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COMPORTAMENTO MECÂNICO DE INFILTRANTES EXPERIMENTAIS E COMERCIAL

MECHANICAL BEHAVIOR OF EXPERIMENTAL AND COMMERCIAL INFILTRANTS

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Trabalho de Conclusão de Curso apresentado à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Cirurgião Dentista.

Undergraduate final work presented to the Piracicaba Dental School of the University of Campinas in partial fulfillment of the requirements for the degree of Dental Surgeon

Orientador: Profa. Dra. Giselle Maria Marchi Baron

Coorientador: Priscila Regis Pedreira

ESTE EXEMPLAR CORRESPONDE À VERSÃO FINAL DO TRABALHO DE CONCLUSÃO DE CURSO APRESENTADO PELA ALUNA JULIANA MINTO BOLDIERI E ORIENTADO PELA PROFESSORA DRA. GISELLE MARIA MARCHI BARON.

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RESUMO

O objetivo desse estudo foi avaliar a influência da adição de sal de iodônio (DFI) e quitosana ao infiltrante experimental em comparação com o comercial Icon® nas propriedades físicas. Foram preparados nove infiltrantes experimentais contendo a base monomérica de trietilenoglicol dimetracrilato (TEGDMA) 75% e bisfenol-A dimetracrilato etoxilato (BisEMA) 25%; 0,5 mol% de canforoquinona (CQ) e 1 mol% de 4- dimetilaminobenzoato de etila (EDAB) como sistema fotoiniciador e 10% do monômero hidroxietil metacrilato (HEMA). Na formulação foram adicionadas variantes de concentração de quitosana em 0; 0,12; e 0,25% e DFI em 0; 0,5 e 1%; e o infintrante Icon® como grupo controle comercial. Foi realizado o teste de ângulo de contato (n=5) por um goniômetro. Em segunda etapa, molares humanos foram submetidos à indução de lesão cariosa, depois infiltrados e submetidos à escovação simulada na máquina de ensaio (15.000 ciclos). A rugosidade superficial foi avaliada no rugosímetro, (n=10) em três tempos (T0 - após simulação de lesão cariosa, T1 - após infiltração e T2 após escovação). Os dados do ângulo de contato foram analisados por meio de ANOVA one-way com post-hoc de Tukey e o de rugosidade pelo modelo linear generalizado para medidas repetidas no tempo, ambos com nível de significância de 5%. Os grupos com a associação de DFI e quitosana obtiveram maior ângulo de contato e após a a escovação, uma maior rugosidade de superfície. Assim, podemos sugerir que a adição de DFI e quitosana pode produzir melhorias nas propriedades físicas dos infiltrantes resinosos, porém pode aumentar a viscosidade do material.

Palavras-chave: Cárie Dentária; Infiltração Dentária; Ônio compostos; Quitosana.

Abstract

The objective of this study was to evaluate the influence of the addition of iodonium salt (DFI) and chitosan on the experimental infiltrant in comparison with the commercial Icon® on the physical properties. Nine experimental infiltrants were prepared containing the monomeric base of triethylene glycol dimetracrylate (TEGDMA) 75% and bisphenol-A dimetracrylate ethoxylate (BisEMA) 25%; 0.5 mol% of camphorquinone (CQ) and 1 mol% of ethyl 4-dimethylaminobenzoate (EDAB) as a photoinitiator system and 10% of the hydroxyethyl methacrylate monomer (HEMA). Variations of chitosan concentration at 0 were added to the formulation; 0.12; and 0.25% and DFI at 0; 0.5 and 1%; and the Icon® infiltrant as a commercial control group. The contact angle test (n = 5) was performed using a goniometer. In the second stage, human molars were subjected to induction of carious lesions, then infiltrated and subjected to simulated brushing on the testing machine (15,000 cycles). The surface roughness was evaluated on the rugosimeter, (n = 10) in three stages (T0 - after simulation of a carious lesion, T1 - after infiltration and T2 after brushing). The contact angle data were analyzed using one-way ANOVA with Tukey's post-hoc and roughness analysis using the generalized linear model for repeated measures over time, both with a 5% significance level. The groups with the association of DFI and chitosan had a greater contact angle and after brushing, a greater surface roughness. Thus, we can suggest that the addition of DFI and chitosan can produce improvements in the physical properties of resinous infiltrants, however, it can increase the viscosity of the material.

Key-Words: Dental cavity; Tooth infiltration; Onium compounds; Chitosan.

SUMÁRIO

1) Introdução

Nas últimas cinco décadas ocorreu um declínio na incidência da cárie dental devido a políticas de implementação da fluoretação da água e uso de creme dental e enxaguatórios bucais fluoretados, acarretando em melhoria na saúde bucal em nível mundial (Whelton et al., 2019). Porém, a cárie ainda é a doença bucal que mais acomete grande parte da população mundial. (Gao et al., 2016). Sendo uma doença que na sua fase inicial apresenta uma lesão com aspecto de mancha branca, opaca e rugosa, com sua etiologia composta por multifatores como bactérias cariogênicas, carboidratos fermentáveis e disfunção salivar (Sfalcin, 2015; Silverstone et al., 1988). A cárie possui o seu desenvolvimento e progressão através do processo de desmineralização da estrutura dental, que varia entre lesões cavitadas e não cavitadas. Neste processo, a hidroxiapatita sofre transformações estruturais em seus cristais (Silverstone et al., 1988). Esta, por sua vez, sofre dissolução ácida por meio da diminuição do pH, a qual é dada pela metabolização de carboidratos simples, formando ácidos fracos pelas bactérias cariogênicas como *Streptococcus mutans,* dando início a lesão cariosa (Tanaka et al., 1999).

Pode-se ressaltar que as lesões de cárie com maior incidência na população são as localizadas nas regiões interproximais dos dentes, em decorrência do difícil acesso para realização de uma higiene eficaz (Meyer- Lueckel, 2016). E são mais frequentes, também, em pacientes em tratamento ortodôntico com braquetes, devido ao acúmulo de restos alimentares ao redor desses e sua custosa higienização. (Mahmoudzadeh et al., 2018).

O tratamento mais realizado para cárie dentária é o restaurador, com a remoção de todo o tecido dentário infectado e, posteriormente, a colocação do material para restaurar a parte perdida do órgão dental (Zhang et al., 2016). Porém, o procedimento restaurador convencional pode provocar a extensão excessiva da cavidade, causando o enfraquecimento da estrutura dental. Assim, aumentando as chances de fratura e diminuindo a longevidade da restauração. Devido a isso, houve o surgimento da Odontologia Minimamente Invasiva (OMI) (Chalmers, 2006), que tem como seu principal objetivo interromper o ciclo vicioso de restaurações repetidas, com o conceito de manter a estrutura do dente sadia, aumentando a vitalidade dental e preservando a sua funcionalidade na cavidade oral (Frencken et al., 2012).

E para evitar o tratamento restaurador devido à progressão do desenvolvimento da lesão de mancha branca (MB), é primordial que o Cirurgião Dentista faça a orientação comportamental do paciente. Como exemplo, aconselhamento sobre a frequência da ingestão de açúcares, instrução adequeada de higiene oral com creme dental com flúor. (Frencken et al., 2012). Após a orientação e manejo do paciente, é possível recorrer a diversas opções de tratamento na Odontologia Minimamente Invasiva para o tratamento da doença cárie, sendo que a fluorterapia, previamente, era a primeira opção quando se trata de lesões superficiais de mancha branca (Sant'anna et al., 2016). Essa, no entanto, não leva a resultados satisfatórios quando aplicada em estágios mais avançados da doença e depende muito da colaboração do paciente para resultados satisfatórios em lesões iniciais (Paris et al., 2007). Existe, também, a utilização do verniz CPP-ACP que é um nanocomplexo derivado da proteína do leite, porém é menos eficiente que os fluoretos para estabilização da lesão inicial de cárie (Wu et al., 2020). Posteriormente, criaram o CPP-ACFP, uma combinação do verniz CPP-ACP com flúor para uma maior eficácia no combate da lesão no esmalte (Baafif et al., 2020). Mas também, há a utilização dos selantes, os quais formam somente um obstáculo mecânico externo na superfície da lesão de mancha branca (Lasfargues et al., 2013).

A tentativa de infiltrar e paralisar lesões em esmalte vem desde 1970, com estudos em adesivos. Porém, nas últimas décadas, estudos descobriram que mesmo após o ataque ácido do esmalte com ácido clorídrico, a infiltração por adesivos mostrou-se superficial e heterogênea, ou seja, ineficiente (Paris et al., 2012). Visto isso, com o objetivo de solucionar as desvantagens das técnicas citadas, em 2009 foi lançado no mercado o tratamento microinvasivo, através do uso de infiltrante resinoso – resina de baixa viscosidade. Este material, comercializado pelo nome Icon®, foi produzido pela empresa DMG, da Alemanha (Golz et al., 2016). O infiltrante resinoso possui a função de barrar a difusão de substâncias para dentro da lesão porosa interrompendo a progressão da cárie dental (Altarabulsi et al., 2014). Tem a capacidade de adentrar praticamente toda a lesão de cárie, com a correlação de quanto menos profunda a lesão, melhor será o coeficiente de infiltração (Abbas et al., 2018). Este material inovador é promissor, pois preserva a estrutura dental evitando procedimentos com teor de invasividade maior como preparos cavitários extensos em lesões proximais de cárie. Podendo a expansão do seu uso levar a benefícios diversos para os pacientes, principalmente em tratamentos de lesões de mancha branca, onde é fundamental a paralisação da lesão para evitar a progressão da doença cárie (Prasada K. et al., 2018; El Meligy et al., 2020).

Com a técnica infiltrativa, cria-se uma barreira para bloquear a difusão de ácidos cariogênicos para dentro da lesão. É interessante ressaltar que esta barreira ocorre dentro do tecido lesionado e não na sua superfície, facilitando a aplicação do infiltrante resinoso (Phark et al., 2009). Para realizar a técnica de aplicação do infiltrante resinoso, o condicionamento ácido com ácido clorídrico antes de aplicar o material é um passo importante, pois a penetração do infiltrante resinoso é impulsionada pelas forças capilares, e a camada superficial do esmalte retém baixo volume de poros dos capilares. Assim, após o ataque ácido, a porosidade da superfície aumenta, gerando um potencial de penetração maior do material. O que resulta em uma melhor remineralização de lesões iniciais de cárie (Meyer-Lueckel et al., 2006).

É relevante salientar que o infiltrante resinoso tem chamado a atenção de Cirurgiões Dentistas, pois é indicado para lesões de esmalte até o terço mais externo da dentina (Faghihian et al., 2019). Com a característica de ser inicialmente um material resinoso com o objetivo de tratar lesões de cárie em áreas de difícil acesso, como no caso de regiões interproximais, é também aplicado para mascarar lesões de mancha branca em superfícies lisas e mais recentemente está sendo indicado para manchas brancas nas oclusais de fóssulas e fissuras (Kielbassa et al., 2009). Ademais, este material resinoso ajuda, também, aos pacientes não colaborativos, os quais não aderem a boas práticas de higiene bucal e possuem maior risco de cárie (Wang et al., 2021).

Ao decorrer do tempo, vários estudos (Meyer-Lueckel et al., 2016; Stenhagen et al., 2019) vêm sendo realizados com a finalidade de buscar um infiltrante com características ideias – em similiaridade ou melhores que a marca comercial existente (Icon®). Com o objetivo de obter uma menor viscosidade há o emprego de metracrilatos sem os grupos hidroxila, como no caso do bisfenol A dimetacrilato etoxilado (BisEMA), o qual possui também menor massa molecular do que outros metacrilatos como o monômero bisfenol A glicidil dimetacrilato (BisGMA) (Gonçalves et al., 2008). Como diluente para o bisfenol A dimetacrilato etoxilado (BisEMA), ocorre a utilização do monômero trietilenoglicol dimetacrilato (TEGDMA), conforme a sua maior fluidez e baixa massa molecular (Asmussen e Peutzfeldt, 2001).

O estudo de Diolosà et al. (2014) demostrou que a incorporação de substâncias como a quitosana em materiais resinosos, traz indícios de que ela atue no fortalecimento da estrutura interna dos metacrilatos, o que consequentemente poderia aumentar a resistência desses, melhorando a durabilidade das restaurações dentárias. Sendo a quitosana um polissacarídeo originado através da alcalinização de uma parte da quitina, ela pode ser extraída da carcaça de crustáceos e classificada como biodecomposta e atóxica (Elsaka e Elnaghy, 2012). A característica notória da quitosana é de que ela possui elevado espectro antimicrobiano, dificultando a formação de biofilme e, consequentemente, elevando a duração do infiltrante no meio bucal (Stenhagen et al., 2019; Flor-Ribeiro et al., 2019). Além da quitosana, o sal de iodônio apresentou alto espectro antimicrobiano no estudo realizado por Flor-Ribeiro et al., 2019.

Este sal é classificado como sal iniciador bivalente, significando que ele pode dar início a polimerização tanto pelo meio catiônico quanto pelo radicalar, através da produção de um radical arila (DeVoe et al., 1992). Deste modo, há relatos sobre a incorporação de sal de ônio em materiais resinosos, a qual pode aumentar a eficácia da polimeralização e elevar a resistência à flexão. As evidências citadas mostram que estes componentes são promissores e acarretam melhoras significativas nas propriedades dos materiais (Ogliari et al., 2007; Gonçalves, et al., 2013). Essas promissoras melhoras possuem importante papel em aumentar a resistência do infiltrante, pois em caso de lesões de mancha branca na superfície livre ou em fissuras oclusais, o polímero no material pode sofrer modificações devido aos desafios da cavidade oral, como por exemplo a abrasão da escovação mecânica, podendo aumentar a rugosidade superficial (Souza et al., 2016). Com a finalidade de uma eficaz remoção do biofilme, as partículas abrasivas do dentifrício dental aumentam o atrito e, consequentemente, acentuam o desgaste do polímero (Davis, 1980).

Diante dos desafios de obter uma viscosidade adequada para eficaz infiltração dentária e da exposição do material perante abrasão mecânica pela escovação, esse estudo possui como objetivo avaliar o comportamento mecânico do infiltrantes experimentais com a adição de diferentes concentrações do sal de iodônio e da quitosana em comparação com o infiltrante comercial Icon®. E assim, desenvolver um infiltrante com adequadas características mecânicas.

2) **ARTIGO**: COMPARISON OF CONTACT ANGLE AND SURFACE ROUGHNESS OF EXPERIMENTAL AND COMMERCIAL INFILTRANTS.

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ABSTRACT

Objectives: The aim of this study was to evaluate the physical properties of experimental infiltrants containing iodonium salt (DPI) and chitosan in comparison with the commercial Icon® infiltrant.

Material and methods: For this purpose, nine experimental infiltrants were prepared containing the monomeric base of triethylene glycol dimetracrylate (TEGDMA) 75% and bisphenol-A dimetracrylate ethoxylate (BisEMA) 25%; 0.5 mol% of camphorquinone (CQ) and 1 mol% of ethyl 4-dimethylaminobenzoate (EDAB) as a photoinitiator system and 10% of the hydroxyethyl methacrylate monomer (HEMA). Variations of chitosan concentration were added to the formulation at 0; 0.12; and 0.25% and DPI at 0; 0.5 and 1%; and the Icon® infiltrant as a commercial control group. The contact angle test (n = 5) was performed using a goniometer. In the second stage, human molars were subjected to induction of carious lesions, then infiltrated and subjected to simulated brushing on the testing machine (15.000 cycles). The surface roughness was evaluated on the rugosimeter (n = 10), in three stages (T0 - after simulation of a carious lesion, T1 - after infiltration and T2 after brushing). The contact angle data were analyzed using one-way ANOVA with Tukey's post-hoc and roughness analysis using the generalized linear model for repeated measures over time, both with a 5% significance level.

Results: In the contact angle test, the groups that have the highest concentrations of iodonium salt and chitosan showed a higher result of the contact angle, differing significantly from the other groups. The group that presented the lowest result of the contact angle did not contain chitosan and DPI. After infiltration, there was a significant increase in the roughness of the groups with no iodonium salt (DPI). And after brushing simulation, groups with an association of iodonium salt and chitosan obtained greater surface roughness. The commercial infiltrant group Icon®, on the other hand, had the lowest surface roughness result.

Conclusion: It was concluded that the groups that contained 1% DPI had less surface roughness. The association of chitosan and DPI seems to result in less modification of the condition of the tooth surface after exposure to toothbrush simulation. The Icon® commercial infiltrating group, on the other hand, had a significant change in the roughness of the dental surface, compared to the other groups, its roughness was significantly lower after the

toothbrushing simulation. Thus, groups with higher concentrations of iodonium salt and chitosan obtained higher viscosity.

INTRODUCTION

Caries has its development and progression through the process of demineralization of the dental structure, which varies between cavitated and non-cavitated lesions. At the beginning of the carious lesion, the site brings with it the appearance of a white, opaque and rugged ax, its etiology being composed of multifactors such as cariogenic bacteria, fermentable carbohydrates and salivary dysfunction (Sfalcin, 2015; Silverstone et al., 1988). The treatment of caries disease most often used by dental surgeons is the removal of all infected dental tissue and, subsequently, the placement of restorative material to restore the lost part of the dental (Zhang et al., 2016). However, the traditional restorative procedure can cause the removal of healthy dental tissue, causing the weakening of the dental structure and thus, increasing the chances of fracture and decreasing the longevity of the restoration. Due to this Minimally Invasive Dentistry (MID) was appeared (Chalmers, 2006), which has the function of stopping the restorations vicious cycle, with the concept of maintaining the structure of the healthy tooth, thus increasing the dental vitality and preserving the function of the tooth within the oral environment (Frencken et al., 2012).

Thus, it is essential that the dental surgeon provide dietary guidance with advice on the frequency of ingestion of fermentable sugars and instruction on efficient oral hygiene, using fluoride toothpaste. Thus, to avoid the progression of the development of the white spot lesion (WSL) (Frencken et al., 2012). After patient orientation, it is possible to resort to several minimally invasive treatment options with options such as the use of remineralizing therapy through fluorotherapy, which is the first choice of dentists, however it does not lead to satisfactory results when applied in advanced stages disease (Paris et al., 2007). And varnishes such as CPP-ACFP (Baafif et al., 2020); in addition to the application of sealants, which form only an external mechanical obstacle on the surface of the white stain lesion (Lasfargues et al., 2013).

Due to the need to remedy some deficiencies of the materials existing in dental market, studies in the Minimally Invasive Restorative Dentistry area advanced, in 2009 the microinvasive treatment was launched on the market, through the use of infiltrant - low viscosity resin. This material marketed under the name Icon®, was produced by the company DMG, from Germany (Golz et al., 2016). The purpose of this material is to stop the diffusion of substances into the porous lesion by stopping the progression of dental caries (Altarabulsi et al., 2014). With the main characteristic of being applied at the time of early detection of caries, avoiding a subsequent need for invasive treatments (Doméjean et al., 2015).

The literature is advancing in search of the formulation of an infiltrant with ideal characteristics - similar to or better than the existing trademark (Icon®) (Meyer-Lueckel et al., 2016; Stenhagen et al., 2019). Several monomers are being studied with the goal of obtaining a lower viscosity, such as metracrylates without hydroxyl groups, as in the case of bisphenol A ethoxylated dimethacrylate (BisEMA), which also has a lower molecular weight than other methacrylates such as the bisphenol A glycidyl dimethacrylate (BisGMA) monomer (Gonçalves et al., 2008). As a diluent for bisphenol ethoxylated dimethacrylate (BisEMA), triethylene glycol dimethacrylate (TEGDMA) is used, due to its greater fluidity and low molecular weight (Asmussen and Peutzfeldt, 2001).

Advances in the composition of infiltrants are also occurring with the incorporation of some particles such as chitosan, which has a high antimicrobial spectrum, hinders the formation of biofilm and consequently increases the duration of the infiltrant in the oral environment (Stenhagen et al., 2019). In addition, there are indications that it acts in strengthening the internal structure of methacrylates (Diolosà et al., 2014), which consequently could increase their resistance, improving the durability of dental restorations (Flor-Ribeiro et al., 2019). There are also reports on the incorporation of onium salt in resinous materials, which can increase the effectiveness of the polymerization, and increase the flexural strenght. Chitosan and DPI have the objective of improving mechanical properties of the infiltrant, because in case of white spot lesions on the free surface or in occlusal cracks, the polymer in the material can undergo modifications due to the challenges of the oral cavity, such as the abrasion of mechanical brushing, which can increase surface roughness (Souza et al., 2016).

The resinous infiltrant is a promising material in dentistry, as it reduces the performance of invasive procedures and guarantees the preservation of the dental structure (Prasada K. et al., 2018). Given the obstacles to obtain an adequate viscosity, and therefore an effective dental infiltration and the challenge of exposing the material to mechanical abrasion by brushing, this study aims to evaluate the mechanical behavior of the experimental infiltrants with the addition of different concentrations of iodonium salt and chitosan compared to the commercial Icon® infiltrant. And so, develop an infiltrant with adequate mechanical characteristics.

MATERIALS AND METHODS

a) Formulation of experimental infiltrants

Table 1. Groups of experimental infiltrants according to the composition.

Samples	Composition
G1	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%).
G2	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), Chitosan (0,12%).
G3	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), Chitosan (0,25%).
G4	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), DPI (0,5%).
G5	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), Chitosan (0,12%), DPI (0,5%).
G6	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), Chitosan (0,25%), DPI (0,5%)
G7	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), DPI (1%).
G8	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), Chitosan (0,12%), DPI (1%).
G9	BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0,5%), Chitosan (0,25%), DPI (1%).
G10	Commercial resin infiltrant Icon®.

Description of acronyms used in the table: Bisphenol A polyethylene glycol dimethacrylate (BisEMA) ESSTECH, Triethylene glycol dimethacrylate (TEGDMA) - ALDRICH, monomer 2-hydroxyethylmethacrylate (HEMA) – ESSTECH, Camphoroquinone (CQ) - ALDRICH, Ethyl 4-dimethylamino benzoate (EDAB) - ALDRICH, Chitosan - ALDRICH, diphenyliodonium hexafluorophosphate salt (DPI) – ALDRICH.

b) Basic composition of experimental infiltrants

The monomeric base was composed of 75% Triethylene glycol dimethacrylate (TEGDMA, Sigma Aldrich Chemical Co., St Louis, MO, USA) and 25% Bisphenol A polyethylene glycol dimethacrylate (BisEMA, Esstech Inc., Essington, PA, USA). The diluent used was the monomer 2-hydroxyethylmethacrylate (HEMA, Esstech Inc., Essington, PA, USA) at a concentration of 10%. The photoinitiator system composed of 0.5% camphorquinone (CQ, Sigma Aldrich Chemical Co., St Louis, MO, USA) and 1% Ethyl 4-dimethylamino benzoate (EDAB, Sigma Aldrich Chemical Co., St Louis, MO, USA).

Regarding the salt added to the infiltrating base, the concentrations of 0.0%, 0.5% and 1% developed in the study by Ogliari et al. (2007) of the diphenyliodonium hexafluorophosphate salt (DPI) - in mol%. Chitosan was diluted with 0.2 g of powder of the same in 1 ml of acetic acid 1% (v / v). Then, in 99 ml of distilled water to obtain the desired proportion (0.12% and 0.25% Chitosan in weight%, following the assessments by Elsaka, 2012). The materials were weighed on a high-precision analytical balance and handled in an environment with adequate temperature and humidity and yellow light.

c) Contact angle test

The principle of this test was to evaluate the interaction of a solid surface with a given liquid (experimental infiltrant). For the analysis of the contact angle, blocks of planned human enamel were used as the base where the infiltrant was dripped. To measure the contact angle, a goniometer (Ramé hart-500f1 Succasunna, NJ, USA) was used coupled to a camera. Each group of infiltrant was dispensed into the enamel by a high-precision syringe (Ramé hart-500f1 Succasunna, NJ, USA). In total, there were 10 measurements per sample, with a time interval of 0.05 s and around 60 frames per second. At the end, the images were analyzed by DROPimage Advanced Software (Ramé hart-500f1 Succasunna, NJ, USA) and generated the results.

d) Preparation of specimens

Sixty human molars were used, after approval of the Research Ethics Committee of Piracicaba Dental School - UNICAMP (protocol 23612619.0.0000.5418). Cleaning was performed by manual scraping with a periodontal curette to remove organic debris and prophylaxis with rubber cups, pumice paste (Maquira Dental Products, Maringá, PR, Brazil) and water. The clean teeth were stored in a solution of distilled water and Timol at 0.1% under refrigeration. In the final stage, the section of the dental elements of the crown-root occurred at 2 mm from the cementum-enamel junction with the use of a diamond cutting disc (Extec Dia. Wafer Blade 102 x 0.3 x 12.7 mm) coupled in a Metallographic Cutter (Isomet 1000, Buehler Ltd. Lake Buff, IL, USA). The coronary part of the tooth was fixed on an acrylic plate with hot glue. Then, the tooth blocks were obtained through a diamond cutting disk (Extec Dia. Wafer Blade 102 x 0.3 x 12.7mm) coupled to a Metalographic Cutter (Isomet 1000, Buehler Ltda. Lake Buff, IL, USA). These blocks (n = 100) were obtained from the enamel portion of the free, buccal and lingual / palatal faces, with a dimension of approximately 5 x 5 x 2 mm. And the standardization of the surfaces was carried out through the planning in polishing machine (Aropol E, Arotec S / A Indústria e Comércio, Cotia - SP) with 600, 1200 and 2400 granulation sandpaper (Buehler Worldwide Headquarters 41 Waukegan Rd. P.O. Box 1Lake Bluff, IL 60044-1699, USA) under refrigeration. Polishing was performed with felt discs and diamond solution (1 µm; Buehler) also coupled to a rotary polisher (Aropol E, Arotec S). Between each planning and polishing step, the specimens were washed with distilled water in an ultrasonic vat (Marconi, Piracicaba, SP, Brazil) to remove any debris present on the enamel surface. At the end, they were stored in ependorfs with distilled water and taken to the oven at 98,6 °F.

e) Artificial simulation of dental caries

The principle of this simulation was to induce the formation of artificial caries lesions to perform the roughness test. To perform the carious lesion, a demineralization and remineralization protocol adapted from the methodology used in the study by Tem Cate and Dujisters, 1982 was used. The specimens were individually immersed in 50 mL of demineralizing solution (2.2 mmol $CaCl_2$, 2.2 mmol NaH_2PO_4, and 50 mmol of acetic acid, adjusted to pH 4.5 with NaOH), for 6 hours, in an oven at 37 ºC and then washed with distilled water and placed in remineralizing solution (1.5mM Ca, 0.9mM P, 130mM KCl, 0.02M buffer solution with pH = 7.0) for 18 hours and this protocol was done for 7 days to develop superficial enamel lesions. After this process, the specimens were washed in deionized water.

f) Infiltration of carious lesions with experimental and commercial infiltrants

The dental blocks in which they were previously induced to form a carious lesion were infiltrated with the experimental and commercial infiltrants, according to the instructions of the commercial manufacturer. For the beginning of the infiltration, the superficial layer of the enamel was removed, to allow access to the body of the lesion, by conditioning the surface of the enamel lesion with 15% hydrochloric acid (Icon Etch, DMG, Hamburg, Germany), for 120s, followed by washing the acid with water for 30s and drying with air jets, for 30s. After that, Icon-Dry (99% alcohol) was applied for 30s, and finally, application of the infiltrant on the surface of the lesion for 180s, followed by photoactivation for 40 seconds (Valo, Ultradent, Salt Lake City, USA) with 1000mW / cm^2 of irradiance. The manufacturer indicates to do a reapplication of 60 seconds, without applying the air jet, and a new photoactivation for 40 seconds, and so it was done.

g) Surface roughness test

The principle of this test was to assess the surface roughness of the resinous infiltrant (n = 10). The samples were measured with a contact surface rugosimeter (Surr-corder SE 1200-Kosaka Lab, Tokyo, Japan) over a measurement range of 4.0 mm at a speed of 3.0 mm / s with a cut-off value of 0.8 mm.

h) Toothbrushing simulation

In the brushing test, the Oral B-30 toothbrush, with an active tip with a conventional shape, was used. In a brushing machine (Toothbrush Simulation / Abrasion 4 - Top, Odeme, Luzerna, SC, Brazil) the samples were subjected to brushing with a slurry (1: 3) of deionized water and Colgate Total 12 Clean Mint toothpaste (Colgate ®- Palmolive Company, São Paulo, SP, Brazil), with a mass of 8 grams of toothpaste being stirred in the magnetic stirrer, for 24 ml of deionized water, for each sample.

The specimens were fixed in the central hole (8 mm in diameter) of the plexiglass plate (47 mm long x 20 mm wide and 73 mm wide x 2 mm thick) with hot glue and were adapted in the sample holder device, located at the bottom of the specimen brushing machine container. The brush was positioned and fixed in the brush holder device of the brushing device, with the bristles perpendicular to the central hole. The simulation occurred from linear brushing movements, with a speed of 83 movements per minute, for 3 hours, totaling 15,000 cycles per dental sample.

i) Statistical analysis

For the data of the contact angle test, SPSS 2.0 software (IBM SPSS software, IBM Analytics) was used. Statistical analysis showed normality in the data (Kolmogorov-Smirnov and Shapiro-Wilk), and the *one-way* ANOVA parametric statistical test was applied, with Tukey's post-hoc, considering the significance level of 5%. For the roughness test, descriptive and exploratory data analyzes were performed. As the data presented an asymmetric distribution, a generalized linear model for repeated measures over time was used in the analysis. The analyzes were performed in the R Core Team* program, considering the significance level of 5%.

* R Core Team (2020). A: A language and environment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria.

RESULTS

1. Contact Angle

Table 2 shows the results of the contact angle test. There was a statistical difference ($p < 0.05$) between the groups. The G9 group, which has the highest concentrations of iodonium salt and chitosan, showed the greatest result of the contact angle, differing significantly from groups G1, G2, G3, G8 and G10. The group that presented the lowest result of the contact angle was G1, differing significantly from groups G5, G7 and G9 ($p < 0.05$).

Table 2: Mean and standard deviation of the contact angle

Group	Contact angle
G1	46.24 (6.2) d
G2	48.52 (2.7) cd
G3	51.27 (6.2) bcd
G4	53.88 (8.0) abcd
G5	60.53 (3.3) ab
G6	55.68 (3.4) abcd
G7	56.38 (4.3) abc
G8	50.12 (5.1) cd
G9	63.37 (2.4) a
G10	49.54 (2.1) cd

Average values followed by the same letters do not differ statistically ($p > 0.05$) ($\alpha = 5\%$). Lower case letters compare different treatment groups (lines).

2. Surface roughness:

Table 3 and Figure 1 show the results of the roughness analysis. There was a significant interaction between the study factors group and time ($p < 0.05$). In the initial time, the G6 group showed greater roughness, differing significantly from the G1, G2, G3, G5 and G10 groups.

Still in the initial time, groups G1 and G5 showed less roughness, differing significantly from groups G4, G6 and G9 ($p < 0.05$).

After infiltration, a significant increase in the roughness of all groups was observed ($p < 0.05$). At that time, the G3 group showed greater roughness than the other groups and the groups G1, G2, G4, G5 and G6 showed greater roughness than the groups G7, G8, G9 and G10 ($p < 0.05$). Among the groups that had the lowest reading of roughness, the G10 group was the one that expressed the lowest surface roughness.

So that, after brushing simulation, there was a significant decrease in the roughness of groups G3 and G4 ($p < 0.05$). In this final time, the G10 group had a significantly lower roughness than the others ($p < 0.05$) and there was no significant difference between the other groups ($p > 0.05$). The surface roughness of the G2, G5 and G8 groups ($p < 0.05$) did not show any significant change in the result when compared to the reading before the brushing simulation.

Table 3. Mean (standard deviation), median (minimum and maximum value) of roughness as a function of group and time.

Group	Time					
	Initial		After infiltration		After infiltration and brushing	
	Mean (standard deviation)	Median (minimum and maximum value)	Mean (standard deviation)	Median (minimum and maximum value)	Mean (standard deviation)	Median (minimum and maximum value)
G1	0,2683 (0,0315)	0,2724 (0,1957; 0,3063) Bc	1,0042 (0,1850)	0,9767 (0,7887; 1,3173) Ab	0,8673 (0,1918)	0,8615 (0,4853; 1,1130) Aa
G2	0,3041 (0,0176)	0,3047 (0,2727; 0,3233) Bbc	0,9771 (0,2048)	0,9324 (0,7400; 1,2893) Ab	0,9835 (0,2080)	0,9778 (0,5653; 1,3160) Aa
G3	0,3038 (0,0470)	0,3185 (0,2040; 0,3450) Cbc	1,2384 (0,2684)	1,3092 (0,8027; 1,5890) Aa	0,9715 (0,2634)	0,9567 (0,6020; 1,3373) Ba
G4	0,3284 (0,0574)	0,3489 (0,1937; 0,3833) Cab	0,9821 (0,2277)	1,0187 (0,6447; 1,2597) Ab	0,8021 (0,1523)	0,7885 (0,5443; 1,0537) Ba
G5	0,2767 (0,0330)	0,2704 (0,2357; 0,3360) Bc	1,0388 (0,2831)	1,0187 (0,7087; 1,5353) Ab	1,0152 (0,2593)	0,9607 (0,7327; 1,4563) Aa
G6	0,3424 (0,0211)	0,3525 (0,3123; 0,3653) Ba	1,0286 (0,3822)	0,9434 (0,4680; 1,8750) Ab	0,9070 (0,2756)	0,8875 (0,5830; 1,3933) Aa
G7	0,3046 (0,0716)	0,3422 (0,1960; 0,3843) Babc	0,8467 (0,3851)	0,6027 (0,5183; 1,5227) Ac	0,9813 (0,3329)	1,0332 (0,5177; 1,4643) Aa
G8	0,3335 (0,0265)	0,3404 (0,2877; 0,3737) Babc	0,7153 (0,1713)	0,6928 (0,4840; 0,9943) Ac	0,7632 (0,2908)	0,6764 (0,4630; 1,2993) Aa
G9	0,3301 (0,0527)	0,3465 (0,2320; 0,3870) Bab	0,7353 (0,3158)	0,5719 (0,4420; 1,2527) Ac	0,8938 (0,3588)	0,8760 (0,4803; 1,3097) Aa
G10	0,2971 (0,0467)	0,2885 (0,2290; 0,3630) Bbc	0,6734 (0,3579)	0,5112 (0,3327; 1,2147) Ac	0,5653 (0,1679)	0,5115 (0,3907; 0,9310) Ab

Different letters (uppercase in the horizontal and lowercase in the vertical) indicate statistically significant differences ($p \leq 0.05$). p (group) = 0.0212; p (time) = <0.0001; p (interaction) <0.0001. G1: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%); G2: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.12%); G3: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.25%); G4: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), DFI (0.5%); G5: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.12%), DFI (0.5%); G6: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.25%), DFI (0.5%). G7: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), DFI (1%); G8: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.12%), DFI (1%); G9: BisEma (25%), TEGDMA.

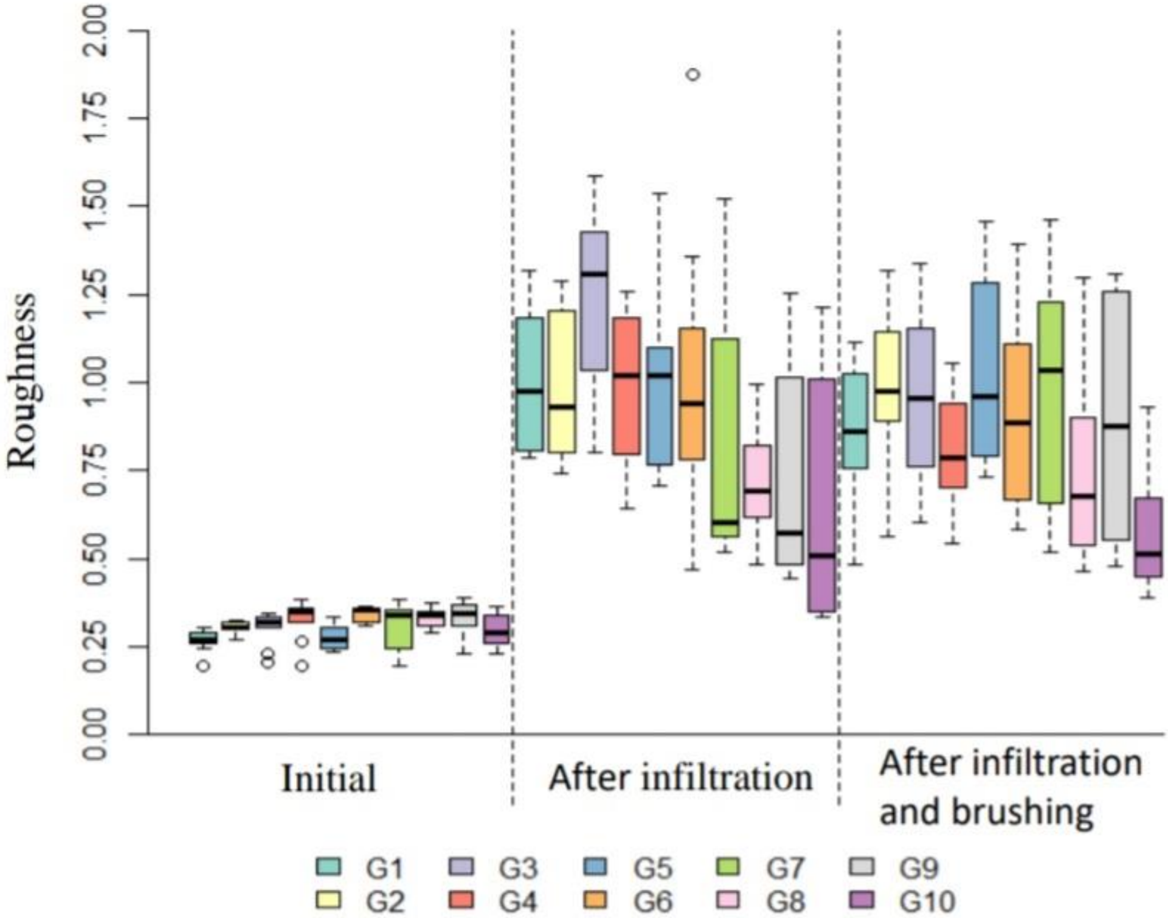

Figure 1. Box plot of roughness as a function of group and time. G1: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%); G2: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.12%); G3: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.25%); G4: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), DFI (0.5%); G5: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.12%), DFI (0.5%); G6: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.25%), DFI (0.5%). G7: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), DFI (1%); G8: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.12%), DFI (1%); G9: BisEma (25%), TEGDMA (75%), HEMA (10%), EDAB (1%), Camphorquinone (0.5%), Chitosan (0.25%), DFI (1%) and G10: Commercial resinous infiltrant ICON®.

DISCUSSION

The aim of this study was to evaluate the mechanical properties of experimental infiltrants with the addition of DPI and chitosan particles, to evaluate changes in the formulation of infiltrants, in comparison with the commercial infiltrant Icon®. It is Known that an angle less than 90 ° indicates greater wettability, that is, greater hydrophilicity while contact angles greater than 90 ° indicate greater hydrophobicity, that is, less ability to penetration of the material in the substrate (Menees et al., 2015). Thus, resins with smaller contact angles are more likely to penetrate the porous enamel. In this study, the higher the concentration of DPI and chitosan added, the greater the contact angle, resulting in a possible decrease in penetration of the infiltrant. This finding corroborates the study by Mati-Baouche N et al (2019) and Desbrières (2004) who also mentioned the increase in viscosity when added higher concentrations of chitosan due to intermolecular hydrophobic interactions that play an important role in the gelation process and, with this, it confers a change in the physicochemical (rheological) aspect. It is important to emphasize positively that in this study, all groups formulated and evaluated, obtained the value of contact angle below 90 °, indicating hydrophilic characteristics of both commercial and experimental material.

The Icon® commercial control showed a smaller contact angle, demonstrating its high capacity to penetrate the carious surface. Corroborating the study by Gaglianone et al., 2020 in which Icon® obtained the lowest result of contact angle, when evaluated at room temperature. The explanation for this result is possible because the base component of the commercial infiltrant is the monomer TEGDMA, which has a high fluidity index and low molecular weight (Ulrich et al., 2015).

Regarding roughness, at T0 (after the simulation of a carious lesion) was obtained a statistical difference in some groups, despite the standardization in the planning and polishing of the samples, in addition to the use of randomization to define the groups. This statistical difference may have occurred due to the large amount of dental fragments (n = 100), also given the difference of each human substrate. At T1 (after infiltration), there was an increase in the surface roughness of all groups, with a greater result for G3. In the absence of DPI as observed in the composition of the G3 group, the surface roughness increased. It is known that the addition of DPI in resinous materials can improve their reactivity and polymerization potential (Flor-Ribeiro et al., 2019; Ogliari et al., 2007; Gonçalves et al., 2013; Augusto et al., 2017). In addition, the photoinitiator system used, camphorquinone or EDAB, may have reacted with the salt and, even if the DPI does not absorb the blue wavelength (440-485nm) of the light curing devices alone, it may react with inactive camphoroquinone radicals in the photoactivation process (Ogliari et al., 2007), which may have had an effect in reducing the roughness observed in the G7, G8 and G9 groups, where there is a higher concentration of iodonium salt of 1%. In highlight, the commercial infiltrant Icon® obtained the lowest result of surface roughness, being statistically similar to the G7, G8 and G9 groups (after infiltration and infiltration and brushing). The explanation results from the fact that most of the Icon® formulation consists of a monomer with a high fluidity index due to its low molecular weight, this monomer being TEGDMA, according to the study by Ulrich et al., 2015, causing high penetration capacity of the infiltrant to the dental substrate, thus promoting greater surface smoothness.

The literature suggests that oral hygiene procedures, such as brushing, can change the quality of the material on the dental surface (da Costa et al., 2010; Roselino et al., 2015), that is, abrasion can wear off the material leaving the most polished surface. For the simulation of oral brushing in the cavity, the method of mechanical brushing was used in our study, as it guarantees a pattern in the applied force, in the distance and in the frequency of brushing in the samples during the brushing cycles (da Costa et al., 2010; Roselino et al., 2015). 15,000 cycles were performed for a simulation compatible with 1 year of brushing, as some authors suggest that 14,600 cycles are equivalent to one year of brushing (Wang et al., 2004; Suzuki et al., 2009).

In general, after brushing simulation there was a decrease in the surface roughness of the infiltrated samples. This result may indicate a weakening of the infiltrant layer by brushing abrasion, reinforcing the possibility of the polishing action, mentioned by Wang et al. (2004), being a factor indicative of the weakening of the protective layer of caries. The groups with an association of DPI and chitosan obtained greater surface roughness after the brushing simulation, according to Gonçalves et al. (2013) the iodonium salt improves the polymerization properties, which is related to the greater durability of the infill layer. And in the groups with the concentration of Chitosan (0.12%), at the same time, it was observed that the surface roughness had a minimal or insignificant change. However, in the literature there are no studies that corroborate the hypothesis generated by the constant concentration of Chitosan.

According to the literature, the Icon® infiltrant has practically only TEGDMA in its composition, and this compound is known for its high hydrophilicity, derived from the ether bonds present in the monomer (Fróes-Salgado et al., 2015), and also to the wide versatility of its polymeric chain that allows a greater release of water in the formation of the polymer (Ito et al., 2005). So that the G10 group composed of Icon® obtained less roughness than the others. In other words, possibly the commercial resinous infiltrant was removed in mechanical brushing.

To encourage the promotion of the quality of oral health, through Minimally Invasive Dentistry, we believe in the research of the line of infiltrating materials. Therefore, more clinical and in vitro studies need to be carried out to understand the questions opened by this project and, mainly, for the rise of this class of materials in the dental market.

CONCLUSION

- High concentrations of DPI and chitosan had a negative influence on the viscosity of the infiltrant. Thus, it can be harmful for the penetration of the material on the dental surface.
- In the groups that contained 1% of DPI there is the possibility of presenting a greater smoothness on the dental surface after enamel infiltration.
- The association of chitosan and DPI in the infiltrant, could suggests making it more difficult to remove it from the tooth surface after tooth brushing.
- There is a possibility that the Icon® Commercial Infiltrant group could be more easily removed from the tooth surface after toothbrushing abrasion.

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CONCLUSÃO

- Os grupos com maiores concentrações de sal de iodônio e quitosana obtiveram maior viscosidade.

- Após a infiltração, os grupos que continham DFI a 1% apresentaram menor rugosidade superficial.

- - A associação de quitosana e DPI no infiltrante, pode sugerir dificultar sua remoção da superfície dentária após a escovação.

- O grupo Infiltrante comercial Icon® obteve significativa modificação da rugosidade da superfície dentária, em comparação com os demais grupos a sua rugosidade foi significativamente menor, após a simulação da escovação dentária.

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ANEXOS

Anexo 1 – Verificação de originalidade e prevenção de plágio

COMPORTAMENTO MECÂNICO DE INFILTRANTES EXPERIMENTAIS E COMERCIAL

RELATÓRIO DE ORIGINALIDADE

6%	6%	5%	6%
ÍNDICE DE SEMELHANÇA	FONTES DA INTERNET	PUBLICAÇÕES	DOCUMENTOS DOS ALUNOS

FONTES PRIMÁRIAS

1	scidoc.org Fonte da Internet	1%
2	Submitted to Universidade Estadual de Campinas Documento do Aluno	1%
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4	periodicos.sbu.unicamp.br Fonte da Internet	1%
5	Livia Aguilera GAGLIANONE, Carmem Silvia PFEIFER, Caroline MATHIAS, Regina Maria PUPPIN-RONTANI, Giselle Maria MARCHI. "Can composition and preheating improve infiltrant characteristics and penetrability in demineralized enamel?", Brazilian Oral Research, 2020 Publicação	1%

Anexo 2 – Comitê de Ética em Pesquisa

UNICAMP - FACULDADE DE ODONTOLOGIA DE PIRACICABA DA UNIVERSIDADE DE CAMPINAS - FOP/UNICAMP

PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Comportamento biomecânico de infiltrantes experimentais e comercial
Pesquisador: PRISCILA REGIS MATOS PEDREIRA
Área Temática:
Versão: 2
CAAE: 23612619.0.0000.5418
Instituição Proponente: Faculdade de Odontologia de Piracicaba - Unicamp
Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 3.694.787

Apresentação do Projeto:
Transcrição editada do conteúdo do registro do protocolo e dos arquivos anexados à Plataforma Brasil
TÍTULO DO PROTOCOLO EM INGLÊS: Biomechanical behavior of experimental and commercial infiltrants.
A LISTA DE PESQUISADORES citada na capa do projeto de pesquisa inclui: PRISCILA REGIS MATOS PEREIRA (Cirurgiã-dentista, Doutoranda no PPG de Clínica Odontológica da FOP-UNICAMP, Pesquisadora responsável), JULIANA MINTO BOLDIERI (Graduanda no curso de Odontologia da FOP-UNICAMP, Pesquisadora participante), GISELLE MARIA MARCHI BARON (Cirurgião-dentista, Docente na área de Dentística da FOP-UNICAMP, Pesquisadora participante), o que é confirmado na declaração dos pesquisadores e na PB.

Situação do Parecer:
Aprovado

Necessita Apreciação da CONEP:
Não

PIRACICABA, 09 de Novembro de 2019

Assinado por:
jacks jorge junior
(Coordenador(a))

Endereço: Av.Limeira 901 Caixa Postal 52
Bairro: Areião **CEP:** 13.414-903
UF: SP **Município:** PIRACICABA
Telefone: (19)2106-5349 **Fax:** (19)2106-5349 **E-mail:** cep@fop.unicamp.br

Anexo 3 – Iniciação Científica

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Declaração

Declaro para os devidos fins, que o(a) aluno(a) **Juliana Minto Boldieri**, RA 200425, foi bolsista junto ao Programa Institucional de Bolsas de Iniciação Científica do Serviço de Apoio ao Estudante - PIBIC/SAE - Unicamp, com bolsa vigente no período de 01/08/2019 a 31/08/2020, sob a orientação do(a) Prof(a). Dr(a). GISELLE MARIA MARCHI BARON (FACULDADE DE ODONTOLOGIA DE PIRACICABA - FOP, UNICAMP) para o desenvolvimento do Projeto "*Comportamento biomecânico de infiltrantes experimentais e comercial*".

Pró-Reitoria de Pesquisa, 6 de maio de 2021.

Caio Cesar Ribas
PR ASSADMINISTRATIVOS / TÉCNICO EM ADMINISTRAÇÃO

Anexo 4 – Comprovante de submissão do Artigo.

Submission Confirmation for Journal of Dentistry

Sinalizar para acompanhamento.

JD

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Sex, 14/05/2021 01:38
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Dear Mrs Boldieri,

Your submission entitled "Comparison of contact angle and surface roughness of experimental and commercial infiltrants." has been received by the Journal of Dentistry.

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Kind regards,

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