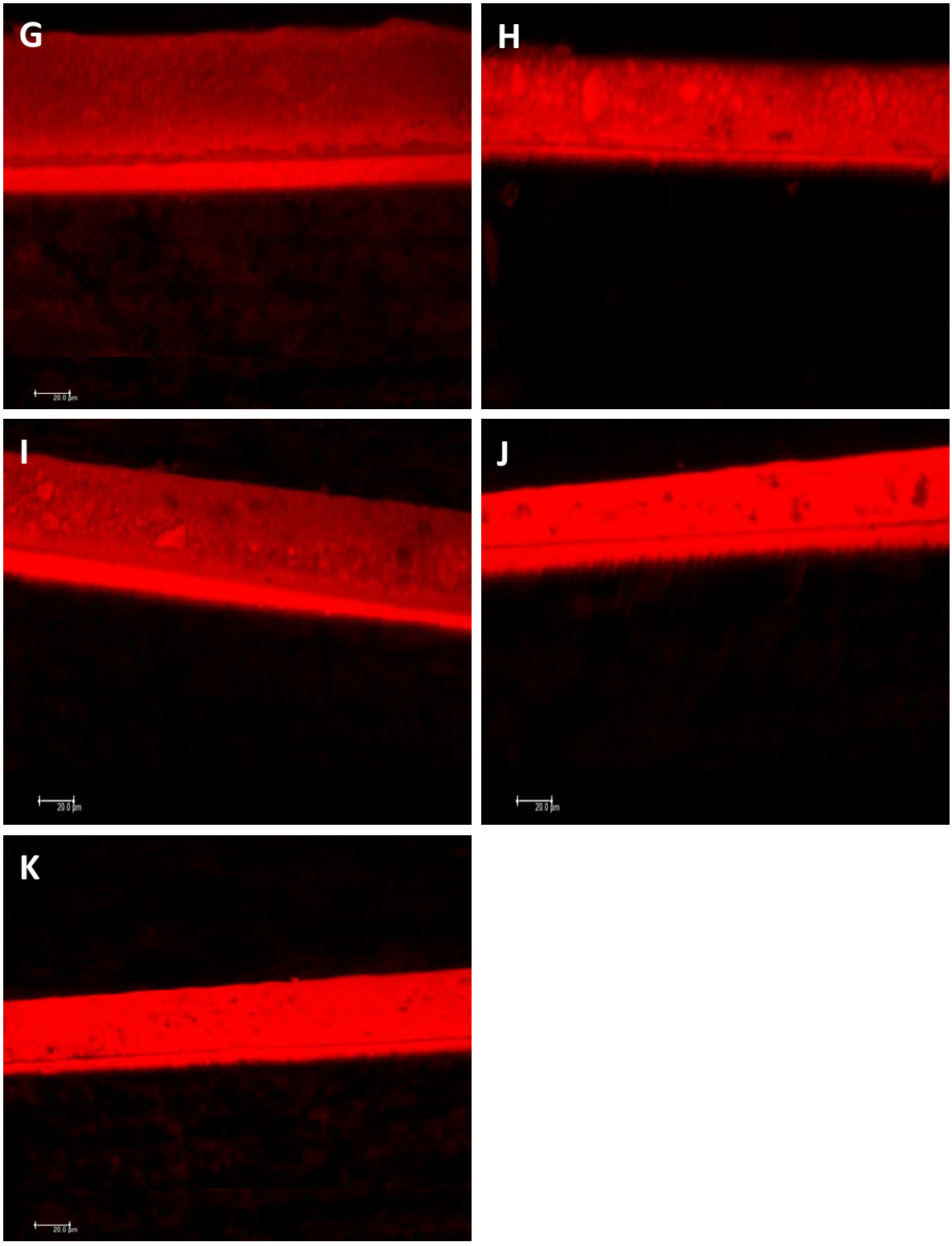
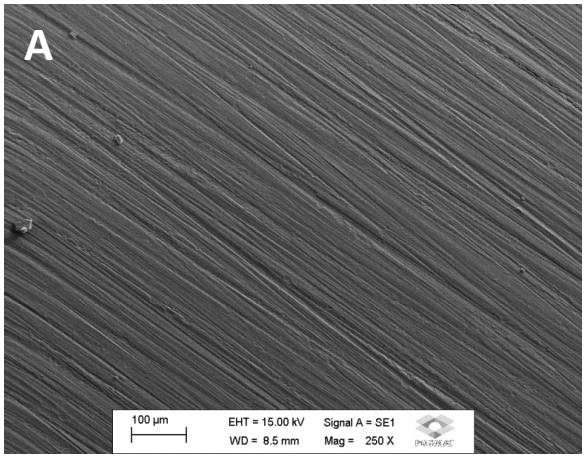
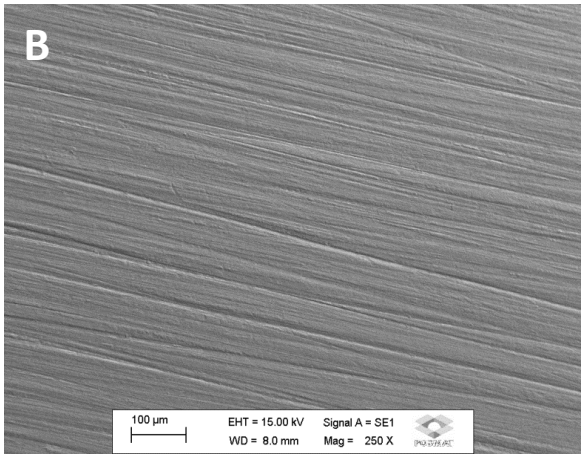
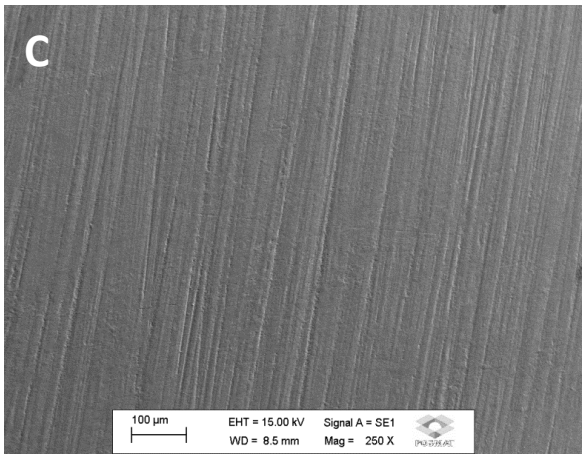
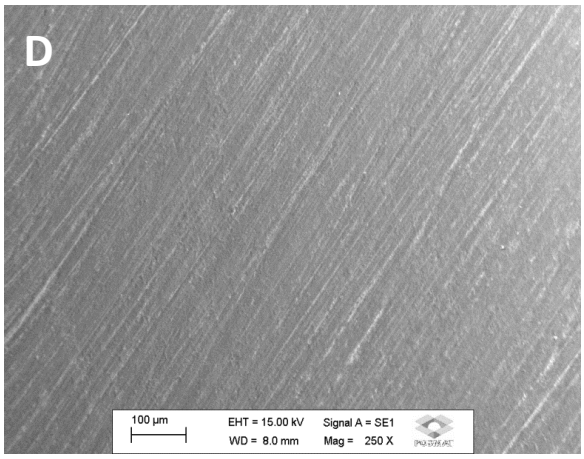

G
H
I
J
K
20.0 µm
20.0 µm
20.0 µm
20.0 µm
20.0 µm

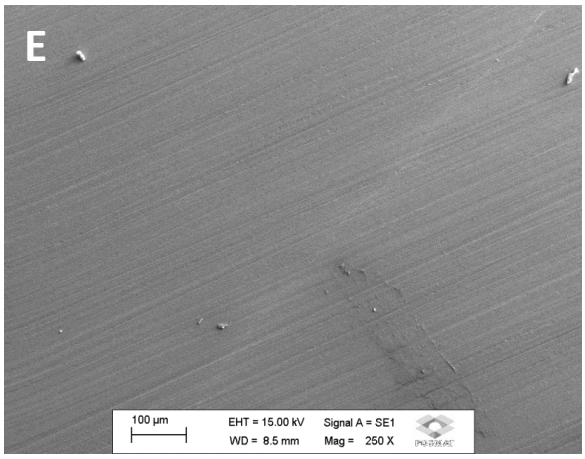
Fig. 7. Scanning electron micrographs of enamel surface according to the bleaching treatment and antioxidant solutions (original magnification ×250). A – unbleached and non-treated enamel with antioxidant solutions. B – bleached and non-treated enamel with antioxidant solutions. C – unbleached and treated enamel with 10% ascorbic acid antioxidant solution. D – bleached and treated enamel with 10% ascorbic acid antioxidant solution. E – unbleached and treated enamel with 10% α-tocopherol antioxidant solution. F – bleached and treated enamel with 10% α-tocopherol antioxidant solution.


A
100 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 250 X


B
100 µm
EHT = 15.00 kV
WD = 8.0 mm
Signal A = SE1
Mag = 250 X


C
100 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 250 X


D
100 µm
EHT = 15.00 kV
WD = 8.0 mm
Signal A = SE1
Mag = 250 X


E
100 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 250 X

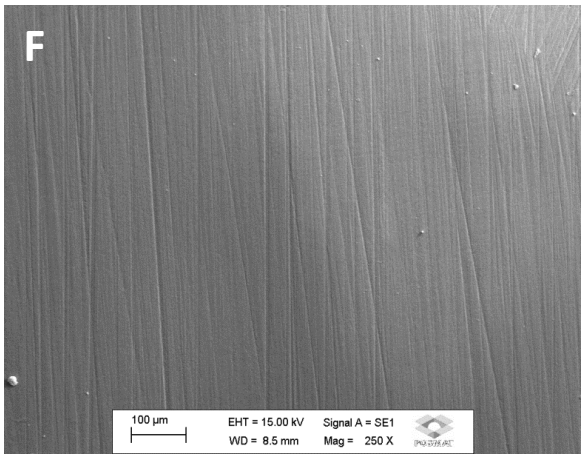
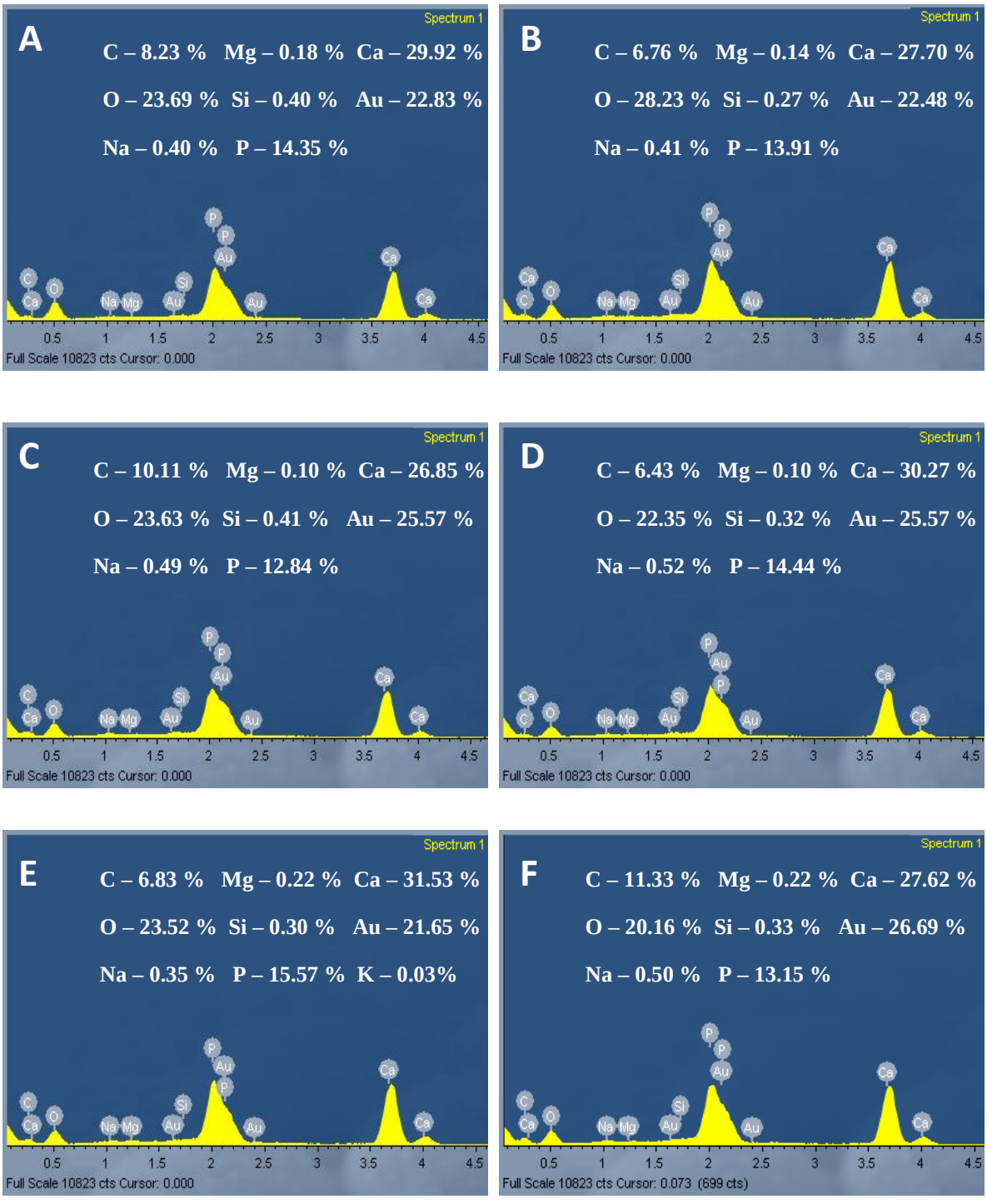

F
100 µm
EHT = 15.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 250 X

Fig. 8. Energy dispersive x-ray spectra of enamel surface according to the bleaching treatment and antioxidant solutions. A – unbleached and non-treated enamel with antioxidant solutions. B – bleached and non-treated enamel with antioxidant solutions. C – unbleached and treated enamel with 10% ascorbic acid antioxidant solution. D – bleached and treated enamel with 10% ascorbic acid antioxidant solution. E – unbleached and treated enamel with 10% α-tocopherol antioxidant solution. F – bleached and treated enamel with 10% α-tocopherol antioxidant solution.



Anexos

ANEXO A – Comprovante de aprovação pela Comissão de Ética no Uso de Animais

UNIVERSIDADE ESTADUAL PAULISTA
"JÚLIO DE MESQUITA FILHO"

CAMPUS ARAÇATUBA
FACULDADE DE ODONTOLOGIA
FACULDADE DE MEDICINA VETERINÁRIA

CEUA - Comissão de Ética no Uso de Animais
CEUA - Ethics Committee on the Use of Animals

CERTIFICADO

Certificamos que o Projeto de Pesquisa intitulado "**Efeito de agentes antioxidantes pós-clareamento na cimentação de laminados cerâmicos**", Processo FOA nº 01058-2017, sob responsabilidade de Paulo Henrique dos Santos apresenta um protocolo experimental de acordo com os Princípios Éticos da Experimentação Animal e sua execução foi aprovada pela CEUA em 12 de Dezembro de 2017.

VALIDADE DESTE CERTIFICADO: 12 de Dezembro de 2021.
DATA DA SUBMISSÃO DO RELATÓRIO FINAL: até 12 de Janeiro de 2022.

CERTIFICATE

We certify that the study entitled "**Effect of antioxidant agents post-bleaching in the luting of ceramic laminates**", Protocol FOA nº 01058-2017, under the supervision of Paulo Henrique dos Santos presents an experimental protocol in accordance with the Ethical Principles of Animal Experimentation and its implementation was approved by CEUA on December 12, 2017.

VALIDITY OF THIS CERTIFICATE: December 12, 2021.
DATE OF SUBMISSION OF THE FINAL REPORT: January 12, 2022.

Prof. Ass. Dr. Leonardo Perez Faverani
Coordenador da CEUA
CEUA Coordinator

CEUA - Comissão de Ética no Uso de Animais
Faculdade de Odontologia de Araçatuba
Faculdade de Medicina Veterinária de Araçatuba
Rua José Bonifácio, 1193 – Vila Mendonça - CEP: 16015-050 – ARAÇATUBA – SP
Fone (18) 3636-3234 Email CEUA: ceua@foa.unesp.br

ANEXO B – Referências da Introdução Geral

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