RELATÓRIO DE ATIVIDADES

1 – DADOS CADASTRAIS

1.1 Nome do Beneficiário **ANDRESSA BORIN VENTURINI**	1.2 CPF / Passaporte **015.822.500-73/FU495028**
1.3 Instituição **UFSM**	1.4 Programa CAPES/ nº do AUXPE **GERAL**
1.5 Projeto **GERAL**	1.6 Coordenador Projeto **PAULO RENATO SCHNEIDER**
1.7 Programa de Pós-Graduação **PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS ODONTOLÓGICAS**	

2 – BENEFÍCIO

2.1 Modalidade (X) Bolsa **CAPACITAÇÃO EM CURSOS DE CURTA DURAÇÃO**		
2.2 Instituição de Destino (nome da instituição e nome do centro/instituto/departamento/grupo de pesquisa) **DEPT. OF DENTAL MATERIALS SCIENCE, ACADEMISCH CENTRUM TANDHEELKUNDE AMSTERDAM, UNIVERSITY OF AMSTERDAM AND VRIJE UNIVERSITY AMSTERDAM (THE NETHERLANDS)**	2.3 Período da Atividade 2.3.1 Início 01/02/2020	2.3.2 Término 15/02/2020

3 – RECURSOS RECEBIDOS (R$)

3.1 Auxílio-deslocamento	PAGAS PELA CAPES
3.2 Auxílio-instalação	€1.300,00
3.3 Seguro-saúde	€90,00
3.4 Adicional-localidade	€400,00
3.5 Mensalidade	€650,00

4 – DESCRIÇÃO DAS ATIVIDADES

4.1 Objetivos da missão:

A presente missão se enquadrou no contexto do projeto Materiais Inteligentes/*Smart Materials*. No geral, o curso de capacitação teve como objetivo o aperfeiçoamento em ensaios de fadiga com base em diferentes metodologias de ensaio, a confecção de espécimes (formato de disco), execução de ensaios em fadiga e posterior discussão dos dados obtidos para a escrita de artigos neste contexto. Ademais, a missão também objetivou o treinamento em análises topográficas dos materiais envolvidos e análises fractográficas para caracterização do padrão de falha observado.

Portanto, as seguintes metas da presente missão podem ser destacadas:

1. Qualificação de recursos humanos pela participação da pós-doutoranda a fim de promover aperfeiçoamento da formação acadêmica, amadurecimento científico e tecnológico na área de materiais dentários, que repercutirá positivamente no contexto do trabalho desenvolvido pelo Programa de Pós-Graduação em Ciências Odontológicas da UFSM;
2. Desenvolvimento e propagação de conhecimento científico obtido a fim de implementar novas metodologias de ensaios e equipamentos nas práticas diárias de atuação na instituição;
3. Fortalecimento da cooperação interinstitucional (Universidade Federal de Santa Maria e Academic Centre for Dentistry Amsterdam), pela aproximação entre pesquisadores e instituições copartícipes.

4.2 Atividades Realizadas:

Todas as atividades previstas no plano de atividades previamente estabelecido foram cumpridas em sua plenitude. Nesse sentido foram realizadas:

- discussões da temática que convergiram principalmente para a submissão de artigos (expostos na seção 4.3 abaixo, e em anexo neste relatório);
- participação como ouvinte em defesa de doutorado, em 14 fevereiro de 2020, intitulada “The protective effect of topical fluoride treatments in dentine lesions”, defendida pela Ms. Marwa Alhothali (Anexo 1);
- participação como ouvinte em conferência de pesquisa promovida pela instituição e denominada “ACTA Research Meeting” na cidade de Lunteren - the Netherlands (programação do evento em anexo neste relatório – Anexo 2);
- treinamento na obtenção de amostras para ensaios (Apêndice);
- ensaios de fadiga com o objetivo de desenvolver uma nova metodologia de ensaio a fim de acelerar a caracterização de materiais em flexão de 3 pontos sob fadiga (Apêndice);
- treinamento em análise fractográfica de espécimes que falharam (Apêndice);
- discussão de novos contextos a serem explorados no futuro, em decorrer do fortalecimento das atividades interinstitucionais.

Diversos tópicos foram discutidos no contexto de desenvolvimento de ensaios de fadiga, na busca de novos métodos e de delineamentos que permitam a aprofundização na temática que têm sido desenvolvida pelos grupos de pesquisa. Ademais, salienta-se que dentre o grande rol de infraestrutura e de conhecimento dos profissionais da ACTA destaca-se o conhecimento na predição de comportamento de materiais restauradores em relação a análises de abrasão, onde enfatiza-se que a instituição possui uma máquina que fora totalmente desenvolvida por estes e validada para predição de performance clínica. Desta forma, foram discutidos aspectos para que pesquisas nesse sentido também possam vir a ser desenvolvidas, e desta forma, prevê-se o fortalecimento dessa linha de pesquisa em um futuro próximo.

4.3 Resultados e/ou Impactos:

Artigos submetidos (Anexo):

1- One-step ceramic primer as surface conditioner: effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations. Kiara Serafini Dapieve, Renan Vaz Machry, Rafaela Oliveira Pilecco, Cornelis Johannes Kleverlaan, Gabriel Kalil Rocha Pereira, Andressa Borin Venturini, Luiz Felipe Valandro. Artigo submetido para o periódico *Journal of the Mechanical Behavior of Biomedical Materials* (Qualis A1; FI 3.485).

A colaboração científica entre instituições é justificada quando existem diferentes competências que podem ser complementadas com o propósito de qualificar o conhecimento científico e seus produtos (artigos científicos, desenvolvimento de novos materiais e metodologias), assim como quando existem anseios mútuos de ampliação de relações que tenham repercussões fundamentalmente na formação de recursos humanos mais qualificados e impacto institucional. Em suma, prospecta-se que a presente missão apresentará impactos relevantes na qualificação de recursos humanos, na produção do conhecimento científico e na consolidação da cooperação entre instituições a fim de fortalecer a internacionalização do Programa de Pós-graduação em Ciências Odontológicas da UFSM. Ademais, a interação e troca de experiências dos grupos de pesquisa têm proporcionado uma contribuição mais consistente para a literatura científica da área. Os docentes-pesquisadores da instituição holandesa apresentam muita experiência na caracterização de materiais, representado pelo histórico de produção intelectual qualificada, fruto de conhecimento acumulado e estrutura física qualificada. Portanto, a interação com esta instituição tem se mostrado profícua e pretende ser ainda mais intensificada no futuro próximo.

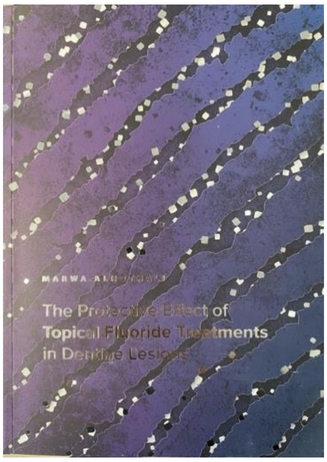
Santa Maria, 04 de março de 2020.

Andressa Borin Venturini

ANEXOS

1. Defesa de doutorado, em 14 fevereiro de 2020, intitulada "The protective effect of topical fluoride treatments in dentine lesions", defendida pela Ms. Marwa Alhothali

Tive a oportunidade de como ouvinte presenciar a defesa de doutorado acima citada. Fotos do evento e da capa do livro componente da tese, encontram-se abaixo.



2. Participação na conferência de pesquisa promovida pela instituição e denominada "ACTA Research Meeting" na cidade de Lunteren - the Netherlands.

PROGRAMME

ACTA research meeting
Hotel & Conference Center 'De Werelt'
Lunteren, the Netherlands, 20 and 21 February 2020

Thursday February 20

11.30 – 12.30	Registration	
12.15 – 13.20	Lunch	
13.20 – 13.30	Welcome	Bruno Loos (director of research ACTA)
13.30 – 15.00	SESSION I	(chair: Frank Lobbezoo)
	Tijmen Münker	Dental Material Sciences
	Teun de Vries	Periodontology
	Amy Pabbla	Social Dentistry
	Lingfei Wei	Implantology
	Magdalini Thymi	Oral Kinesiology
	Ivana Nedeljkovic	Dental Material Sciences
15.00– 15.30	Tea Break	
15.30 – 17.15	SESSION II	(chair: Teun de Vries)
	Erwin Berkhout	Oral Radiology
	Vivian Wu	Oral Cell Biology/Oral Maxillofacial Surger
	Alexa Laheij	Oral Medicine
	Lin Shang	Preventive Dentistry
	Mingjie Wang	Implantology
	Elmira Boloori	Periodontology
17.15 – 18.15	Aperitif & poster session	
18.15 – 19.45	Dinner & Discussion	
19:50 - 20:00	Award Ceremony: 'NTVT Debuutprijs'	
20.00 – 21.00	SESSION III - Guest Lecture prof.dr. A.E. Eiben (Professor of Artificial Intelligence, Vrije Universiteit Amsterdam) From Artificial Intelligence to Artificial Life	
from 21.00	Drinks	

Dia 1

PROGRAMME

Friday February 21

06.30 – 07.15	Early morning 5K run	(coach: Bart Hattink)
07.30 – 8.30	Breakfast and Registration	
08.30 – 10.00	SESSION IV	(chair: Bastiaan Krom)
	Nina Nijland	Periodontology
	Nota Katsiki	Periodontology
	Dandan Ma	Oral Biochemistry
	Denise Duijster	Social Dentistry
	Zainab Assy	Oral Biochemistry
	Astrid Bakker	Oral Cell Biology
10.00 – 10.30	Coffee Break	
10.30 – 12.00	SESSION V	(chair: Sue Gibbs)
	Madeline Kosho	Periodontology
	Ilkay Evren	Oral Pathology
	Jiayi Cheng	Implantology
	Maaike Waasdorp	Oral Cell Biology
	Gang Wu	Implantology
	Bart Hattink	Research Institute
12.00 – 13.30	Lunch	
13.30 – 15.00	SESSION VI	(chair: Astrid Bakker)
	Karin van Nes	Pediatric Dentistry
	Liza van de Rijt	Oral Kinesiology
	Leon Wils	Oral Pathology
	Marije Kaan	Preventive Dentistry
	Patrick Rijkschroeff	Periodontology
	Bastiaan Krom	Preventive Dentistry
15.00 – 15.05	Presentation of award for the best oral presentation of a junior scientist during the Lunteren meeting 2020.	
15.05 – 15.15	Closing Remarks	Bruno Loos (director of research ACTA)

Location:
Hotel & Conference Center 'De Werelt'
Westhofflaan 2, 6741 KH Lunteren

https://dewerelt.nl/contact/routebeschrijving.html

Dia 2

Esse evento fora consolidado na participação como ouvinte em uma discussão no formato de mesa redonda, onde o presidente da sessão alimentava e conduzia a discussão de temas de importante relevância no contexto odontológico, juntamente com pesquisadores de referência na temática, entre os temas destacam-se: disfunção temporo-mandibular, bruxismo, periodontia, implantodontia, ciência dos materiais dentários, radiologia, dentística preventiva (odontopediatria), medicina oral (patologia), biologia celular oral, bioquímica oral e cirurgia maxilofacial. Ademais, o Professor Dr. A.E. Eiben apresentou aspectos da Inteligencia artificial e suas perspectivas de impacto no dia-dia da sociedade.

3. Artigo

Artigo submetido para o periódico *Journal of the Mechanical Behavior of Biomedical Materials* (Qualis A1; FI 3.485).

One-step ceramic primer as surface conditioner: effect on the load-bearing capacity under fatigue of bonded lithium disilicate ceramic simplified restorations

Kiara Serafini Dapieve[a], Renan Vaz Machry[a], Rafaela Oliveira Pilecco[a], Cornelis Johannes Kleverlaan[b], Gabriel Kalil Rocha Pereira[a], Andressa Borin Venturini[a], Luiz Felipe Valandro[a]

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Luiz Felipe Valandro (valandrolf@gmail.com)

Running title: Ceramic primer on the fatigue performance of lithium disilicate.

Abstract
The aim of the study was to evaluate the effect of a ceramic primer and its increased passive application on the fatigue performance of adhesively cemented lithium disilicate simplified restorations. Ceramic discs (Ø= 10mm; thickness= 1.0mm) were submitted to an in-lab simulation of CAD/CAM milling and allocated into 8 groups (n= 15), considering 2 factors: "surface treatment"– PRIMER, only coupling agent application (Monobond N); HF5+PRIMER, 5% hydrofluoric acid and coupling agent; E&P 20s+40s and E&P 20s+5min, ceramic etching/priming (Monobond Etch & Prime, E&P) for 20 s of active application followed by 40 s or 5 min of passive application, respectively; and "aging condition"– baseline, storage for 24 h to 5 days; aged, storage for 90 days + 12,000 thermal cycles. Adhesive cementation (Multilink N) was performed onto epoxy discs (Ø= 10mm; thickness= 2mm), and the cemented assemblies were subjected to step-stress fatigue tests (initial load of 200 N; step-size of 50 N; 10,000 cycles per step; 20 Hz). The results showed that the groups had similar fatigue performance in the baseline condition (except for E&P 20s+5min: 940.0 N; 123,000 cycles > PRIMER: 786.7 N; 92,333 cycles). When aged, the PRIMER group presented the worst fatigue performance (480.8 N; 31,154 cycles) compared to the other groups (810.0–840.0 N; 97,000–103,000 cycles). In addition, only the PRIMER treatment showed unstable fatigue performance (baseline > aged). Therefore, ceramic surface treatment promoting micromechanical interlocking and chemical bonds is mandatory for stable fatigue performance of adhesively cemented lithium disilicate restorations. The one-step ceramic primer/conditioner promoted similar fatigue performance to the 5% hydrofluoric acid + coupling agent, but increased E&P etching time did not improve the fatigue behavior.
Keywords: Computer Aided Design/Computer Aided Machining. Etching. Topographical changes. Mechanical phenomena. Survival Probability. Weibull Analysis.

Highlights
- Etching is required for proper mechanical behavior of lithium disilicate restorations.
- One-step ceramic primer is an alternative to hydrofluoric acid etching + coupling agent.
- Increased etching time of one-step ceramic primer does not improve fatigue behavior.

1. Introduction

Successful adhesion among ceramic, cement and substrate requires a bonding mechanism which associates micro-mechanical interlocking and chemical bonding (Manso et al., 2011; Blatz et al., 2018). In this context, adhesive cementation with resin cement is the gold standard for providing reinforcement and retaining silica-based restorations (Peutzfeldt et al., 2011; Blatz et al., 2018; Johnson et al., 2018; Sousa et al., 2019). The strengthening effect of resin-based luting agents can occur due to the formation of a ceramic-resin hybrid layer resulting from the resin interpenetrating onto the etched ceramic surface (Fleming et al., 2006; Spazzin et al., 2016). Thus, the fracture resistance of ceramic restorations increases when the adhesion mechanism occurs (May et al., 2012), as it improves stress transmission through the bonded interface, consequently enhancing the clinical survival of these restorations (Malament and Socranski, 2001).

Resin bonding requires multiple pre-treatment steps on the intaglio surface of the restoration (Blatz et al., 2018) to achieve an intimate bond between the ceramic surface and the luting agent (Fleming et al., 2006). In contrast, hydrofluoric acid (HF) etching and silane coupling agent application is the classic surface treatment protocol for a glass ceramic (based on feldspathic, leucite-enhanced and lithium disilicate) (Blatz et al., 2003; Blatz et al., 2018). HF promotes surface alterations (mechanical interlocking) for the micro-retention of resin cements and silane chemically acts in this set (Horn, 1983; Dimitriadi et al., 2018; Matinlinna et al., 2018). Although HF acid can effectively improve the bond strength between glass-ceramics and resin cement (Brentel et al., 2007), this acid can be toxic and may trigger acute and chronic symptoms in the human body (Kirkpatrick et al., 1995; Ozcan et al., 2012). Moreover, an over-etched ceramic surface can lead to deleterious effects on the flexural strength of lithium disilicate ceramics, mainly as a result of the increase in surface defect population (Zogheib et al., 2011; Xiaoping et al., 2014).

Nowadays, a one-step self-etching ceramic primer (E&P, Monobond Etch & Prime, Ivoclar Vivadent) has been proposed as an alternative to HF etching to treat glass ceramic surfaces. According to the manufacturer, this primer is less harmful and requires shorter clinical time due to less application steps compared to the conventional protocol (HF etching plus coupling agent). Still in this sense, the E&P promotes a physicochemical conditioning through a mild etchant (ammonium polyfloride) and a trimethoxypropyl metracrylate for silanization, resulting in a reduced number of defects on the ceramic surface due to the more superficial alterations than those produced by HF etching, which might favor better fatigue performance (Tribst et al., 2019). However, the few existing studies evaluating fatigue performance showed similar results between the classical protocol and the treatment with E&P (Schestatsky et al., 2019), or superior fatigue behavior of hydrofluoric acid plus coupling agent (Scherer et al., 2018). It is important to clarify that the aforementioned studies evaluated the etching time recommended by the manufacturer (20 s of active application and 40 s of passive).

In addition to studies on HF etching time (Zogheib et al., 2011; Xiaoping et al., 2014; Puppin-Rontani et al., 2017; Wong et al., 2017), the application time of one-step self-etching ceramic primer may be changed in an attempt to improve and ensure the best mechanical performance (bond and mechanical improvements) of lithium disilicate restorations. This time variation affects the introduction of surface defects, which might have dichotomic consequences, such as: i) it might be positive when additional defects produced by a longer etching time are filled in with resin cement, inducting better stress distribution to the support material, and consequently reducing the probability for failure (May et al., 2012); ii) it will have negative effects when the number/size/shape of the defects make it difficult for the resin cement to penetrate the defects, thus impairing bonding, inducing slow crack growth mechanisms and poor mechanical performance (Anusavice and Hojjatie, 1992).

Taking into consideration the toxicity of hydrofluoric acid and the absence of studies evaluating the increased of E&P ceramic primer application time on the fatigue behavior of simplified lithium disilicate restorations, this study aimed to answer two main questions: i) Does the one-step ceramic primer promote similar or better fatigue behavior of the glass-ceramic restorations than the treatment with hydrofluoric acid etching?; ii) Does the longer conditioning time of the one-step ceramic primer improve the fatigue performance of the restorations?

Therefore, the present study aims to evaluate the effect of increased etching time of E&P one-step ceramic primer compared to the classical protocol (hydrofluoric acid plus coupling agent) on the short and long-term fatigue performance of lithium disilicate glass-ceramic restorations adhesively cemented onto a dentin analogue substrate. The assumed hypotheses are: (1) the increased etching time of E&P will promote similar fatigue behavior among the proposed conditionings; and (2) fatigue performance will be influenced by aging.

2. Material and Methods

2.1. Materials and study design

The general description of the materials used in the present study, their manufacturers, composition, and batch numbers are listed in Table 1.

This study was designed in 8 study groups, considering 2 factors (n= 15) (Table 2):

i) “Ceramic surface treatment” in 4 levels: PRIMER: only coupling agent application; HF5 + PRIMER: 5% hydrofluoric acid etching and coupling agent application; E&P 20s + 40s and E&P 20s + 5min: ceramic etching/priming (E&P, Monobond Etch & Prime) for 20 s of active application followed by 40 s or 5 min of passive application, respectively;

ii) “Aging condition” in 2 levels: baseline: tests after 24 h until 5 days from the cementation; or aged: water storage for 90 days + 12,000 thermal cycles levels before testing.

2.2. Specimen assembly description and sample preparation

The simplified test assembly employed in the present study has been widely used in the literature (Chen et al., 2014; Prochnow et al., 2018; Scherer et al., 2018). It consists of a simplified occlusal restoration for a posterior tooth represented by a lithium disilicate disc with a final diameter of 10 mm - average occlusal table of a first molar (Ferrario et al., 1999), which is adhesively cemented onto a glass fiber reinforced epoxy resin disc as a dentin analogue substrate with the same diameter of the ceramic disc.

2.2.1. Preparation of dentin analogue discs

The dentin substrate was represented by an epoxy resin disc (thickness= 2.0mm; Ø= 10mm). These discs were produced from epoxy resin plates (150 × 350 × 2.0 mm; Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany), which were shaped into cylinders using a diamond drill (internal diameter= 10 mm; Diamant Boart, Brussels, Belgium) coupled to a bench drill (SBE 1010 Plus, Metabo; Nürtingen, Germany) under refrigeration. The discs were manually polished on both sides after cutting with grit silicon carbide papers

(SiC, #400- and #1200-grit) and cleaned in an ultrasonic bath (distilled water; 5 min).

2.2.2. Preparation of ceramic discs

Lithium disilicate glass ceramic blocks for CAD/CAM (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were shaped into cylinders using a diamond drill (Ø = 10 mm; Diamant Boart) in a drilling machine (SBE 1010 Plus, Metabo) under refrigeration. The cylinders were cut (Isomet 1000, Buehler, Lake Bluff, USA) under water cooling, resulting in 120 discs. They were polished with manual pressure in a polishing machine (EcoMet/AutoMet 250, Buehler, Lake Bluff, USA) on both surfaces with #120-, #400, and #1200-grit SiC papers to achieve a thickness of 1.0 mm and standardize the surfaces.

2.2.2.1. In-lab simulation of CAD/CAM milling roughness

After polishing, the ceramic discs were subjected to an in-lab simulation of the CAD/CAM milling roughness, since machining process introduces defects and promotes a higher roughness in the cementation surface of the ceramic restorations (Fraga et al., 2017). A standardized size (100 mm × 50 mm) of #60 grit SiC paper was used for each slice. The ceramics discs were marked (axes x and y) with a marking pen and submitted to manual grinding with humidified SiC papers by a single trained operator (Rodrigues et al., 2018). The manual grinding was performed applying light digital pressure for 15 s on each axis (x and y). After the milling simulation, the ceramic slices were crystallized (Vacumat 6000 MP, VITA Zahnfabrik, Bad Sackingen, Germany) according to the manufacturer’s instructions, and the roughness values of all cementation ceramic surfaces were measured to compare them to those generated by CAD/CAM machining (Fraga et al., 2017), as well as to certify that all groups received similar roughness prior to the surface treatments investigated herein. Next, six measurements were performed for each specimen (axes x and y) on a profilometer (Mitutoyo SJ-410, Mitutoyo Corporation, Kawasaki, Japan), and the average of each specimen was used for statistical analysis. The roughness parameters achieved by the in-lab simulation before ceramic surface treatment for Ra (µm) and Rz (µm) were respectively (mean ± standard deviation): PRIMER: 1.75±0.40; 10.44±1.17; HF5 + PRIMER: 1.67±0.15; 10.65±0.81; E&P 20s + 40s: 1.65±0.16; 10.48±0.83; E&P 20s + 5min: 1.60±0.15; 10.19±0.83. The roughness means were statistically similar (one-way ANOVA test, $\alpha = 5\%$), being compatible to the one generated by CAD/CAM machining (Ra: 1.84±0.18; Rz: 11.07±1.00) obtained by Fraga et al. (2017). The specimens were subsequently cleaned in an ultrasonic bath (1440 D, Odontobras, Ind. and Com. Equip. Med. Odonto. LTDA, Ribeirão Preto, Brazil) with isopropyl alcohol for 5 min.

2.3. Ceramic surface treatments

After the in-lab simulation of CAD/CAM milling and crystallization, the intaglio ceramic surfaces received one of the following surface treatments (Table 2):

PRIMER group: an alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate coupling agent (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein) was rubbed over the ceramic surface with a microbrush for 15 s and left to react for 45 s (for a total of 60 s), as recommended by the manufacturer.

HF5 + PRIMER group: 5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) was applied with a microbrush for 20 s, removed with air-water spray for 30 s and air dried for 30 s, and then the aforementioned coupling agent was applied as previously described for the PRIMER group.

E&P groups: a ceramic self-etching primer (E&P, Monobond Etch & Prime, Ivoclar Vivadent) was rubbed on the ceramic surface with a microbrush for 20 s actively and then left to react for 40 s (E&P 20s + 40s group - time recommended by the manufacturer), or 5 min (experimental time - E&P 20s + 5min group). It is emphasized that one drop had to be dispensed/reapplied at the time of 2:30 min in order to allow 5 min of passive exposure to the agent and to counteract its volatilization. The product was removed with air-water spray for 30 s and air dried for 30 s in all E&P groups.

All discs were subjected to an ultrasonic cleaning (distilled water for 5 min) after the ceramic surface treatments (i.e. before coupling agent application for PRIMER and HF + PRIMER groups, after etching in one-step for E&P group).

2.4. Cementation procedure

The cementation surface of dentin analogue discs was etched with 10% hydrofluoric acid (Condac Porcelana, FGM, Joinville, Brazil) for 1 min, followed by rinsing with air-water spray (30 s), air spray (30 s) and ultrasonic cleaning (5 min) with distilled water. Next, Multilink Primers A and B (Ivoclar Vivadent, Schaan, Liechtenstein), a dentinal primer recommended for dual-curing resin cement (Multilink N, Ivoclar Vivadent), were mixed in a 1:1 ratio, scrubbed onto the epoxy surface (30 s), and air-dried until a thin layer was obtained. The resin cement was manipulated according to the manufacturer’s instructions and applied onto the treated surfaces of the ceramic discs. Each ceramic disc was adhesively cemented to an epoxy disc under a constant load of 2.5 N for 10 min. The resin cement excesses were subsequently removed and the assemblies were light-cured (Radii-cal LED curing light, SDI, Bayswater, Australia) for five exposures of 20 s each (one in each direction of 5 positions: 0°, 90°, 180°, 270°, and on top).

2.5. Aging conditions

The cemented assemblies of each condition were randomly assigned into two following conditions, based on a previous study (Scherer et al., 2018): “**baseline**” - the samples were tested under fatigue after storage in distilled water at 37°C for approximately 24 h until 5 days, featuring a short-term situation; and “**aging condition**” – the samples were subjected to thermocycling (Nova Ética, São Paulo, Brazil) - 12,000 cycles (Andreatta Filho et al., 2005), 30 s baths at 5 and 55°C, transfer time of 5 s; and storage in distilled water at 37°C for 90 days before testing procedures, representing a long-term situation.

2.6. Step-stress fatigue test

The cemented assemblies (n= 15) were tested using the step-stress fatigue test approach according to previous studies (Dapieve et al., 2018; Schestatsky et al., 2019) in an electric machine (Instron ElectroPuls E3000, Instron, Norwood, USA). Cyclic loads were applied with a 40 mm diameter stainless-steel hemispheric piston (Kelly et al., 2010; Prochnow et al., 2018) under distilled water at a frequency of 20 Hz. An adhesive tape (110 µm) was placed on the occlusal surface of the restoration and a thin sheet of a non-rigid material (cellophane, 2.50 µm) was placed between the piston and the ceramic surface to reduce contact stress concentration (Kelly, 1999). An initial load of 200 N for 5000 cycles was performed to accommodate piston/specimen relation. Then, incremental steps of 50 N for 10,000 cycles starting from 400 N were applied until the failure (fracture or radial cracks) of the sample. The specimens were checked for cracks at the end of each step by light oblique transillumination (Dibner and Kelly, 2016). The evaluated outcome was radial crack or fracture, and therefore if this failure were found the sample was categorized as 'failed', and the fatigue test of this sample ended. The corresponding failure data (load and number of cycles to failure) of each sample were recorded for statistical analysis.

2.7. Fractographic analysis

All the specimens were inspected by stereomicroscope (Discovery V20, Carl Zeiss, Gottingen, Germany) after the fatigue test and representative samples (n= 1) were selected from each condition, in which the ceramic fragments were detached to access the origin of the defects. The fragments were then ultrasonically cleaned with 78% isopropyl alcohol (5 min), air-dried, gold-sputtered and analyzed under scanning electron microscopy (SEM - Vega3, Tescan, Czech Republic) at 200× and 1,000× magnifications to determine the crack origin characteristics.

2.8. Topographic analysis

Additional ceramic samples were produced to be inspected regarding the topographical changes, microstructure features and alterations after the surface treatments (n= 1), being that no coupling agent was applied for it, i.e. the surface in the PRIMER group was maintained as simulated CAD/CAM milling roughness. The samples for the analysis were cleaned in an ultrasonic bath with 78% isopropyl alcohol (5 min), air-dried and analyzed by Field Emission Scanning Electron Microscopy (FE-SEM, Sigma 300 VP, Carl Zeiss, Cambridge, England) at 500× and 20,000× magnifications. No alloy sputtering was necessary prior to FE-SEM analysis.

2.9. Cementation interface analysis

Additional sets of three layers (treated ceramic – resin cement – treated ceramic, n= 1) were produced based on previous studies (Spazzin et al., 2017; Coelho et al., 2019) for each surface treatment condition tested to inspect the morphology of the adhesive interfaces, the defects introduced by the treatments and the filling of these defects by resin cement. To do so, two ceramic discs of the same condition were adhesively cemented after performing the ceramic surface treatments, according to item 2.3. After storage (distilled water – 24 h), they were transversely sectioned in a cutting machine (Isomet 1000, Buehler). Then, the cross-sectional was mirror-polished (EcoMet/AutoMet 250, Buehler) using #600-, #1200-, and #2000-grit SiC papers, cleaned in an ultrasonic bath with 78% isopropyl alcohol (5 min) and air dried to be analyzed by Field Emission Scanning Electron Microscopy (FE-SEM, Sigma 300 VP, Carl Zeiss) at 10,000× magnification. No alloy sputtering was performed prior to FE-SEM analysis.

2.10. Data analysis

A statistical software program (IBM SPSS Software; IBM, Armonk, NY, USA) was used with a significance level of 0.05. Data distribution of fatigue failure load (FFL) in Newton and the number of cycles for failure (CFF) was accessed by the Shapiro-Wilk and Levene tests. Thus, a two-way ANOVA and post-hoc Tukey's test was executed to analyze the influence of the "surface treatment" and "aging" factors, as well as the interaction of both, and for comparing the surface treatments in the baseline and aging conditions. A survival analysis was also performed using the Kaplan Meier and Mantel-Cox (Log Rank) tests, and the survival probability was tabulated for each step of the test.

Additionally, FFL and CFF data were submitted to Weibull analysis using the Super SMITH Weibull 4.0k-32 software program (Wes Fulton, Torrance, United States) under the maximum-likelihood method to obtain the Weibull modulus of each condition. The Weibull modulus is used as a measure for the distribution of the values, which is a way to statistically access the mechanical reliability of a condition/parameter.

Fractographic, topographic and cementation interface analyses were qualitatively analyzed.

3. Results

Two-way ANOVA and Tukey's post-hoc test of FFL and CFF data revealed significant influence of the "surface treatment" ($p= 0.000$, $F= 27.167$) and "aging" ($p= 0.000$, $F= 28.764$) factors, as well as the interaction of both "aging x surface treatment" ($p= 0.000$, $F= 8.770$). In comparing the groups at baseline conditions, the surface treatments had no statistically significant difference except for the E&P 20s + 5min group (940.0 N; 123,000 cycles) compared to the PRIMER group (786.7 N; 92,333 cycles). In comparing the aging groups, the PRIMER presented the worst fatigue performance, while the other treatments showed no difference. Only the PRIMER group degraded when analyzing the fatigue stability (baseline Vs aging for each surface treatment), highlighting a dramatic decrease: 38.9% for FFL and 63.3% for CFF (Table 3), respectively.

The Weibull analysis showed greater mechanical structural reliability (Weibull modulus) using E&P 20s + 5min in comparison to only PRIMER application after aging, while there was no statistically significant difference among the tested groups in the baseline condition (Table 4). Furthermore, the survival analysis corroborates the aforementioned fatigue findings: the PRIMER groups samples failed earlier (lower survival rates), while the other surface treatments lasted longer until failure (higher survival rates) (Table 5).

The fractographic analysis showed that failures (fracture or radial crack) originated from the defects of the cement-ceramic interface (Fig. 1). Topographic images demonstrated the potential of surface treatments to promote surface alterations (glassy matrix removal and pull out of lithium disilicate crystals) at different intensities, where a higher number of defects were observed after the etching by the HF5 + PRIMER and E&P 20s + 5min groups (Fig. 2). In addition, FE-SEM revealed unfilled areas at the cementation interface with all the distinct topographic patterns introduced by the surface treatments. Also, the HF promoted deeper defects than the E&P treatments in the cementation interface analysis, in which shallower defects were observed with the increased exposure time to E&P (Fig. 3).

4. Discussion

The present study demonstrated that bonded lithium disilicate simplified restorations present similar fatigue behavior when submitted to increased etching time of E&P one-step ceramic primer compared to the classic surface treatment protocol (HF5 + PRIMER) and the recommended time by the manufacturer of the ceramic primer (E&P 20s + 40s). However, the E&P 20s + 5min group showed better fatigue performance compared to the PRIMER groups (baseline and aging conditions), rejecting the first hypothesis. All surface treatments were similar and stable in the aged condition, except for the coupling agent application (PRIMER group), which had the worst performance. This highlights that the adhesion promoted by the coupling agent alone is dramatically susceptible to degradation, affecting the fatigue behavior of the restorations. Thus, the second hypothesis was accepted.

As already known, the longevity of dental ceramics mainly depends on the adhesion durability among the substrate and the load-bearing capacity under fatigue of the restoration/assembly, in particular when performing minimally invasive preparations, i.e., restorations depending on adhesion (laminates, inlays, onlays) (Morimoto et al., 2016). Chemical and physical surface treatments have been proposed together for adhesion improvements in glass-ceramics, i.e. treatments for surface alterations (micromechanical bond mechanism) such as acid etchant, and for chemical activation (bond promoters) to adhere ceramic and resinous materials via siloxane bonds (Manso et al., 2011). The present study agrees with this premise, as it showed that it was not possible to promote stable fatigue performance of restorations under intermittent cyclical loading and wet environment if only a chemical mechanism is applied (coupling agent application only – PRIMER group).

The dual bonding mechanisms to etched and silane primed glass-ceramic surfaces are important due to the contribution of each individual treatment (Dimitriadi et al., 2019). Dental silane primers (included Monobond N used in the present study) are composed by the ɣ-methacryloxypropyl trimethoxysilane silanols (MPTMS), solvents (acetone/water), acidic and phosphate monomers. When silane is applied on a hydrated surface, the pH becomes acidic and, consequently, a low pH strongly accelerates hydrolysis (Dimitriadi et al., 2018). In fact, Dimitriadi et al (2018) showed that the thermal aging, led to pronounced hydrolysis and impaired the system performance, in accordance to our results (Table 3, Table 5) and as also proven by Heikkinen et al., 2013. Thus, the HF acid etching is clearly a fundamental step on the stability of the system under fatigue, where etching promotes adequate surface topography changes necessary to resin cement infiltration and enhancement of chemical interactions among substrates, which will optimize the restorative system stability (Scherer et al., 2018; Tribst et al., 2019).

Although there are superficial topographic changes produced by the In-lab simulation of CAD/CAM milling roughness in the present study, only the coupling agent was not able to promote long-term fatigue performance without a previous etching step (such as HF or E&P etching for silica-based ceramics). This assumption is corroborated by the smallest Weibull modulus and the high percentage of decrease in fatigue failure load (38.9%) and cycles for failure (66.3%) when comparing PRIMER group at the baseline and aging conditions (Table 3, Table 4, Table 5). The main reasons for this worse fatigue behavior include adhesion involving the high instability and hydrolytic degradation of bonds in the interfacial layer, which depends on the coupling agent molecular structure, concentration, pH, temperature and humidity, among others (Lung and Matinlinna, 2012; Matinlinna et al., 2018). Thus, it has to be highlighted that the topographical changes promoted by hydrofluoric acid etching (group HF5 + PRIMER) or the one-step ceramic conditioner primer (E&P 20s + 40s or E&P 20s + 5min) of the intaglio surface are key for bond improvements, in addition to chemical bonds, as the bond strength is dependent on the quantity and quality of defects inserted through surface conditioning (Fleming et al., 2006).

Representative images of FE-SEM (Fig. 2, Fig. 3) demonstrated smoother topographic changes when using the one-step conditioner primer following the manufacturer's instructions (E&P 20s + 40s) compared to classical hydrofluoric acid conditioning, according to previous literature (Scherer et al., 2018; Moreno et al., 2019). On the other hand, it is possible to observe substantial changes in this topography by increasing the time of E&P application, since a larger amount of lithium disilicate crystals was exposed (Fig. 2). In this sense, the E&P 20s + 5min group seems to present shallower defects when observing the cementation interface analysis (Fig. 3) due to the more homogeneous aspect of the interface, but these characteristics did not deleteriously impact the evaluated mechanical properties (Table 3, Table 4, Table 5).

Moreover, it important to state that the interfacial relationship (ceramic-cement) is crucial for the restoration performance, since it is the region with the highest concentration of tensile stresses (May et al., 2012); this is also observed by the fractography analysis (Fig. 1), which shows that all cracks started from defects in the cementation surface. Therefore, the defects produced by surface treatments play a strong role in inducing cracking, unless these defects are filled/healed by resin cement, promoting a strengthening effect by the ceramic (Fleming et al., 2006; Spazzin et al., 2017). Furthermore, it is important that the intimate contact between the ceramic and the cementing agent occurs, which should form a continuous and homogeneous interface, as the presence of unfilled porosity or irregularities can concentrate stresses and reduce the overall stress/load-bearing capacity (Spazzin et al., 2017; Bacchi et al., 2018). In this sense, it can be observed that all evaluated conditions presented bubbles and defects which were not filled in by resin cement in the cementation interface analysis performed in the present study (Fig. 3). Thus, resin-based systems which present different filling potentials from the one used in the present study could change the performance observed herein.

It also has to be pointed out that the tested one-step ceramic conditioner primer showed promising results from the fatigue evaluation standpoint (Table 3, Table 4 and Table 5). The physicochemical mechanism present in this ceramic primer composed of an ammonium polyfluoride (mild etchant) silane system based on trimethoxypropyl metacrylate is suitable for priming/etching the tested glass-ceramic. The manufacturer advises that, when applied, the product must be rubbed with a microbrush (for 20 s actively) and then left in contact with the ceramic surface to let it react (for 40 s passively) and then it should be washed with water. This step is required to remove the acid component and reaction byproducts leaving only a thin layer of silane that is chemically bonded with ceramic surface. It seems that the water washing step is being effective in removing residuals and, therefore, an additional treatment may not be necessary

(Moreno et al., 2019).

While some authors corroborate the similar or even better mechanical performance of E&P when compared to HF etching plus coupling agent (Schestatsky et al., 2019; Tribst et al., 2019), Scherer and collaborators (2018) reported superiority by the classic protocol. Even though it is a very similar methodology, this difference can be explained by the in-lab simulation of CAD/CAM milling surface roughness which occurred in the present study. The topographic changes generated by this in-lab simulation are observed in the roughness parameters identified in the methodology session and in the PRIMER group micrographs (Fig. 2, Fig. 3). Therefore, while in-lab simulation is not sufficient to create a suitable micromechanical interlocking, it can influence the surface topographic pattern generated by conditioning.

Finally, we have to state that our study has some limitations, such as using an in-lab simulation of CAD/CAM milling roughness instead of real CAD/CAM milled restorations and the fatigue evaluation of simplified restorations. Nevertheless, our findings contribute to clarifying an important clinical issue: the chemical and mechanical bonds (as promoted by hydrofluoric acid + coupling agent or one-step ceramic conditioner primer tested by us) are a must for fatigue improvements of glass-ceramic lithium disilicate restorations apart from the topography changes promoted by machining.

5. Conclusion

- The chemical and mechanical bond promoted by etching/priming treatments are necessary to promote higher fatigue performance of adhesively cemented lithium disilicate restorations.
- The one-step ceramic etching/priming agent demonstrated to be a suitable and promising glass-ceramic treatment in terms of its fatigue performance tested herein.
- The increased etching time did not improve the fatigue performance of glass-ceramic restorations, being a dispensable protocol.

ACKNOWLEDGEMENTS

The authors declare no conflict of interests and emphasize that this study was partly financed by the Brazilian Federal Agency for Coordination of Improvement of Higher Education Personnel (*CAPES*) (Finance code 001) and by the Foundation to Research Support of the Rio Grande do Sul State (*FAPERGS*). We especially thank Ivoclar Vivadent for donating the research materials, and finally we emphasize that those institutions had no role in the study design, data collection or analysis, decision to publish or in preparing the manuscript.

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FIGURES

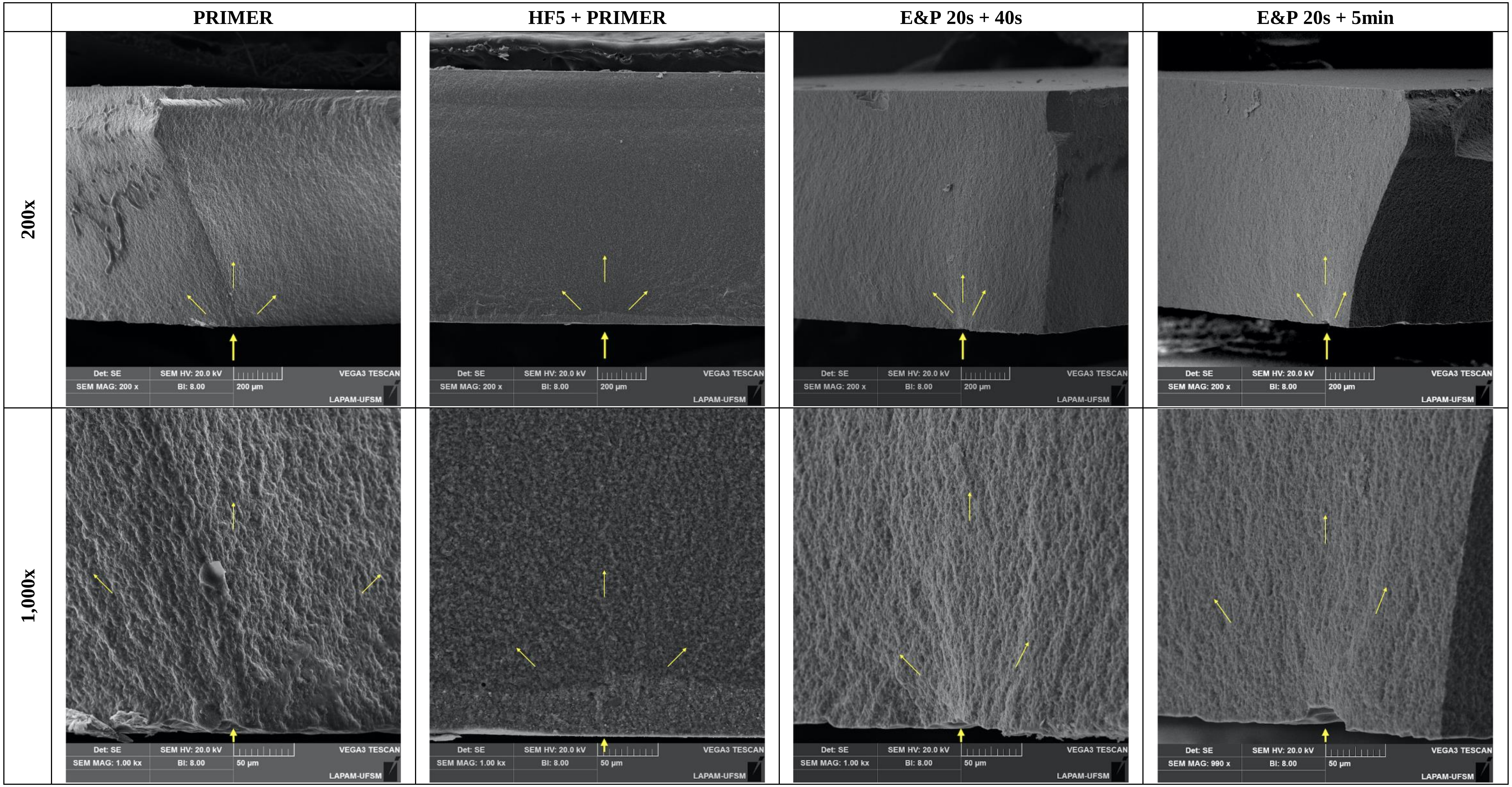


Figure 1. Micrographs (200× – top; 1,000× – bottom) obtained by SEM illustrating that all failures originated at cementation surface from defects present at the ceramic surface, pointed by yellow arrows, and then propagated to the opposite side (oclusal/top surface), where it notices the compression curl.

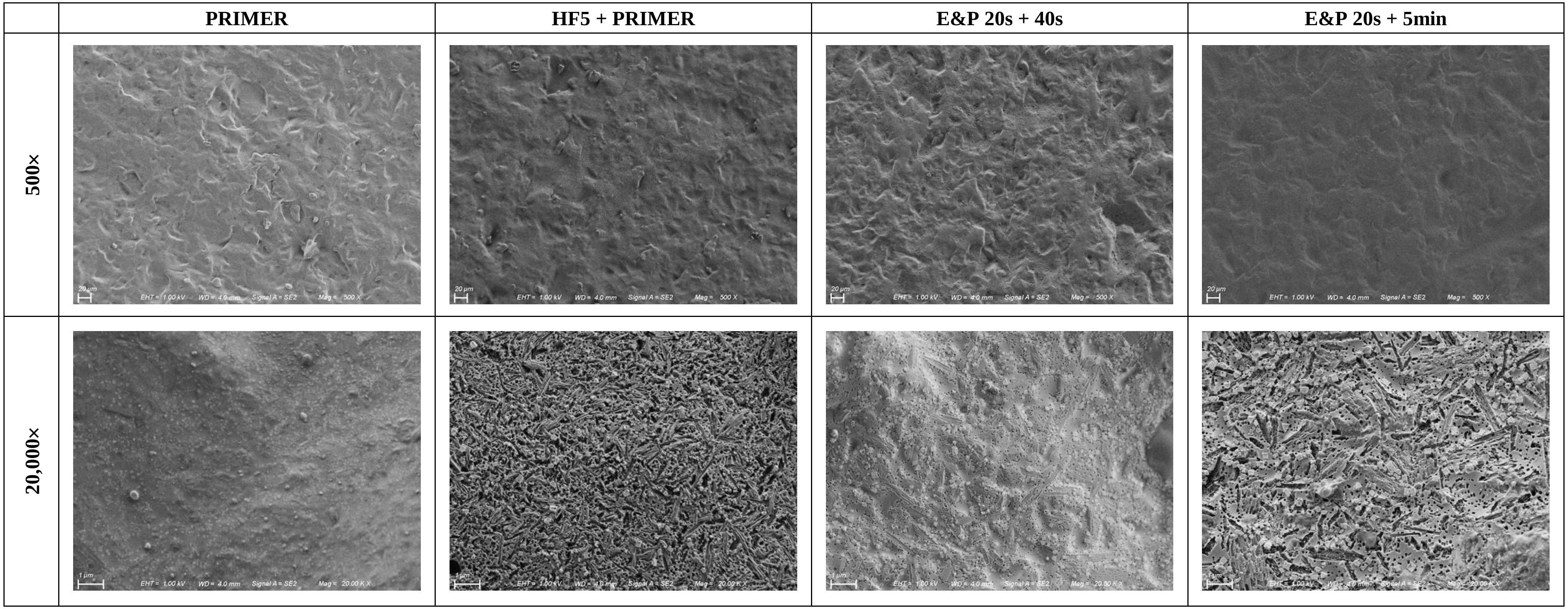


Figure 2. Topographic images (500× – top, 20,000× – bottom) obtained at FE-SEM analysis. It becomes clear the potential of the surface treatments on promote glass matrix dissolution in different intensities (higher at HF5+PRIMER and E&P 20s+5min), exposing crystallographic intergranular regions that will enable micromechanical interlocking, as also enhance the posterior chemical interaction with resin cement.

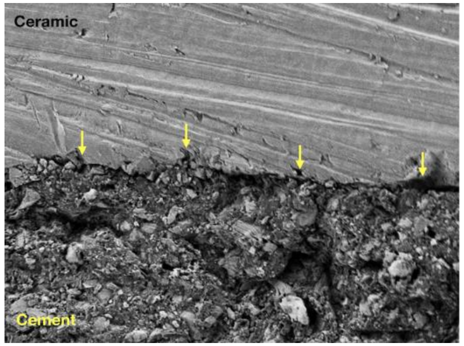
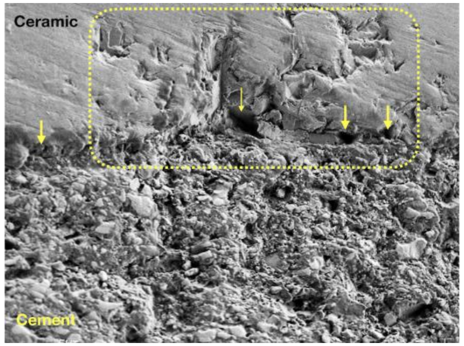
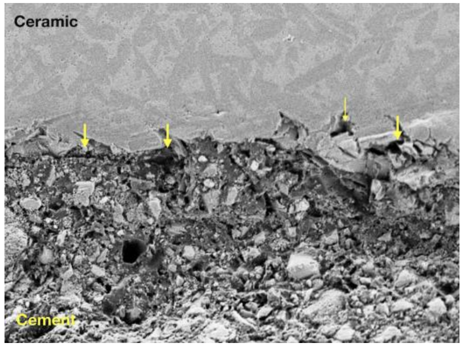
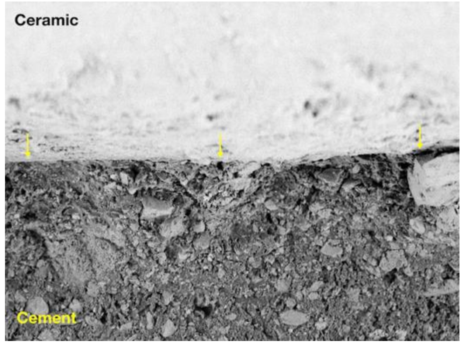
	PRIMER	HF5 + PRIMER	E&P 20s + 40s	E&P 20s + 5 min
10,000x				

Figure 3. Micrographs (10,000×) obtained by FE-SEM illustrating the intaglio surface between the ceramic and the cement, where it notices the presence of unfilled areas (pointed by yellow arrows) between these substrates in all conditions explored. The surface treatments (HF5 + PRIMER and E&P) introduces different patterns of defects, since HF promotes deeper defects than E&P and the increased exposure time to E&P leads to the presence of shallower defects.

TABLES

Table 1. Description of materials, commercial name, manufacturer, composition and batch number.

Material	Commercial name/manufacturer	Composition	Batch number
Lithium disilicate glass ceramic	IPS e.Max CAD, Ivoclar Vivadent, Schaan, Liechtenstein	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, other and colouring oxides	W93126
Ceramic primer coupling agent	Monobond N, Ivoclar Vivadent	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	W90329
5% hydrofluoric acid	IPS Ceramic Etching Gel, Ivoclar Vivadent	< 5% hydrofluoric acid	W14921
10% hydrofluoric acid	Condac Porcelana, FGM, Joinville, Brazil	< 10% hydrofluoric acid	W140319
Self-etching ceramic primer	Monobond Etch & Prime, Ivoclar Vivadent	Ammonium polyfluoride, silane system based on trimethoxypropyl methacrylate, alcohols, water and colorant	W40212
Dual cure resin cement	Multilink N, Ivoclar Vivadent	Silicate glass, ytterbium trifluoride, highly dispersed silica, catalysts and stabilizer, pigments	W44613
Primer	Multilink Primer (A and B), Ivoclar Vivadent	Primer A: water, initiators; primer B: phosphonic acid acrylate, hydroxyethyl methacrylate, methacrylate mod. polyacrylic acid, stabiliser	Primer A: W89775 Primer B: W92311
Epoxy resin	Carbotec GmbH & Co. KG, Königs Wusterhausen, Germany	Continuous filament woven fiberglass bonded with epoxy resin	-

*The chemical composition is described according to the manufacturers' information.

Table 2. Experimental design.

Group	Surface treatment	Aging condition
PRIMER	Coupling agent application	Baseline* Aging**
HF5 + PRIMER	5% HF acid etching for 20 seconds + coupling agent application	Baseline Aging
E&P 20s + 40s	20 seconds of active application and 40 seconds of react***	Baseline Aging
E&P 20s + 5min	20 seconds of active application and 5 minutes of react	Baseline Aging

* 1 up to 5 days of water distilled storage (37 ◦C) before testing;
**90 days of storage in distilled water + thermocycling: 12,000 cycles between 5 and 55 ºC with a dwell time of 30 s and a transfer time of 5 s;
***recommended by the manufacturer.

Table 3. Mean fatigue failure load (FFL) in Newton, number of cycles for failure (CFF) with respective standard deviation (SD) and the percentage of decrease comparing the baseline condition to aging in both outcomes.

Groups	FFL			CFF		
	Baseline	Aging		Baseline	Aging	
	Mean (SD)	Mean (SD)	% of mean FFL decrease (Baseline / aging)	Mean (SD)	Mean (SD)	% of mean CFF decrease (baseline / aging)
PRIMER	786.67 (213.36) B	480.77 (90.23) C	38.9%	92,333 (42,673) B	31,154 (18,046) C	66.3%
HF5 + PRIMER	830.00 (127.90) AB	810.00 (82.81) AB	2.4%	101,000 (25,579) AB	97,000 (16,562) AB	4%
E&P 20s + 40s	880.00 (106.57) AB	840.00 (96.73) AB	4.5%	111,000 (21,314) AB	103,000 (19,346) AB	7.2%
E&P 20s + 5min	940.00 (98.56) A	840.00 (54.12) AB	10.6%	123,000 (19,712) A	103,000 (10,823) AB	16.3%

* Different uppercase letters indicate statistical differences based on Two-Way ANOVA and Tukey's post-hoc test.

Table 4. Weibull modulus for fatigue failure load (FFL) and cycles for failure (CFF).

Groups	FFL		CFF	
	Baseline	Aging	Baseline	Aging
	Weibull modulus (CI)	Weibull modulus (CI)	Weibull modulus (CI)	Weibull modulus (CI)
PRIMER	4.67 (2.93 – 6.93) A	5.30 (3.43 – 7.48) B	2.40 (1.49 – 3.60) B	1.94 (1.24 – 2.79) B
HF5 + PRIMER	7.91 (5.08 – 11.43) A	9.87 (6.57 – 13.69) AB	4.78 (3.05 – 6.94) AB	6.12 (4.06 – 8.52) A
E&P 20s + 40s	8.74 (5.78 – 12.22) A	10.51 (6.75 – 15.18) AB	5.66 (3.73 – 7.95) A	6.47 (4.14 – 9.40) A
E&P 20s + 5min	10.45 (6.93 – 14.47) A	16.24 (10.71 – 22.80) A	6.99 (4.61 – 9.73) A	10.14 (6.67 – 14.26) A

* Different uppercase letters indicate statistical differences based on maximum-likelihood estimations for Weibull analysis.

Table 5. Survival rates – probability of specimens to exceed the respective fatigue failure load (FFL) and number of cycles for failure (CFF) step without crack propagation, and its respective standard error values.

Surface treatment	Aging condition	FFL (N) / CFF																
		200 / 5,000	**400 / 15,000**	**450 / 25,000**	**500 / 35,000**	**550 / 45,000**	**600 / 55,000**	**650 / 65,000**	**700 / 75,000**	**750 / 85,000**	**800 / 95,000**	**850 / 105,000**	**900 / 115,000**	**950 / 125,000**	**1000 / 135,000**	**1050 / 145,000**	**1100 / 155,000**	**1150 / 165,000**
PRIMER	**Baseline**	1	0.93 (0.06)	0.87 (0.09)	0.87 (0.09)	0.80 (0.10)	0.73 (0.11)	0.67 (0.12)	0.67 (0.12)	0.53 (0.13)	0.53 (0.13)	0.47 (0.13)	0.40 (0.13)	0.13 (0.09)	0.13 (0.09)	0.0	-	-
	Aging	1	0.69 (0.13)	0.39 (0.14)	0.23 (0.12)	0.15 (0.10)	0.08 (0.07)	0.08 (0.07)	0.0	-	-	-	-	-	-	-	-	-
HF5 + PRIMER	**Baseline**	1	1	1	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.80 (0.10)	0.73 (0.11)	0.47 (0.13)	0.33 (0.12)	0.27 (0.11)	0.20 (0.10)	0.0	-	-	-
	Aging	1	1	1	1	1	1	1	0.87 (0.09)	0.60 (0.13)	0.40 (0.13)	0.20 (0.10)	0.07 (0.06)	0.07 (0.06)	0.0	-	-	-
E&P 20s + 40s	**Baseline**	1	1	1	1	1	1	1	0.93 (0.06)	0.80 (0.10)	0.80 (0.10)	0.47 (0.13)	0.27 (0.11)	0.13 (0.08)	0.13 (0.08)	0.07 (0.06)	0.0	-
	Aging	1	1	1	1	1	1	1	0.87 (0.09)	0.60 (0.13)	0.60 (0.13)	0.53 (0.13)	0.13 (0.09)	0.07 (0.06)	0.0	-	-	-
E&P 20s + 5min	**Baseline**	1	1	1	1	1	1	1	0.93 (0.06)	0.93 (0.06)	0.93 (0.06)	0.87 (0.08)	0.60 (0.13)	0.27 (0.11)	0.13 (0.09)	0.07 (0.06)	0.07 (0.06)	0.0
	Aging	1	1	1	1	1	1	1	1	0.93 (0.06)	0.53 (0.13)	0.27 (0.11)	0.07 (0.06)	0.0	-	-	-	-

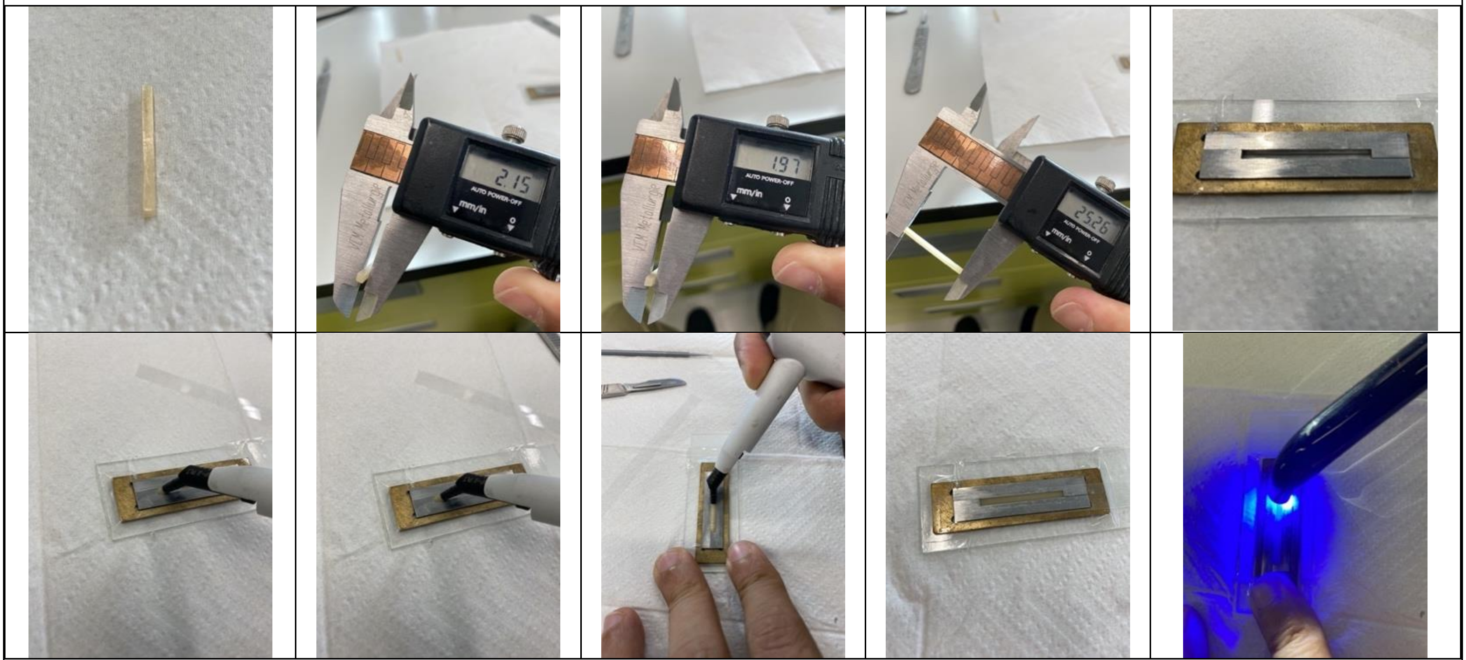
* Kaplan Meier and Mantel-Cox tests.

** The symbol '-' indicates absence of specimens being submitted to the respective category.

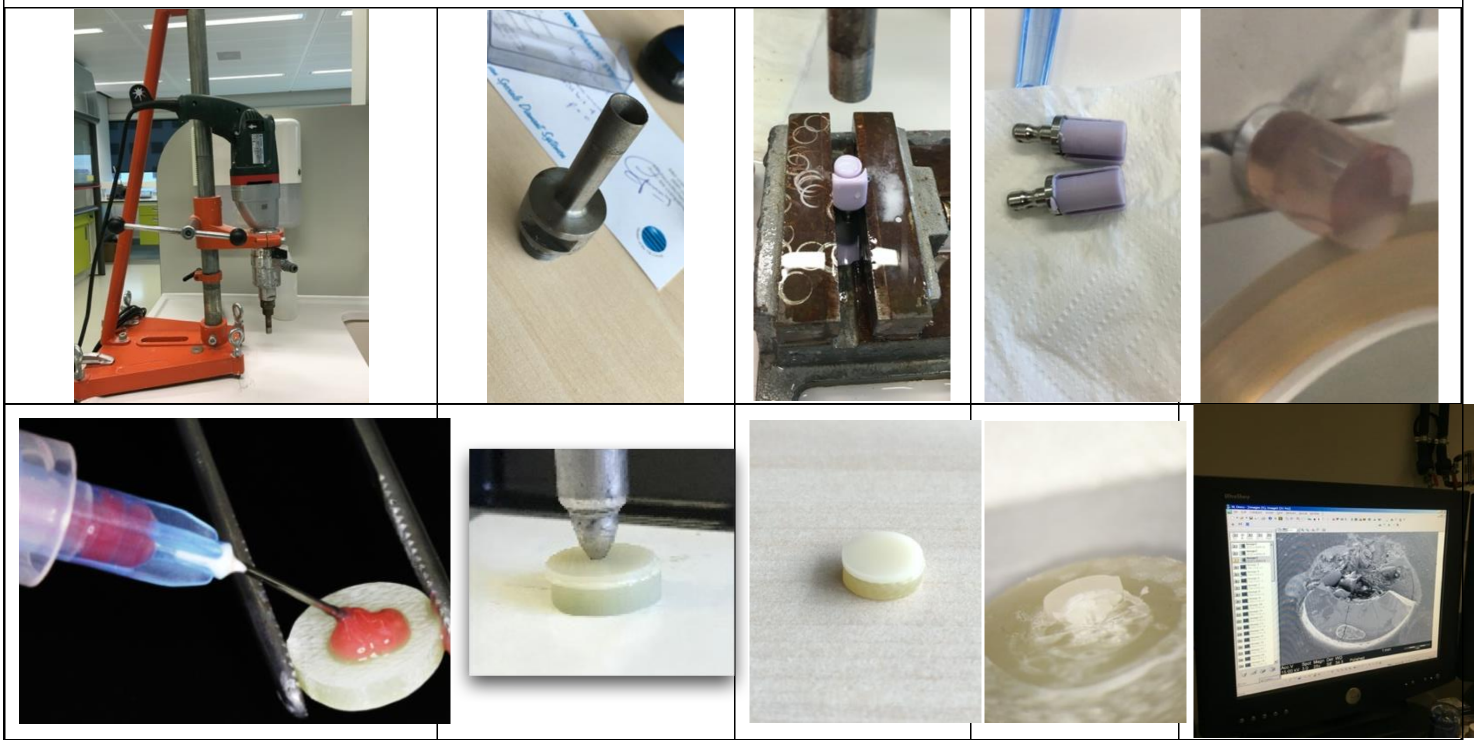
APÊNDICE TÉCNICO

- Treinamento na obtenção de amostras para ensaios em fadiga e análises topográficas e fractográficas posteriores.

Preparo de amostras para fadiga sob flexão em 3 pontos.



Preparo de amostras para fadiga sob compressão de geometrias cimentadas (g10 – cimento – material restaurador).

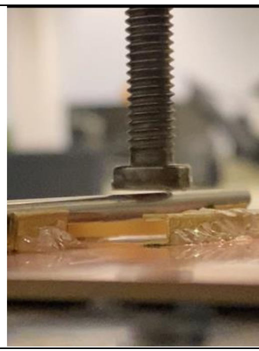


- ensaios de fadiga no sentido de desenvolver nova metodologia de ensaio para acelerar caracterização de materiais em flexão de 3 pontos.

O professor Cornelis Johannes Kleverlaan propôs um conjunto de ensaio em flexão 3 pontos que permite testar dez amostras simultâneas a fim de acelerar a obtenção de dados e a caracterização do comportamento de materiais restauradores utilizados no contexto odontológico. Estudos pilotos foram executados e resultados promissores encontrados. Nesse sentido, a metodologia encontra-se em otimização para posterior escrita de artigo para publicação. Imagens ilustrativas do conjunto de ensaio encontram-se abaixo.







- Ensaio de resistência flexural 3 pontos, obtenção de dados, análise estatística e discussão dos dados obtidos para escrita de um artigo.

O foco desse estudo é a análise da influência da região do bloco utilizado na usinagem do sistema CAD/CAM de restaurações indiretas. No momento da confecção da restauração indireta, o técnico de laboratório seleciona o posicionamento ideal do bloco para a obtenção de características ópticas desejadas no caso em tela. Entretanto, inexistem dados na literatura que corroborem que a compactação destes blocos pelos fabricantes propicie materiais que apresentam performance mecânica similar nas diferentes regiões consideradas. Além disso, não há estudos que comparem materiais resinosos indiretos (blocos CAD/CAM) com diretos, os quais foram confeccionados e compactados pelos próprios pesquisadores. Neste sentido, diversas barras para ensaio de flexão em 3 pontos foram confeccionadas e ensaiadas conforme preconizado na ISSO 6872:2015. Por conseguinte, os dados foram submetidos a análise descritiva e a análise de variância 1 via, considerando os diferentes materiais em cada região do bloco (interna ou externa). Além disso, testes de comparação t de Student foram utilizados para comparar diferentes regiões dentro de cada material e uma análise de variância 1 via também foi executada para comparar os materiais com os dados associados das diferentes regiões. Adicionalmente, análise de Weibull para obtenção de resistência característica e do modulo de Weibull em cada um desses cenários também foi calculado. Os dados estão expostos nas tabelas abaixo:

Table 1 – Mean flexural strength (Standard deviation) of the three point bending test considering the different materials and regions (inside, outside or merged).

Material	Flexural strength		
	Inside	Outside	Merged
KAV	248.08^{Ba} (36.36)	249.01^{Ba} (17.24)	248.49^{B} (29.08)
NICE	196.97^{Ca} (34.56)	200.10^{Ca} (27.96)	198.32^{C} (31.56)
APX	189.09^{Ca} (31.20)	196.18^{Ca} (30.74)	191.60^{C} (30.72)
FSUP	112.81^{Ea} (39.41)	130.37^{Da} (31.76)	120.92^{E} (36.81)
LULT	232.51^{Ba} (18.51)	219.31^{BCb} (19.63)	226.23^{B} (19.97)
ENAM	142.02^{DEa} (14.13)	142.76^{Da} (15.75)	142.42^{DE} (14.87)
EMP	144.40^{Da} (25.92)	144.51^{Da} (38.84)	144.45^{D} (32.36)
VMII	122.35^{DEa} (12.58)	112.53^{Da} (8.85)	119.54^{DE} (12.29)
EMAX	347.49^{Ab} (55.08)	392.10^{Aa} (78.28)	369.79^{A} (70.52)

* Different uppercase letters indicate statistical differences on each column.
* Different lowercase letters indicate statistical differences on comparing inside and outside regions for each material.

Table 2 – Weibull analysis (Characteristic strength – σ_c; and Weibull modulus - m) for flexural strength considering inside, outside regions and merged measurements for each material.

Material	σ_c (95% CI)		m (95% CI)		σ_c (95% CI)	m (95% CI)
	Inside	Outside	Inside	Outside	Merged	
KAV	264.00^{Ba} (247.50 – 281.50)	256.70^{Ba} (248.70 – 265.00)	7.32^{ABb} (4.97 – 10.78)	16.41^{Aa} (10.86 – 24.81)	261.30^{B} (251.90 – 271.00)	9.59^{ABC} (7.27 – 12.65)
NICE	209.20^{Ca} (197.50 – 221.60)	209.20^{Ca} (199.00 – 219.80)	7.42^{Ba} (6.09 – 9.05)	10.33^{ABa} (8.68 – 12.30)	209.20^{C} (201.40 – 217.40)	8.60^{BC} (7.54 – 9.81)
APX	201.60^{Ca} (188.80 – 215.20)	209.60^{Ca} (190.60 – 230.50)	7.03^{Ba} (4.96 – 9.97)	6.67^{ABCa} (3.97 – 11.20)	204.20^{C} (194.00 – 215.10)	7.17^{BCD} (5.37 – 9.57)
FSUP	126.10^{DEa} (111.20 – 143.00)	143.20^{Da} (129.80 – 158.10)	3.10^{Ca} (2.31 – 4.18)	4.30^{Ca} (3.07 – 6.02)	134.50^{E} (123.90 – 145.90)	3.51^{E} (2.80 – 4.39)
LULT	240.60^{Ba} (233.50 – 248.00)	227.70^{Ca} (219.70 – 235.90)	14.62^{Aa} (10.71 – 19.97)	13.08^{ABa} (9.29 – 18.44)	234.90^{C} (229.30 – 240.50)	13.43^{A} (10.74 – 16.79)
ENAM	148.10^{Da} (142.50 – 153.90)	149.60^{Da} (143.80 – 155.60)	11.67^{ABa} (8.19 – 16.64)	10.43^{ABa} (7.61 – 14.30)	148.80^{DE} (144.80 – 152.80)	11.40^{AB} (9.05 – 14.36)
EMP	155.30^{Da} (144.80 – 166.50)	158.60^{Da} (142.80 – 176.10)	6.07^{Ba} (4.34 – 8.49)	4.21^{Ca} (3.06 – 5.79)	156.80^{D} (147.80 – 166.50)	5.13^{DE} (4.13 – 6.35)
VMII	127.70^{Ea} (121.80 – 134.00)	116.40^{Ea} (109.30 – 124.00)	11.13^{ABa} (7.45 – 16.61)	13.66^{ABa} (6.66 – 28.01)	124.70^{E} (119.90 – 129.70)	11.51^{AB} (8.39 – 15.79)
EMAX	369.80^{Aa} (347.10 – 393.90)	420.50^{Aa} (389.80 – 453.50)	7.31^{Ba} (5.28 – 10.12)	6.15^{BCa} (4.57 – 8.27)	395.30^{A} (376.00 – 415.60)	6.64^{CD} (5.50 – 8.02)

* Different uppercase letters indicate statistical differences on each column.

* Different lowercase letters indicate statistical differences on comparing inside and outside regions for each material at the same measurement (σ_c or m).