



GIOVANA SPAGNOLO ALBAMONTE DE ARAÚJO

INFLUÊNCIA DO TRATAMENTO CLAREADOR NA ALTERAÇÃO  
DE COR DE LESÕES EM ESMALTE INFILTRADAS E NA  
DEGRADAÇÃO DO INFILTRANTE

BLEACHING TREATMENT INFLUENCE ON COLOR STABILITY OF  
INFILTRATED ENAMEL CARIES AND ON INFILTRANT  
DEGRADATION

PIRACICABA

2015





Universidade Estadual de Campinas  
Faculdade de Odontologia de Piracicaba

**GIOVANA SPAGNOLO ALBAMONTE DE ARAÚJO**

INFLUÊNCIA DO TRATAMENTO CLAREADOR NA ALTERAÇÃO  
DE COR DE LESÕES EM ESMALTE INFILTRADAS E NA  
DEGRADAÇÃO DO INFILTRANTE

Tese apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para obtenção do título de Doutora em Materiais Dentários.

Orientadora: Prof<sup>ª</sup> Dr<sup>ª</sup> Regina Maria Puppini Rontani

Este exemplar corresponde à versão final da  
tese defendida pela aluna Giovana S. A. Araújo e  
orientada pela Prof<sup>ª</sup> Dr<sup>ª</sup> Regina Maria Puppini Rontani

---

Assinatura da orientadora

Piracicaba

2015

Ficha catalográfica  
Universidade Estadual de Campinas  
Biblioteca da Faculdade de Odontologia de Piracicaba  
Marilene Girello - CRB 8/6159

Ar15i Araújo, Giovana Spagnolo Albamonte de, 1986-  
Influência do tratamento clareador na alteração de cor de lesões em esmalte infiltradas e na degradação do infiltrante / Giovana Spagnolo Albamonte de Araújo. – Piracicaba, SP : [s.n.], 2015.

Orientador: Regina Maria Puppim Rontani.  
Tese (doutorado) – Universidade Estadual de Campinas, Faculdade de Odontologia de Piracicaba.

1. Clareamento dental. 2. Pigmentação. 3. Cárie dentária. I. Puppim-Rontani, Regina Maria, 1959-. II. Universidade Estadual de Campinas. Faculdade de Odontologia de Piracicaba. III. Título.

Informações para Biblioteca Digital

**Título em outro idioma:** Bleaching treatment influence on color stability of infiltrated enamel caries and on infiltrant degradation

**Palavras-chave em inglês:**

Tooth bleaching

Pigmentation

Dental caries

**Área de concentração:** Materiais Dentários

**Titulação:** Doutora em Materiais Dentários

**Banca examinadora:**

Regina Maria Puppim Rontani [Orientador]

Ana Paula Teixeira Boscaroli

Roberta Tarkany Basting Höfling

Marcelo Giannini

Andreia Bolzan de Paula

**Data de defesa:** 24-02-2015

**Programa de Pós-Graduação:** Materiais Dentários

## RESUMO

Os objetivos desta tese composta por dois capítulos foram: **Capítulo 1-** Avaliar a estabilidade de cor e efetividade de tratamentos clareadores em lesões iniciais em esmalte infiltradas com Icon<sup>®</sup>. **Capítulo 2-** Avaliar a efetividade do clareamento com um gel experimental contendo natrosol substituindo o carbopol e avaliar a degradação causada no infiltrante por estes géis. **Capítulo 1:** Foram confeccionados espécimes à partir de dentes bovinos (N=30, 5x5x3mm) e divididos em três grupos (n=10): controle, desmineralizado e infiltrado. Lesões subsuperficiais artificiais foram desenvolvidas utilizando solução para produção de cárie, exceto no grupo controle (esmalte hígido). Nos espécimes do grupo infiltrado, após o desenvolvimento das lesões foi aplicado Icon<sup>®</sup> (DMG, Hamburgo, Alemanha). Medidas iniciais de cor foram realizadas utilizando espectrofotômetro (CM-700d, Konica Minolta), utilizando o sistema CIE L\*a\*b\*. Os espécimes foram imersos em café, três vezes ao dia, durante 15 minutos, por 14 dias e em seguida armazenados em saliva artificial. Após a pigmentação, medidas de cor foram realizadas novamente. Os espécimes foram submetidos ao clareamento com gel de peróxido de carbamida 16%, por 4h diárias durante 21 dias, e medidas finais de cor foram realizadas. Para comparar medidas iniciais e finais, foi aplicado o teste-t ( $\alpha=0,05$ ). A comparação entre os grupos foi realizada por ANOVA 1 fator e teste de Tukey ( $\alpha=0,05$ ). A pigmentação com café promoveu redução significativa nos valores de L\* e aumento nos valores de a\* e b\* em todos os grupos (controle, clareado e infiltrado). O clareamento promoveu aumento nos valores de L\* e redução nos valores de a\* and b\* em todos os grupos. Não houve diferença nos valores de  $\Delta E$  entre os grupos cariados e infiltrados antes do clareamento. Após o clareamento, o grupo infiltrado apresentou os menores valores de  $\Delta E$ , similares ao controle. Pode-se concluir que o esmalte infiltrado com Icon<sup>®</sup> apresentou alteração de cor após a pigmentação, similares ao esmalte cariado. Entretanto, se ocorrer pigmentação em dentes infiltrados, o tratamento com géis clareadores pode ser realizado com sucesso. **Capítulo 2:** Foram confeccionados 50 espécimes de infiltrante (Icon<sup>®</sup>, DMG, Hamburgo, Alemanha), divididos em 5 grupos (n=10), de acordo com o gel a ser exposto: controle (G1), peróxido de carbamida 16% com natrosol (G2), peróxido de carbamida 16% com carbopol(G3), natrosol(G4), carbopol(G5). Os tratamentos foram realizados durante 21 dias, 4 horas ao dia. Medidas de dureza Knoop (HMV-2,

Shimadzu), rugosidade (Surfcorder SE1700, Kosaka), análise de cor utilizando espectrofotômetro (CM-700d, Konica Minolta) e imagens de microscopia por força atômica (EasyScan 2, Nanosurf) foram realizadas. Além disso, 50 blocos obtidos a partir de dentes bovinos foram submetidos à pigmentação com chá preto e clareamento utilizando os géis citados acima. Os resultados foram submetidos a ANOVA 1-fator para comparação entre os grupos e teste-t para comparação inicial e final de cada grupo ( $\alpha=0,05$ ). Pode ser observada redução de dureza nas amostras de Icon® após aplicação do peróxido de carbamida, independente do espessante (G2 inicial: 13,21/final:10,99 e G3 inicial:12,00/final:11,33). Comparando-se todos os grupos após o tratamento, o G4 apresentou maior valor de dureza (13,92), seguido pelo G1(12,77) e G5(12,27). Houve aumento de rugosidade após o tratamento em todos os grupos, porém não houve diferença estatística entre os grupos. Através das imagens de microscopia por força atômica pode-se observar maior irregularidades na superfície do G5. Em relação à efetividade do clareamento, os grupos G2 e G3 promoveram aumento nos valores de L\* e maiores valores de  $\Delta E$ . Os grupos G4 e G5 apresentaram valores de L\* semelhantes. Pode-se concluir que o peróxido de carbamida associado com natrosol ou carbopol é eficaz no clareamento de dentes infiltrados com Icon®, porém causam degradação superficial no infiltrante.

Palavras-Chave: Clareamento Dental, Pigmentação, Cárie Dental.

## ABSTRACT

The objectives of this thesis, accomplished on two chapters, were: **Chapter 1-** To evaluate color stability of white spot lesions with Icon<sup>®</sup> after staining and the bleaching effect in the infiltrated and stained surfaces. **Chapter 2-** to evaluate surface degradation of Icon<sup>®</sup> after application of bleaching gels containing natrosol as a thickener and their bleaching effectiveness. **Chapter 1-** Enamel–dentine specimens (N=30, 5x5x3mm, 1 mm enamel + 2 mm dentine thickness) were prepared from bovine incisors and randomly allocated into three groups (n=10): control, demineralized, infiltrated. Artificial enamel subsurface lesions were created using 50 mL of 0.05 M acetate buffer solution. Specimens were produced by the Icon<sup>®</sup> (DMG, Hamburgo, Alemanha) application in the enamel caries like-lesions. Baseline color reading were assessed using a spectrophotometer (CM-700d, Konica Minolta), and CIE L\*a\*b\* measurements of each specimen were performed using a white background. In order to simulate extrinsic dietary staining, specimens were placed into 4 mL coffee infusion, three-times daily for 15 minutes, for 14 days. After the staining procedure, color measurements were performed again. Then, bleaching procedures were performed using 16% carbamide peroxide gel, for 4h daily, for 21 days and final measure assessment was performed. To compare baseline and final measurements, t-test was used (p<0.05). The statistical comparison between groups was performed using one-way ANOVA and Tukey tests (p<0.05). Coffee staining provided a significant reduction of L\* values and increase of a\* and b\* in all groups (control, decayed and infiltrated). Bleaching procedure provided a significant increase on L\* and decrease of a\* and b\* values in all groups. There was no significant difference on  $\Delta E$  values between decayed and Infiltrated groups before bleaching, and after bleaching, infiltrated group showed the lowest  $\Delta E$  values. It can be concluded that enamel infiltrated with Icon present significant color alteration after staining, when compared to sound enamel. However, if the infiltrant discoloration occurs, bleaching treatment can be used successfully. **Chapter 2** – 50 specimens of infiltrant (Icon<sup>®</sup>, DMG, Hamburgo, Alemanha) were produced and allocated into 5 groups (n = 10) in accordance with the gel were made to be exposed: Control (G1), carbamide peroxide 16% with natrosol (G2), carbamide peroxide with carbopol (G3), natrosol (G4), carbopol (G5). Bleaching treatments were performed for 21 days, 4 hours per day. Hardness Knoop measurements (HMV-2,

Shimadzu), roughness measurements (Surfcorder SE1700, Kosaka), and atomic force microscopy images (EasyScan 2, Nanosurf) were performed after bleaching treatments. In addition, the 50 blocks obtained from bovine teeth were submitted to bleaching treatment with gels mentioned above. The results were subjected to ANOVA one-way for comparison between groups and t-test for comparing initial and final of each group ( $\alpha < 0.05$ ). Knoop Hardness decreased can be observed after application of carbamide peroxide, regardless of thickener (initial G2: 13.21 / final: 10.99 and G3 initial: 12.00 / final: 11,33). Comparing all groups after treatment, G4 showed higher hardness Knoop value (13.92), followed by G1 (12.77) and G5 (12.27). After bleaching, there was roughness increase for all groups, and there was no statistical difference between them. Atomic force microscopy images show higher surface irregularity in G5. Regarding bleaching effectiveness, G2 and G3 groups presented increase in L \* values and highest  $\Delta E$  values. G4 and G5 groups showed statistically similar L\* values. It can be concluded that carbamide peroxide with natrosol and carbamide peroxide with carbopol may cause surface degradation. Carbamide peroxide associated with natrosol or carbopol is effective in whitening teeth infiltrated with Icon®. Further studies should be performed to evaluate the leaching of components infiltrating subjected to degradation over time.

Key-words: Tooth Bleaching, Pigmentation, Dental Caries.



## SUMÁRIO

DEDICATÓRIA	viii
AGRADECIMENTOS	xi
INTRODUÇÃO	1
CAPÍTULO 1: <i>Influence of staining solution and bleaching on color stability of resin used for caries infiltration.</i>	5
CAPÍTULO 2: <i>Influence of experimental bleaching gel with natrosol on surface of resin used for infiltration.</i>	21
CONCLUSÕES GERAIS	39
REFERÊNCIAS	40
APÊNDICE	44
ANEXO	48

*Dedico este trabalho*

*Aos meus pais Alba, Aldo e meu irmão Leonardo. Sem vocês nada seria possível, e minha vida não teria sentido. Não tenho palavras para agradecer por tudo o que fizeram por mim, muitas vezes abrindo mão de seus próprios sonhos para que os meus se tornassem realidade.*

## *Agradecimentos Especiais*

*À minha orientadora Profa. Dra. Regina Maria Puppini-Rontani. Sua conduta, integridade e seus conhecimentos te transformam em um exemplo a ser seguido como educadora. Obrigada por estar sempre presente, por todo o incentivo e empenho no desenvolvimento deste trabalho.*

*À minha orientadora durante o mestrado, Profa. Dra. Roberta Caroline Bruschi Alonso por ter inicialmente despertado em mim a paixão pela pesquisa, e ter me ensinado que o trabalho árduo sempre é recompensado.*

*Minha gratidão à vocês é eterna.*

## *Agradecimentos*

*À Universidade Estadual de Campinas e à Faculdade de Odontologia de Piracicaba, bem como ao Programa de Pós-Graduação em Materiais Dentários, pela oportunidade de aprimoramento da formação acadêmica.*

*Ao Prof. Dr. Simonides Consani, Titular da Área Materiais Dentários Departamento de Odontologia Restauradora, da Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas.*

*Ao Prof. Dr. Mario Fernando de Goes, Titular da Área Materiais Dentários, Departamento de Odontologia Restauradora, da Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas.*

*Ao Prof. Dr. Lourenço Correr Sobrinho, Titular da Área Materiais Dentários, Departamento de Odontologia Restauradora, da Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas.*

*Ao Prof. Dr. Mário Alexandre Coelho Sinhoreti, Titular da Área Materiais Dentários, Departamento de Odontologia Restauradora, da Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas.*

*À Prof. Dra. Débora Alves Nunes Leite Lima por toda a colaboração, amizade e incentivo durante o desenvolvimento deste trabalho.*

*Aos Professores Dra. Ana Paula Teixeira Boscarioli, Dra. Andréia Bolzan de Paula, Dr. Marcelo Giannini e Dra. Roberta T. Basting por terem aceito participar da banca da defesa desta tese.*

*Aos funcionários da Área Materiais Dentários da Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, Selma Aparecida Barbosa Segalla e Marcos Blanco Cangiani pelo auxílio dado a todos os alunos do curso.*

*Ao funcionário do Departamento de Odontologia Infantil, da Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, Marcelo Maistro, pelos auxílios e atenção prestados.*

*À CAPES por ter cedido a bolsa de estudos, que me permitiu concluir este trabalho.*

*Aos grandes amigos de mestrado e doutorado, Andréia, Gislaine, Carlos e Fabiana pela amizade durante esta jornada.*

*Aos demais colegas que participaram diretamente de minha formação.*

## 1 Introdução

Infiltrantes são materiais resinosos de baixa viscosidade desenvolvidos para serem aplicados em lesões de cárie incipientes. Seu conceito baseia-se na obliteração dos poros do esmalte cariado através de forças capilares. (Paris *et al.*,2006). As pesquisas voltadas para odontologia minimamente invasiva apontam que esta nova técnica para deter a progressão de lesões de cárie em esmalte é promissora, pois previne a difusão de bactérias através dos poros do esmalte e impede o desenvolvimento da lesão (Kugel *et al.*,2009).

Além de impedir a progressão da lesão (Paris *et al.*,2006), estes materiais permitem restabelecer a estética perdida em dentes anteriores que apresentam lesões de mancha branca. Isso ocorre devido à penetração do infiltrante nos poros presentes no esmalte, que altera o índice de refração da luz e conseqüentemente a aparência clínica. Uma vez que o índice de refração do infiltrante é próximo ao da hidroxiapatita, este é capaz de camuflar as lesões de mancha branca (Paris & Meyer-Lueckel, 2009)

Provavelmente, o material comercialmente disponível surgiu a partir de um estudo publicado em 2007 (Paris *et al.*, 2007). Neste estudo, foram realizadas diferentes misturas de monômeros e avaliados parâmetros como viscosidade, tensão superficial, coeficiente de penetração e consistência após a polimerização. A mistura contendo TEGDMA, pequena quantidade de HEMA e etanol apresentou alto coeficiente de penetração, o que significa que o material seria capaz de penetrar nos poros da lesão inicial em esmalte rapidamente e ao mesmo tempo apresentar consistência adequada após a polimerização. Assim, surgiu no mercado o infiltrante comercial, chamado Icon®. Para confirmar a eficácia deste material em longo prazo, estudos foram realizados indicando que o infiltrante é mais eficaz em relação à paralização da lesão, comparando-se a aplicação de fluoretos ou métodos de instrução de higiene. (Martignon *et al.*, 2006; Martignon *et al.*, 2010).

O infiltrante, assim como qualquer material presente no ambiente bucal está sujeito à degradação e pigmentação (Al-Samadani, 2013). O consumo de alimentos e bebidas que contém corantes é o principal fator que leva à pigmentação destes materiais. A intensidade da pigmentação depende de fatores relacionados ao material resinoso, como

composição e volume da matriz orgânica, tipo de polímero e a quantidade de carga inorgânica presente (Schulze *et al.*, 2003). O impacto de alimentos e bebidas na pigmentação de materiais resinosos está diretamente relacionado à quantidade e frequência de ingestão destas. (Omata *et al.*, 2006).

O café e os chás são bebidas consumidas com alta frequência, capazes de pigmentar compósitos resinosos através da absorção dos pigmentos na superfície da resina composta (Awliya *et al.*, 2010;Guler *et al.*, 2005.; Anfe *et al.*, 2011). Partindo-se do princípio que estas bebidas são capazes de pigmentar as resinas compostas disponíveis no mercado que contém, em sua maioria, diferentes monômeros na matriz e a presença de partículas de carga, seu potencial para pigmentação em infiltrantes seria maior. Infiltrantes apresentam na sua composição grande quantidade de TEGDMA e ausência de partículas de carga. Alguns autores afirmam que grande quantidade de TEGDMA presente em um material resinoso, torna-o com características hidrófilas, ou seja, com maior capacidade de sorção de água (Park *et al.*, 2011; Sideridou *et al.*, 2007). Se um material é capaz de absorver água, também pode absorver outros fluidos oriundos de alimentos e bebidas com pigmentos, resultando em alteração de cor (Fontes *et al.*, 2009).

Uma das indicações do uso de infiltrantes é a aplicação em lesões de mancha branca na superfície vestibular de dentes anteriores após a remoção de aparelhos ortodônticos. O uso destes dispositivos aumenta o risco do desenvolvimento de lesões incipientes em esmalte devido ao acúmulo de biofilme ao redor dos braquetes (Ahmed *et al.*, 2011). Se nenhum tratamento for realizado, estas lesões podem progredir até a cavitação. Caso algum paciente que apresente infiltrante nas superfícies vestibulares de dentes anteriores, fizer ingestão de bebidas ou alimentos que contém pigmentos, a estética nestes locais ficaria comprometida.

Desta forma, é necessário estabelecer uma forma de reverter o processo de pigmentação nestes casos. A literatura apresenta trabalhos que indicam o polimento destas superfícies com as lesões de esmalte com infiltrante pigmentado (Paris *et al.*,2013, Borges *et al.*,2013). Entretanto, dependendo da profundidade alcançada pelo pigmento, parece ser impossível remover somente o infiltrante, uma vez que este está embricado no esmalte. O polimento poderia resultar em remoção desnecessária e excessiva de esmalte sadio e,

dependendo dos hábitos deste paciente, provavelmente seria necessário realizar polimento novamente, resultando em maior perda de esmalte. Além disso, a técnica inovadora preconizada no uso de infiltrantes tem como princípio evitar o uso de brocas.

Uma forma de reverter o processo de pigmentação seria a aplicação de agentes clareadores. Atualmente, os géis utilizados são à base de peróxido de hidrogênio e carbamida. O tratamento clareador pode ser realizado no consultório odontológico ou o próprio paciente pode fazer o clareamento em casa, com o uso de moldeiras (Oltu & Gurgan, 2000). O peróxido de carbamida é composto por peróxido de hidrogênio e uréia. A uréia por sua vez separa-se em amônia e dióxido de carbono. O hidrogênio separa-se em água e oxigênio e radicais livres, que são os responsáveis pela oxidação dos pigmentos presentes no dente (Attin *et al.*, 1997).

Estudos têm avaliado os efeitos de géis clareadores em resinas compostas (Wang *et al.*,2011, Turker *et al.*,2003, Sharafeddin & Jamalipour, 2010), e no esmalte (Basting *et al.*,2003, Rodrigues *et al.*,2005), mas a literatura apresenta resultados controversos. Alguns autores observaram alterações superficiais e redução da dureza em materiais restauradores, (Wang *et al.*,2011, Turker *et al.*,2003), enquanto outros estudos afirmam não haver alterações após o tratamento clareador (Sharafeddin & Jamalipour, 2010).

Entretanto, alguns trabalhos sugerem que estas alterações na superfície do esmalte podem não estar relacionadas aos peróxidos presentes nos géis clareadores. Estudos *in vitro* e *in situ* observaram redução na dureza do esmalte após aplicação do espessante utilizado nos géis clareadores, o carbopol (Basting *et al.*,2005, Rodrigues *et al.*,2005). O carbopol é o espessante mais utilizado nos clareadores comercialmente disponíveis. É derivado do ácido carboxílico, portanto apresenta comportamento ácido. Desta forma, alguns autores (Basting *et al.*,2005; Rodrigues *et al.*,2005) sugerem que o carbopol pode contribuir para a desmineralização da estrutura do esmalte. Estes resultados podem colocar em questão a possibilidade de o carbopol, presente nos agentes clareadores ser capaz de degradar a resina composta e em maior magnitude o infiltrante. A degradação causada pelo



carbopol poderia resultar em aumento da rugosidade superficial, manchamento, acúmulo de biofilme e redução da longevidade da restauração (Kula *et al.*, 1986).

Portanto, seria interessante o uso de outro espessante capaz de substituir o carbopol, com o intuito de reduzir a degradação de materiais resinosos e o esmalte. Uma opção seria o uso do natrosol (hidroxietilcelulose). Natrosol é um polímero não-iônico, solúvel em água e utilizado na indústria cosmética e farmacêutica.

O infiltrante está se difundindo cada vez mais na clínica odontológica à medida que sua efetividade na paralisação da progressão da lesão incipiente em esmalte é comprovada e sua eficácia se apresentado superior aos métodos tradicionais no tratamento destas lesões. Suas propriedades físicas e químicas também já foram avaliadas, e atualmente tem se estudado seu comportamento estético.

Entretanto, há poucos relatos na literatura (Paris & Meyer-Lueckel, 2009; Borges *et al.*, 2014) avaliando a pigmentação e até o presente momento não há estudos avaliando a efetividade do clareamento após a pigmentação de lesões infiltradas com Icon®. Além disso, se torna necessário o estudo da degradação causada pelos agentes clareadores nos infiltrantes.

Desta forma, esta tese, composta por dois capítulos, propôs: 1) Avaliar a estabilidade de cor e efetividade de tratamentos clareadores em lesões iniciais em esmalte com infiltrante; 2) Avaliar a degradação causada por géis clareadores, comparando-os com um gel experimental, substituindo o carbopol pelo natrosol.

---

<sup>1</sup> Esta tese foi apresentada no formato alternativo de acordo com as normas estabelecidas pela deliberação 002/06 da Comissão Central de Pós-Graduação da Universidade Estadual de Campinas.

**Capítulo 1** (Trabalho parcialmente aceito ao periódico Operative Dentistry - Anexo 1)

***Influence of staining solution and bleaching on color stability of resin used for caries infiltration***

**Abstract**

The aims of this study were: (1) to evaluate color stability of white spot lesions with Icon<sup>®</sup> after staining and (2) to evaluate the bleaching effect in the infiltrated and stained surfaces. Materials and methods: Enamel blocks (N=30, 5x5x3mm, 1 mm enamel + 2 mm dentine thickness) were prepared from bovine incisors and randomly allocated into three groups (n=10): control (sound enamel), demineralized, infiltrated. Artificial enamel subsurface lesions were created using 50 mL of 0.05 M acetate buffer solution on demineralized and infiltrated groups. Infiltrated specimens were produced by the Icon<sup>®</sup> application in the enamel caries like-lesions. Baseline color reading were assessed using a spectrophotometer (CM-700d, Konica Minolta) and CIE L\*a\*b\* measurements of each specimen were performed using a white background. In order to simulate extrinsic dietary staining, specimens were placed into 4 mL coffee infusion, three-times daily for 15 minutes, for 14 days. After the staining procedure, color measurements were performed again. Then, bleaching procedures were conducted using 16% carbamide peroxide gel, for 4h daily, for 21 days and final assessment was performed. To compare baseline and final measurements, t-test was used (p <0.05). The statistical comparison between groups was performed using one-way ANOVA and Tukey tests (p <0.05). Coffee staining provided a significant decrease of L\* values and increase of a\* and b\* in all groups (control, decayed and infiltrated). Bleaching procedure provided a significant increase on L\* and decrease of a\* and b\* values in all groups. There was no significant difference on ΔE values between decayed and infiltrated groups after staining, and after bleaching, infiltrated group showed the lowest ΔE values. Conclusion: Enamel infiltrated with Icon<sup>®</sup> present significant color alteration after staining, when compared to sound enamel. However, if the infiltrant discoloration occurs, bleaching treatment can be used successfully.

Key-Words: Bleaching, Staining, Dental Caries.

## 1 Introduction

Orthodontic treatment with fixed appliances increases the risk of developing demineralized white spot lesions. The main reason for caries development is biofilm stagnation around the brackets, mostly underneath the arch wires and between the bracket and gingival.<sup>1</sup> An established active white spot lesion has a chalky, opaque appearance, since the light is scattered mainly within the lesion body. Scattering is caused at interfaces between substances with different refractive indexes. Nontreated white spot lesions may result in progression to cavities and severe aesthetic problems.<sup>2</sup> Additionally, sometimes during remineralization of these white spot lesions, stains get incorporated into the lesion, leading to the creation of brown spots and thus increasing the aesthetic problem. Hence, the treatment of these white spot lesions should aim both to prevent caries progression and to improve aesthetics by diminishing opacity and staining.<sup>3</sup>

Lately, a new approach has been launched to arrest noncavitated enamel lesions. After erosion of the pseudo-intact surface layer with chloridric acid, low-viscosity resins (Icon<sup>®</sup>) penetrate within the lesion.<sup>4</sup> Thus, porosities of carious lesions can be occluded, and diffusion of acids and minerals is reduced. Hence, lesion progression is hampered, and caries progression is slowed down or even arrested.<sup>4</sup> A study conducted by Paris and Meyer-Lueckel<sup>5</sup> demonstrated that the infiltration technique might be an alternative to microabrasion and restorative treatment, particularly for white spot lesions of aesthetically relevant teeth, as in cases of white spot lesions in buccal surfaces.

According to the manufacturer, Icon<sup>®</sup> is a TEGDMA-based resin matrix. Some authors have suggested that the addition of TEGDMA to a restorative material could

increase water sorption,<sup>6,7</sup> decreases general mechanical properties<sup>8</sup> and hinders color stability.<sup>9</sup> Restorative material discoloration might be attributable to water sorption degree and matrix resin hydrophilicity. If resin material can absorb water, it can also absorb other fluids, resulting in color alteration.<sup>10</sup> This process may cause plasticization and softening of resin matrix and reduction of stain stability. Discoloration of tooth-colored resin-based materials may be caused by intrinsic and extrinsic factors. Extrinsic factors such as adsorption and absorption of stains may also cause discoloration.<sup>11</sup> In this way, surfaces containing infiltrant and submitted to colored solutions can be stained, since their composition contains a greater amount of TEGDMA. For infiltrated lesions (white spot lesions treated with Icon<sup>®</sup>), this could be an aesthetic problem.

To solve this problem caused by staining solutions, some recent studies reported on the polishing of infiltrated lesions and concluded that polishing increases their resistance to staining challenges.<sup>12,13</sup> However, it seems clinically impossible to remove only the infiltrant because it has penetrated the enamel. Over time, the infiltrant could stain again and the polishing procedures would need to be performed, resulting in greater wear and excessive enamel loss.

Some physical and mechanical properties of infiltrants have already been studied,<sup>14</sup> but few studies have been published regarding its aesthetic behavior.<sup>13,15</sup> Rey and others<sup>15</sup> showed that Icon<sup>®</sup> present higher staining susceptibility compared to adhesive systems. Thus, it would be necessary to develop a method to improve Icon<sup>®</sup> appearance after staining. Borges and others<sup>13</sup> suggests repolishing of the infiltrated lesions to

minimize the staining effect. However, this polishing alternative may result in unnecessary enamel wear caused by drills.

A very common procedure to reestablish aesthetical conditions is bleaching treatment. Bleaching methods have been developed, and peroxide compounds at different concentrations are used for tooth whitening procedures. Contemporary bleaching agents are typically either hydrogen peroxide or carbamide peroxide. The procedure may be performed at a dental office or by applying the agent in gel form within the confines of a custom tray by the patient himself.<sup>16</sup>

As a highly hydrophilic material, infiltrant can be stained, but its behavior in bleaching procedures is unknown. Until now, no study has assessed the staining behavior of infiltrated lesions after staining and bleaching. It is questionable whether bleaching treatment is effective after infiltrant staining. Thus, the aims of this study were: (1) to evaluate color stability of white spot lesions with Icon<sup>®</sup> after staining and (2) to evaluate the bleaching effect on these infiltrated and stained surfaces. The hypotheses tested were: 1. Coffee staining causes a color change on infiltrated enamel; 2. A bleaching procedure is effective on infiltrated enamel.

## **2. Materials and methods**

### **2.1 Specimen preparation**

Enamel–dentine specimens (N=30, 5x5x3mm, 1 mm enamel + 2 mm dentine thickness) were prepared from bovine incisors. Enamel surfaces were polished (silicon carbide paper 1200, 2400, 4000) and dentin surfaces were covered with two layers of acid-

resistant nail varnish (Colorama®, São Paulo, Brazil), leaving a 4x4 mm<sup>2</sup> exposed area. All specimens presented the same enamel thickness and, using a simple drawing, they were randomly allocated into three groups (n=10): control, demineralized, or infiltrated.

To demineralized and infiltrated groups, artificial enamel subsurface lesions on the unprotected areas were created. Specimens were stored in 50 mL of 0.05 M acetate buffer solution, considering a ratio of 2.0 mL/mm<sup>2</sup> of exposed enamel, pH 5.0, at 50% hydroxyapatite saturation, for 10 h at 37 °C.<sup>17</sup> Caries-like lesions were etched with 37% phosphoric acid gel Scotch Bond Etchant (3M ESPE St Paul, MN, USA) for 60 s<sup>18</sup>, washed with water spray, and dried for 15s. Then specimens were dried by immersion in 100% ethanol. Icon® (DMG, Hamburg, Germany) was applied on etched enamel surface using a microbrush and let sit for 3 min, according to manufacturer instructions. Specimens were light-cured for 40 s with Free Light 2 (3M/ESPE, St Paul, MN, USA) with 1000 mW/cm<sup>2</sup> irradiance, measured with a power meter (Ophir Optronics Inc, Danvers, MA, USA). Infiltrant was applied for a second time, let sit for 1 min, and then light-cured for 40 s. All specimens were immersed in artificial saliva for 24 h.

## **2.2 Color assessment**

An initial specimen color reading (baseline), before demineralization was performed using a spectrophotometer (CM-700d, Konica Minolta, Japan) in reflectance mode. The samples were placed in a sample carrier in a light cabin (GTI Mini Matcher MM1e, GTI Graphic Technology Inc., Newburgh, NY, USA) to standardize the ambient light during the measurement process, and the samples were subjected to a reading with the spectrophotometer. CIE L\*a\*b\* measurements of each specimen were performed using a

white background. The L\*a\*b\* system allows the numeric definition of a color as well as the difference between two colors using the following formula<sup>19</sup>:  $\Delta E = [(L1 - L0)^2 + (a1 - a0)^2 + (b1 - b0)^2]^{1/2}$ , using On Color QC Lite software (Konica Minolta, Japan) to generate spectral measurements as a function of wavelength for data processing and analysis.

### **2.3 Staining procedure**

In order to simulate extrinsic dietary staining, specimens were placed into 4 mL coffee infusion (Tradition Nescafé, Nestlé, Araras, SP, Brazil) of 8 g (powder): 100 mL boiled water). After preparation, the specimens were immediately placed in contact with the coffee. This procedure was repeated for 15 min 3 times daily for 14 days. After the staining procedure, color measurements were performed again.

### **2.4 Bleaching procedure**

For the bleaching procedure, specimens were fixed in a device, and approximately 1mm of the bleaching agent was applied to the exposed enamel surface. The 16% carbamide peroxide gel (Whiteness Perfect – FGM, Santa Catarina, Brazil) was applied on enamel surfaces 4 hour daily, for 21 days.

The samples were maintained in relative humidity until the final color assessment (after 21 days), which was performed following the procedure of the initial color assessment. L\*, a\*, b\* and  $\Delta E$  were subjected to statistical analysis. To compare baseline and final measurements, a *t* test was used ( $p < 0.05$ ). The statistical comparison between groups was performed using a one-way ANOVA and Tukey test ( $p < 0.05$ ). Statistical analyses were performed by Assistat software (Campina Grande, Brazil).

### 3. Results

#### 3.1 Comparison between baseline and after staining

Table 1 shows L\*, a\*, and b\* mean values of the baseline and after staining procedures, before and after bleaching.

	Color parameters	Time	Control	Decayed	Icon <sup>®</sup> Infiltrated
No bleaching	L	Baseline	88.20(1.37) Ab	93.10(1.53) Aa	87.60(1.14) Ab
		After staining	75.91(3.41) Ba	60.50(3.71) Bb	62.54(1.80) Bb
	a	Baseline	-0.09(0.33) Bb	0.26(0.14) Bb	0.96(0.43) Ba
		After staining	3.42(0.91) Ab	3.08(0.69) Ab	8.45(0.64) Aa
	b	Baseline	11.78(1.77) Ba	5.07(0.68) Bb	9.33(1.13) Bc
		After staining	14.08(1.27) Aa	10.41(1.23) Aa	18.79(1.50) Aa
	Color parameters	Time	Control	Decayed	Icon <sup>®</sup> Infiltrated
Bleaching	L	Baseline	75.90(3.41) Ba	60.49(3.71) Bb	62.17(1.86) Bb
		After bleaching	87.19(2.12) Ab	91.65(2.92) Aa	88.27(1.30) Ab
	a	Baseline	3.42(0.91) Ab	3.07(0.69) Ab	8.34(0.57) Aa
		After bleaching	0.40(0.66) Bb	1.19(0.79) Ba	0.62(0.34) Bb
	b	Baseline	14.07(1.27) Aa	10.41(1.23) Aa	18.64(1.28) Aa
		After bleaching	11.54(2.72) Ba	2.48(2.33) Bb	10.18(1.63) Ba

Capital letters indicate comparison between baseline and final measurement on column. Lower case letters demonstrate comparison between groups on row. Different letters indicate statistically significant difference ( $p < 0.05$ ).

Coffee staining provided a significant reduction of L\* values for all groups (control, decayed, and infiltrated). After coffee staining, the control group presented the highest L\* values compared to the other groups. There was a significant increase for a\* and b\* values for all groups ( $p < 0.05$ ). Infiltrated enamel showed, on baseline, significantly higher values than decayed and sound enamel, concerning a\* and b\* values. Despite a significant baseline difference among all groups, concerning a\* and b\* values, all groups



showed significantly higher  $a^*$  and  $b^*$  values after coffee staining, but they didn't show any differences among each other after staining.

The bleaching procedure provided a significant increase in  $L^*$  values for all groups (control, decayed, and infiltrated). After bleaching, decayed enamel presented the highest  $L^*$  values compared to the other groups, but there was no significant difference between the control and infiltrated groups. There was a significant decrease for  $a^*$  and  $b^*$  values for all groups ( $p < 0.05$ ). After bleaching, decayed enamel showed significantly higher values than infiltrated and sound enamel concerning  $a^*$  values, and there was no significant difference between infiltrated and control groups for  $b^*$  values. The lowest  $b^*$  values were exhibited by the decayed group.

Table 2 –  $\Delta E$  values observed by all groups in all experimental periods

Groups	$\Delta E$	
	Baseline x Staining	Staining x Bleaching
Control	13.1206 (2.08) b	3.7379 (0.57) ab
Decayed	33.1940 (2.22) a	5.6464 (1.04) a
Infiltrated	41.1049 (2.96) a	2.2845 (0.38) b

Different letters indicate statistically significant differences ( $p < 0.05$ ) for independent comparisons considering baseline x staining and staining x bleaching individually.

Table 2 shows  $\Delta E$  mean values for all groups. The lowest  $\Delta E$  values were observed in the control group ( $p > 0.05$ ) when considering baseline x staining. However, after bleaching, stained groups showed the highest  $\Delta E$  for the decayed group compared to

the group infiltrated with Icon<sup>®</sup>. There was no significant difference on  $\Delta E$  values between the decayed and infiltrated groups ( $p < 0.05$ ) before bleaching, indicating that the Icon<sup>®</sup> infiltrated group underwent a significant color change similar to the decayed group. However, after bleaching, the infiltrated group showed the lowest  $\Delta E$  values.

#### **4 Discussion**

Color changes in direct restorative materials, and more specifically in resin materials, have a direct influence on aesthetics and therefore on the clinical longevity of a restoration. Enamel infiltrated by no-filler low-viscosity resin may become an aesthetic problem over time because like any resin material, it can be subject to color alteration. The extent of discoloration varies according to the patient's habits, such as oral hygiene and diet.<sup>20</sup>

Coffee was chosen as a dye testing substance in this study because it is frequently consumed. Coffee exhibits a strong potential for staining both tooth structure and resin materials.<sup>21</sup> The compatibility between the brown dye from coffee and the resin polymer chain has been suggested to facilitate the adsorption and penetration of the dye in the resin.<sup>21</sup>

This study was performed using bovine teeth instead of human teeth because Attia and others<sup>22</sup> found that bovine and human enamel substrates behave similarly in terms of staining and bleaching effect. Besides, the composition, density, and microhardness of bovine substrate are very similar to those of human enamel.<sup>23,24</sup>

Many methods are currently used to assess tooth color. Since spectrophotometers allow an objective color assessment and provide precise quantitative data<sup>25</sup>, this was the method used in the present study. The color alteration measurements were evaluated using reflectance measurements with the CIE Lab Color coordinate system. According to Dietschi and others<sup>26</sup> when the three coordinates of color dimensions are analyzed separately, L\* values, which depict object lightness, appear to be the most relevant parameter for making comparisons under experimental conditions. Also,  $\Delta E$  values were analyzed because they indicate the magnitude of color change at two different moments.<sup>12</sup>

The first hypothesis tested in this study was proved, as it was verified that after staining there was a significant L\* value decrease for all groups. According to Joiner<sup>19</sup>, the L\* value is a measurement of the lightness of an object and is quantified on a scale such that a perfect black has an L\* value of zero and a perfect reflecting diffuser has an L\* value of 100. The L\* values decreased, probably due to incorporation of the dye present in coffee into infiltrant. Besides, the sound enamel presented the highest L\* values, which were significantly different than those of decayed enamel infiltrated with Icon<sup>®</sup>. It means that infiltrated enamel is able to incorporate more dye. Clinically, this could be an aesthetic problem. In addition, coordinates a\* and b\* increased after staining procedures. The values of the coordinates a\* and b\* approached zero, indicating neutral colors (white and gray) and an increase in magnitude for more saturated or intense colors.<sup>19</sup>

These alterations can be explained by Icon's composition. Its matrix is TEGDMA based, with no filler particles.<sup>4</sup> Some authors have suggested that TEGDMA is a

monomer that presents high water sorption and has a hydrophilic behavior compared to other monomers.<sup>4,7</sup> Thus, it can be assumed that Icon<sup>®</sup> would easily absorb dyes that are present in beverages and food. The a\* and b\* coordinate changes drove alterations of  $\Delta E$  values, as sound enamel exhibited the lowest  $\Delta E$  values, and enamel infiltrated with Icon<sup>®</sup> presented  $\Delta E$  values almost three times higher.

The second hypothesis was also proved, as there were significant alterations of color coordinates and  $\Delta E$  values. After staining, all groups submitted to bleaching treatment showed increased L\* values, indicating higher lightness due to carbamide peroxide application. Moreover, decreased a\* and b\* values indicate that there was dye neutralization. Joiner<sup>19</sup> assumed that as values of coordinates a\* and b\* approach zero, they indicate neutral colors. The carbamide peroxide action is due to its own breakdown into hydrogen peroxide and urea. Urea further breaks down into ammonia and carbon dioxide, which accounts for the elevation of the intraoral pH. Hydrogen peroxide breaks down into water, oxygen, and free radicals, which result in oxidation of the pigments in teeth.<sup>27</sup>  $\Delta E$  values showed that enamel infiltrated with Icon<sup>®</sup> provided similar color alteration after bleaching when compared to sound enamel. These results indicate that it is possible to increase the brightness of enamel infiltrated with Icon, making use of polishing procedures unnecessary.

Borges and others<sup>13</sup> showed that demineralized enamel treated with resin infiltration showed significant staining when exposed to coffee and wine, supporting the results presented in this study. Borges and others<sup>13</sup> suggested that repolishing of the specimens could minimize the staining effect. However, polishing procedures may remove

unnecessary enamel structure, causing iatrogenic enamel damage. This study suggests a more conservative approach: the bleaching procedure. A bleaching procedure does not promote enamel removal, and according to the results presented in this study, after carbamide peroxide bleaching,  $\Delta E$  values of demineralized enamel treated with resin infiltration were statistically similar to those of the control group. According to CIE/Lab units, a  $\Delta E$  of less than 1 is excellent, and a  $\Delta E$  value less than 3.3 is considered clinically insignificant.<sup>28</sup>

The effects of staining and bleaching on composite resins have been reported. Villalta and others<sup>29</sup> observed that composite resins were affected by staining solutions, such as wine and coffee, after bleaching. After bleaching, discoloration was removed completely from the composite resins, probably due to superficial cleansing of the specimens, which may explain the results of this study regarding resin infiltration.

## **5 Conclusion**

Based on the results obtained on this study, it can be concluded that enamel infiltrated with Icon<sup>®</sup> presents significant color alteration after staining when compared to sound enamel. However, if infiltrant discoloration occurs, bleaching treatment can be used successfully. *In vivo* studies should be performed in order to assess the staining behavior of Icon<sup>®</sup> more accurately.

## 6 References

1. Ahmed I, Saif-ul-Haque & Nazir R (2011) Carious lesions in patients undergoing orthodontic treatment *The Journal of Pakistan Medical Association* **61(12)** 1176-79.
2. Kidd EAM & Fejerskov O (2004) What constitutes dental caries? Histopathology of carious enamel and dentin related to the action of cariogenic biofilms. *Journal of Dental Research* **83** C35–8.
3. Gugnani N, Pandit IK, Gupta M & Josan R (2012) Caries infiltration of noncavitated white spot lesions: A novel approach for immediate esthetic improvement. *Contemporary Clinical Dentistry* **3** S199-202.
4. Paris S, Meyer-Lueckel H & Kielbassa AM (2007) Resin infiltration of natural caries lesions *Journal of Dental Research* **86(7)** 662–666.
5. Paris S & Meyer-Lueckel H (2009) Masking of labial enamel white spot lesions by resin infiltration-a clinical report. *Quintessence International* **40(9)** 713-718.
6. Park J, Eslick J, Ye Q, Misra A & Spencer P (2011) The influence of chemical structure on the properties in methacrylate-based dentin adhesives. *Dental Materials* **27(11)** 1086-1093.
7. Sideridou ID, Karabela MM & Bikiaris DN (2007) Aging studies of light cured dimethacrylate-based dental resins and a resin composite in water or ethanol/water *Dental Materials* **23(9)** 1142-1149.

8. Gonçalves F, Pfeifer CC, Stansbury JW, Newman SM & Braga RR (2010) Influence of matrix composition on polymerization stress development of experimental composites *Dental Materials* **26(7)** 697-703.
9. Janda R, Roulet JF, Latta M & Ruttermann S (2007) Water sorption and solubility of contemporary resin-based filling materials *Journal of Biomedical Materials Research Part B, Applied Biomaterials* **82(2)** 545-551.
10. Fontes ST, Fernández MR, de Moura CM & Meireles SS (2009) Color stability of a nanofill composite: effect of different immersion media *Journal of Applied Oral Sciences* **17(5)** 388-391.
11. Ertas E, Guler AU, Yucel AC, Koprulu H & Guler E (2006) Color stability of resin composites after immersion in different drinks *Dental Materials Journal* **25(2)** 371-376.
12. Paris S, Schwendicke F, Keltsch J, Dörfer C & Meyer-Lueckel H (2013) Masking of white spot lesions by resin infiltration in vitro *Journal of Dentistry* **41** e28-34.
13. Borges A, Caneppele T, Luz M, Pucci C & Torres C (2014) Color stability of resin used for caries infiltration after exposure to different staining solutions *Operative Dentistry* **39(4)** 433-440.
14. Araújo GS, Sfalcin RA, Araújo TG, Alonso RC & Puppini-Rontani RM (2013) Evaluation of polymerization characteristics and penetration into enamel caries lesions of experimental infiltrants *Journal of Dentistry* **41(11)** 1014-1019.

15. Rey N, Benbachir N, Bortolotto T & Krejci I (2014) Evaluation of the staining potential of a caries infiltrant in comparison to other products *Dental Materials Journal* **33(1)** 86-91.
16. Oltu U & Gürgan S (2000) Effects of three concentrations of carbamide peroxide on the structure of enamel *Journal of Oral Rehabilitation* **27(4)** 332-340.
17. Paes Leme AF, Tabchoury CP, Zero DT & Cury JA (2003) Effect of fluoridated dentifrice and acidulated phosphate fluoride application on early artificial carious lesions *American Journal of Dentistry* **16(2)** 91-95.
18. Meyer-Lueckel H, Paris S & Kielbassa AM (2007) Surface layer erosion of natural caries lesions with phosphoric and hydrochloric acid gels in preparation for resin infiltration *Caries Research* **41(3)** 223-230.
19. Joiner, A. The bleaching of teeth: A review of the literature (2004) *Journal of Dentistry* **34(7)** 3-12.
20. Topcu FT, Sahinkesen G, Yamanel K, Erdemir U, Oktay EA & Ersahan S (2009) Influence of different drinks on the colour stability of dental resin composites *European Journal of Dentistry* **3(1)** 50-56.
21. Park JK, Kim TH, Ko CC, Garcia-Godoy F, Kim HI & Kwon YH (2010) Effect of staining solutions on discoloration of resin nanocomposites *American Journal of Dentistry* **23(1)** 39-42.



22. Attia ML, Aguiar FH, Mathias P, Ambrosano GM, Fontes CM & Liporoni PC (2009) The effect of coffee solution on tooth color during home bleaching applications *American Journal of Dentistry* **22(3)** 175-179.
23. Fonseca RB, Haiter-Neto F, Carlo HL, Soares CJ, Sinhoreti MA, Puppini Rontani RM & Correr-Sobrinho L (2008) Radiodensity and hardness of enamel and dentin of human and bovine teeth, varying bovine teeth age *Archives of Oral Biology* **53(11)** 1023-1029.
24. Mellberg JR (1992) Hard-tissue substrates for evaluation of cariogenic and anti-cariogenic activity in situ *Journal of Dental Research* **71** 913-919.
25. Lima DA, Silva AL, Aguiar FH, Liporoni PC, Munin E, Ambrosano GM & Lovadino JR (2008) In vitro assessment of the effectiveness of whitening dentifrices for the removal of extrinsic tooth stains *Brazilian Oral Research* **22(2)** 106-111.
26. Dietschi D, Benbachir N & Krejci I (2010) In vitro colorimetric evaluation of the efficacy of home bleaching and over-the-counter bleaching products *Quintessence International* **41(6)** 505-516.
27. Attin T, Kielbassa AM, Schwanenberg M & Hellwig E (1997). Effect of fluoride treatment on remineralization of bleached enamel *Journal of Oral Rehabilitation* **24(4)** 282-286.
28. Johnston WM & Kao EC (1989) Assessment of appearance match by visual observation and clinical colorimetry *Journal of Dental Research* **68(5)** 819-822.
29. Villalta P, Lu H, Okte Z, Garcia-Godoy F & Powers JM (2006) Effects of staining and bleaching on color change of dental composite resins *Journal of Prosthetic Dentistry* **95(2)** 137-142.

## CAPÍTULO 2

### *Influence of experimental bleaching gel with natrosol on surface of resin used for infiltration*

#### **Abstract**

The aims of this study were: (1) To evaluate the effectiveness of bleaching gels using natrosol as vehicle and (2) to evaluate Icon<sup>®</sup> surface changes caused by bleaching agents. Materials and methods: To evaluate effectiveness of bleaching gels, enamel blocks (N=50, 5x5x3mm, 1 mm enamel + 2 mm dentine thickness) were prepared from bovine incisors and randomly allocated into five groups (n=10) in accordance with the gel were made to be exposed: Control – no treatment (G1), Carbamide peroxide 16% with natrosol (G2), Carbamide peroxide 16% with carbopol (G3), natrosol (G4), carbopol (G5). Specimens were immersed in a black tea solution for six days at room temperature, in order to stain the enamel surface. Baseline color measurements were assessed using a spectrophotometer (CM-700d, Konica Minolta) and CIE L\*a\*b\* measurements of each specimen were performed. Bleaching treatments were performed according to groups and final color measurements were assessed. To evaluate Icon<sup>®</sup> surface changes, 50 specimens of infiltrant (Icon<sup>®</sup>, DMG, Hamburg, Germany) were produced and allocated into 5 groups (n=10) in accordance with the gel were made to be exposed: Control – no treatment (G1), Carbamide peroxide 16% with carbopol (G2), Carbamide peroxide 16% with natrosol (G3), natrosol (G4), carbopol (G5). Bleaching treatments were performed for 21 days, 4 hours per day. Hardness Knoop measurements (HNV-2, Shimadzu), roughness measurements (Surfcorder SE1700, Kosaka) and images of atomic force microscopy (EasyScan 2, Nanosurf) were performed before and after bleaching treatments. The results were subjected to ANOVA one-way for comparison between groups and t-test for comparing initial and final of each group (p <0.05). Carbamide peroxide with carbopol and natrosol promoted a significant increase of L\* values and decrease of a\* and b\* values and presented the highest ΔE values. Knoop Hardness decreased can be observed after application of carbamide peroxide, regardless of thickener (initial G2: 13.21/ final: 10.99

and G3 initial: 12.00/final: 11.33). Comparing all groups after treatment, G4 showed the highest Knoop hardness values (13.92), followed by G1 (12.77) and G5 (12.27). After bleaching, all groups presented higher roughness values, and there was no statistical difference between them. However, through the images of atomic force microscopy can be observed higher surface irregularity in G5. It can be concluded that carbamide peroxide associated with carbopol or natrosol is able to bleach staining enamel, however this gels promoted a surface degradation on Icon<sup>®</sup> surface. Further studies should be performed to evaluate the leaching of Icon<sup>®</sup> components subjected to degradation over time.

## **1 Introduction**

With the increasing demand for treatments to enhance esthetic appearance, the use of bleaching agents has become increasingly popular for whitening stained teeth. Bleaching methods have been developed and peroxide compounds at different concentrations are used for tooth whitening procedures. Contemporary bleaching agents are typically either hydrogen peroxide or carbamide peroxide. The procedure may be performed at a dental office or by applying the agent in gel form within the confines of a custom tray by the patient himself (Oltu & Gurgan, 2000).

Several studies have evaluated the effects of bleaching on restorative materials (Wang et al.,2011, Turker et al.,2003, Sharafeddin & Jamalipour, 2010), and enamel (Basting et al.,2003, Rodrigues et al.,2005), but literature presents controversial findings. Some authors have reported surface changes and decreased hardness in restorative materials after bleaching (Wang et al.,2011, Turker et al.,2003), while other studies found no changes (Sharafeddin & Jamalipour, 2010).

However, some authors suggest that such tooth surface changes may be not only related to peroxide application. In vitro and in situ studies had observed enamel hardness decrease after treatments with carbopol vehicle only, without peroxides in its composition (Basting et al.,2003). Carbopol is the vehicle agent most used in bleaching gels. It presents acid behavior and derived from carboxylic acid. This way, some authors (Basting et al.,2003; Rodrigues et al.,2005) suggest that carbopol may contribute to the

demineralization of the enamel structure. Rodrigues et al., 2005 observed that the enamel microhardness decrease after bleaching treatments with carbopol only. Basting et al 2003 found differences in enamel microhardness between placebo (carbopol group) and some of the bleaching agents evaluated at different times. Also, McCracken and Haywood (1995) found a significant decrease in microhardness in the enamel surface treated with the product containing carbopol. Those studies suggests that carbopol could act as a demineralizing agent. Thus, carbopol present in bleaching gels, could degraded restorative materials surface.

Therefore, it would be interesting to find a new vehicle to be placed in the bleaching gels in order to cause less material and enamel degradation than those containing carbopol. This degradation may causes surface roughness increase of material, staining, plaque accumulation and reduction of restoration longevity. (Kula et al., 1986). Given this biases, an alternative vehicle could be natrosol (hydroxyethylcellulose). Natrosol is a nonionic, water-soluble polymer, used in the cosmetic and pharmaceutical industry. However, there are no reports in the literature evaluating the effectiveness of bleaching gels with natrosol as a thickener, or even if this vehicle could degrade dental materials.

Recently, a commercially available material (Icon<sup>®</sup>) can be found to infiltrate and arrest initial caries lesions (Paris et al.,2007). Thus, the material would be exposed to oral environment, which may even undergo degradation from the bleaching procedure. Considering its composition (TEGDMA based resin matrix and the absence of filler particles), it is a very susceptible material to surface degradation.

Although some physical and mechanical properties have already been studied of infiltrants (Araujo et al.,2013) and the literature is wide in relation to composite resin degradation, there are no studies evaluating surface degradation of infiltrants, which are also susceptible to bleaching gels degradation. So, the aims of this study was: (1) To evaluate the effectiveness of these bleaching products using natrosol as vehicle and (2) to evaluate the surface changes caused by bleaching agents in Icon<sup>®</sup>.

## **2 Materials and methods**

### **2.1 Effectiveness of bleaching products**

#### **2.1.1 Specimen preparation**

Fifty bovine incisors were stored in a thymol solution at 0.1% after collection and disinfection. These teeth were examined under a light microscope (4x) to investigate the presence of gaps, cracks or any pigmentation that would interfere with the bleaching assessment. If some of these features were found, tooth was discarded and replaced.

The crowns were separated from their roots through a section in the dentin-enamel junction using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil) in a low-speed hand piece. The crowns were cut with a precision, slow-speed, water-cooled diamond saw (Isomet 1000, Buheler, Illinois, EUA) to obtain blocks with an area of 16mm<sup>2</sup>.

The enamel surface was flattened with silicon carbide (SiC) paper of #600 and #1200 grit under constant irrigation. Likewise, the dentine surface was abraded with SiC #600 and #1200 grit, achieving a block 3.0-mm thick (1 mm of enamel and 2 mm of dentin). Each specimen was marked with a diamond bur #1012 (KG Sorensen, Barueri, SP, Brazil) in one side of the block to standardize the sample position to assess the color measurements. The blocks were protected with varnish and the sample was immersed in a black tea solution for six days at room temperature, in order to stain the enamel surface (Sulieman, 2004).

#### **2.1.2 Staining procedure**

Tea solution was obtained by soaking 1.6 g of tea (black tea: Leão, Junior S.A., Curitiba, PR, Brazil) in 100 mL boiling water for five minutes. The solution was changed every 24 hours for six days (Lima et al., 2008). The samples were stored in artificial saliva for 15 days to stabilize the staining. To remove extrinsic stains, enamel surface was cleaned with pumice, using a polishing rubber.

#### **2.1.3 Color assessment**

The initial reading (baseline) was performed using a spectrophotometer (CM-700d, Konica Minolta, Japan). The samples were placed in a sample carrier to obtain the enamel and opposite dentin initial readings, taken in a light cabin (GTI Mini Matcher MM1e, GTI Graphic Technology Inc., Newburgh, NY, USA) to standardize the ambient light during the measurement process, and the samples were subjected to a spectrophotometer reading. The parameters L\*, a\*, and b\* color space and this system are referred to as CIEL\*a\*b\*. In the color space, L\* indicates lightness (L + = lightness and L - = darkness), the a\* coordinate represents the red/green range (a\* + = redness and a\* - = greenness) and the b\* coordinate represents for the yellow/blue range (b\* + = yellowness and b\* - = blueness). The values of the coordinates a\* and b\* approaching zero, indicating neutral colors (white and gray) and an increase in magnitude for more saturated or intense colors. The L\*a\*b\* system allows the numeric definition of a color as well as the difference between two colors using the following formula:  $\Delta E = [(L1 - L0)^2 + (a1 - a0)^2 + (b1 - b0)^2]^{1/2}$  (Joiner, 2004). The data was read by a microcomputer using On Color QC Lite software (Konica Minolta, Japan) to generate spectral measurements as a function of wavelength for data processing and analysis.

#### 2.1.4 Bleaching treatment

Specimens were allocated into five groups (n=10) according to bleaching gel, and treatment were performed as follows in table 1. After the bleaching treatment, final color measurements using spectrophotometer were performed. The bleaching gels were manipulated by Drogal (Piracicaba, São Paulo).

Table 1. Treatment groups and application mode.

	<b>Material</b>	<b>Application mode</b>
<b>G1</b>	Control (Distilled Water)	Distilled water for 21 days.
<b>G2</b>	16% Carbamide Peroxide + Natrosol	
<b>G3</b>	16% Carbamide Peroxide + Carbopol	Daily, four-hour application for 21 days
<b>G4</b>	Natrosol	
<b>G5</b>	2.2 Carbopol	

## **2.2 Surface degradation of infiltrant assessment**

### 2.2.1 Specimen preparation

In this study, the test material was Icon®, a methacrylate-based resin matrix, used to infiltrate white spot lesions. Fifty cylindrical specimens of Icon® (4mm diameter x 2mm thickness) were produced with polyvinylsiloxane molds (Aquasil LV, DentsplyDeTrey, Denver, USA). The specimens were light-cured at 1000 mW/cm<sup>2</sup> for 60 seconds using Free Light 2 (3M/ESPE, St Paul, USA) under a polyester strip (Airon, Maquira Dental Products Industry, Maringá, Brazil). Specimens were dry stored for 24 h in lightproof containers at 37°C and divided according to the treatment agents (n=10), as shown in table 1. The bleaching procedures were performed as follows in table 1.

At the end of each bleaching exposure, the treated specimens were washed under running distilled water for 1 min, and placed in fresh distilled water at 37°C until the next application. Baseline Knoop hardness measurements were made before the first exposure and repeated after bleaching procedures. After treatments, specimens were washed and individually maintained in deionized water until a new bleaching cycle.

### 2.2.2 Hardness Knoop Measurements

Hardness readings were performed using an indenter (HMV-2; Shimadzu Corp, Tokyo, Japan). For each specimen, five indentations were performed under a load of 50 g for 15 seconds. The Knoop Hardness number (KHN, kg/mm<sup>2</sup>) for each specimen was recorded as the average of five readings.

### 2.2.3 Roughness Measurements

Specimens were washed for 10min, dried and fitted to a surface roughness-measuring instrument (Surfcorder SE1700; Kosaka Corp., Tokyo, Japan). Prior to the hardness assessment, roughness analysis was performed to avoid interference in the results of the latter. Moreover, the specimens were divided in the middle, being the left side used for the roughness analysis and the right for hardness. To record roughness measurements, the needle moved at a constant speed of 0.5 mm/second with a load of 0.7 mN. The cut-off value was set at 0.25 mm to maximize filtration of surface waviness. Ra values for each specimen were taken across the diameter over a standard length of 0.25 mm. The mean surface roughness values (Ra,  $\mu\text{m}$ ) of the specimens were obtained from three successive measurements.

### 2.2.4 Atomic Force Microscopy

Topographic images (image size  $15\mu\text{m} \times 15\mu\text{m}$ ) of profilometer scratches were recorded with an atomic force microscope (easyScan 2, Nanosurf, Boston, MA, EUA) operating in tapping mode, using cantilever. The spring constant of the cantilevers ranged between 31 M/m and 71 M/m and their length was  $225\mu\text{m}$  with a resonance frequency of 160–210 kHz. Image processing was performed with Gwyddion 2.30 software.

Results were subjected to statistical analysis. To compare initial and final measurements t-test was used ( $p < 0.05$ ). The statistical comparison between groups was performed using ANOVA one-way and Tukey test ( $p < 0.05$ ).



### 3 Results

#### 3.2 Effectiveness of bleaching products

Table 2 shows the L\*, a\* and b\* means and standard deviations values before and after bleaching treatment.

	CONTROL	CARBAMIDE PEROXIDE + NATROSOL	CARBAMIDE PEROXIDE + CARBOPOL	NATROSOL	CARBOPOL	
L*	Before	81.58(5.45)Aa	83.23(2.43)Ba	80.96(6.67)Ba	83.66(4.62)Aa	83.86(4.16)Aa
	After	81.64(4.33)Ab	87.89(1.13)Aa	88.21(0.95)Aa	79.07(4.62)Ab	79.61(4.27)Ab
a*	Before	2.02(1.30)Aa	1.13(1.12)Aa	2.73(2.49)Aa	1.54(2.07)Aa	1.31(1.46)Aa
	After	1.93(1.57)Aa	-0.99(0.31)Bb	-1.30(0.43)Bb	-0.44(2.04)Bb	-0.70(1.55)Bb
b*	Before	17.07(3.29)Aa	15.31(2.90)Aa	16.97(4.66)Aa	16.40(4.29)Aa	15.46(3.56)Aa
	After	15.46(2.41)Aa	5.05(1.16)Bb	5.93(4.34)Bb	13.51(4.42)Aa	12.19(4.14)Ba

Capital letters indicate comparison among initial and final (vertical). Lower case letters demonstrate comparison among bleaching treatments (horizontal). Different letters indicate statistically significant difference ( $p < 0.05$ ). I: Initial (before the bleaching treatment), F: Final (after the bleaching treatment).

The carbamide peroxide with carbopol and natrosol promoted a significant L\* value increase, indicating increased lightness and decreased a\* and b\*, indicating proximity to neutral colors. Control, natrosol and carbopol groups showed no statistical difference in L\* values. There was decrease in a\* values for all treatments, except for the control group. Control group showed no significant change in any of the parameters studied. After treatment with bleaching gels, carbopol and control groups showed lowest L\* values.

Table 3.  $\Delta E$  means values

Groups	$\Delta E$
Control	4.17 (1.07) B
Carbamide Peroxide + Natrosol	11.64 (1.56) A
Carbamide peroxide + Carbopol	15,01 (2.03) A
Natrosol	9,37 (1.96) AB
Carbopol	7,82 (0.49) AB

Different letters indicate statistically significant difference ( $p < 0.05$ ).

$\Delta E$  values are presented in table 3. Carbamide peroxide with carbopol and natrosol promoted greater  $\Delta E$ , indicating greater variation color when comparing initial and final values. The control group showed the smallest color variation when compared to the others. Natrosol and carbopol groups showed intermediate values of  $\Delta E$ .

### 3.3 Degradation of infiltrant submitted to bleaching treatments

#### 3.2.1. Knoop Hardness measurements

Table 4. Hardness Knoop means (KHN) and standard deviations of Icon<sup>®</sup> samples before and after bleaching treatment.

	Control	Carbamide + natrosol	Carbamide + carbopol	Natrosol	Carbopol	
KHN	I	13.78(1.43)Aa	13.21(0.84)Aa	12.00(1.50)Aa	13.61(0.42)Aa	13.64(2.35)Aa
	F	12.77 (1.37)Aab	10.99(1.58)Bb	11.33(1.79)Bb	13.92(1.48)Aa	12.27(3.49)Aab

Capital letters indicate comparison among initial and final (vertical). Lower case letters demonstrate comparison among bleaching treatments (horizontal). Different letters indicate statistically significant difference ( $p < 0.05$ ). HN: Hardness Knoop (KHN), I: Before bleaching treatment, F: After bleaching treatment.

Knoop hardness values are presented in Table 4. ANOVA one-way showed no significant difference in hardness values in groups natrosol, carbopol and control (distilled water), indicating that treatment with peroxides was able to change the hardness surface of the samples. After whitening procedures, natrosol group showed higher hardness values compared to other treatments. After application, carbamide peroxide with carbopol and carbamide peroxide with natrosol presented lower hardness values.

### 3.2.2. Roughness measurements

Table 5. Roughness values (Ra) and standard deviations of Icon® samples before and after bleaching treatment.

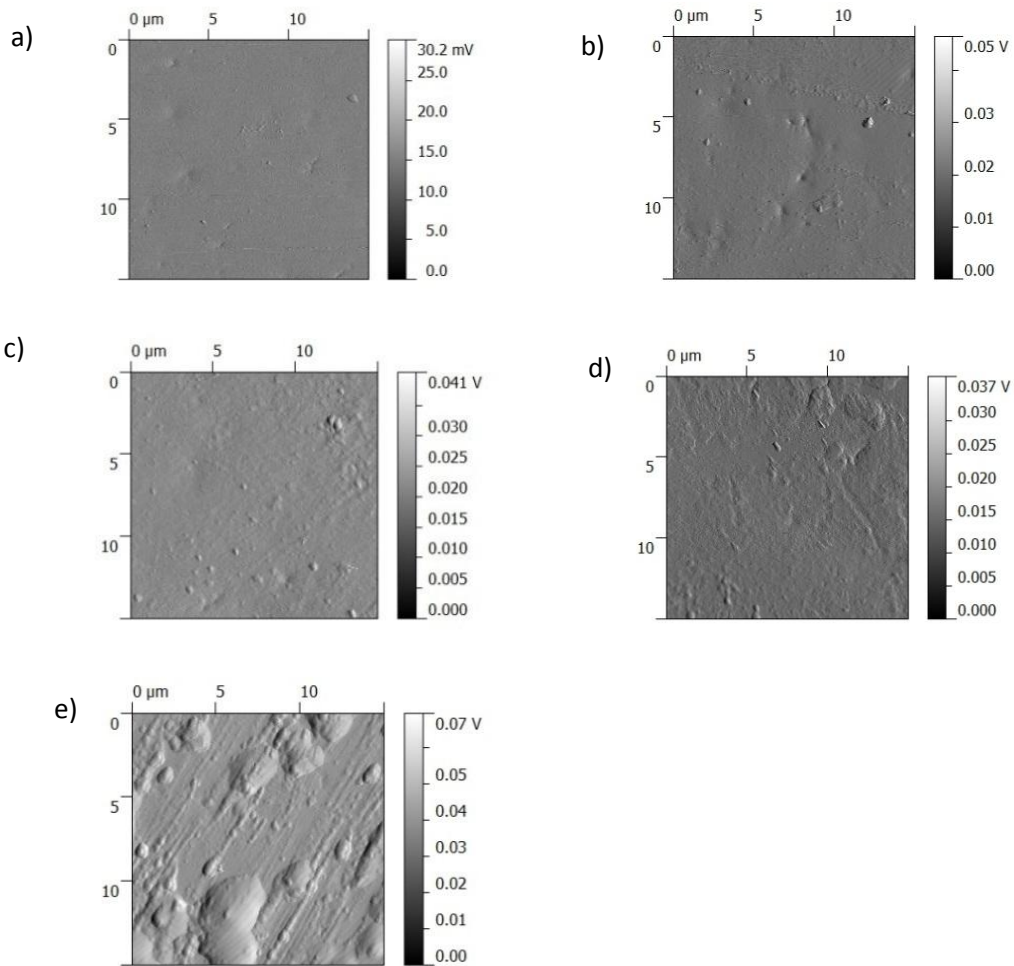
	Control	Carbamide + natrosol	Carbamide + carbopol	Natrosol	Carbopol	
Ra	I	0.1339(0.02)Ba	0.1246(0.01)Ba	0.1200(0.01)Ba	0.1236(0.02)Ba	0.1258(0.02)Ba
	F	0.1986(0.02)Aa	0.1648(0.01)Aa	0.1510(0.01)Aa	0.1944(0.01) Aa	0.1605(0.01)Aa

Capital letters indicate comparison among initial and final (vertical). Lower case letters demonstrate comparison among bleaching treatments (horizontal). Different letters indicate statistically significant difference ( $p < 0.05$ ). I: Before bleaching treatment, F: After bleaching treatment.

Roughness values (Ra) are presented in Table 5. ANOVA one-way showed no significant difference in roughness values in all groups in baseline measures. After whitening procedures, there was a roughness increase for all groups, indicating that whitening procedures promoted a surface degradation of infiltrants.

### 3.2.3 AFM Images

By analyzing the AFM images, it can be observed more irregular surface in carbopol group. The other groups (carbamide peroxide + natrosol, carbamide peroxide + carbopol and natrosol) presented surface images similar to the control group.



a) Control; b) Carbamide peroxide + natrosol, c) Carbamide peroxide+carbopol, d) Natrosol, e) Carbopol

Figure 1. AFM images of Icon<sup>®</sup> surfaces after bleaching procedures.

#### 4 Discussion

Bleaching techniques using peroxides have been used since the beginning of the 20<sup>th</sup> century to whiten non-vital and high-discolored vital teeth (Frysh et al 1995). Several studies have evaluated the effects of bleaching on dental hard tissues and dental materials. However, there are no studies evaluating the effects of bleaching on infiltrants.

Infiltrants are methacrylate-based materials used for arrest initial enamel caries lesions progression (Paris et al.,2007). This material do not contain filler particles and its matrix is TEGDMA-based. Some authors suggests that TEGDMA is a monomer that presents high water sorption (Park et al., 2011; Sideridou et al., 2007). Thus, it can be assumed that Icon<sup>®</sup> would degrade more than composite resin when in contact to bleaching gels. Composite resins present in its composition monomers with hydrophobic characteristics and filler particles, enhancing its properties.

In this study, to simulate extrinsic tooth staining and standardize the initial color of the specimens, teeth were immersed in black tea solution. This staining protocol were proposed by Sulieman et al 2003. In addition, in the present study, the color alteration measurement were evaluated through reflectance measurements with the CIE Lab Color coordinate system. According to Dietschi *et al.* (2010) when the three coordinates of color dimensions are analyzed separately, L\* values, which depict the object lightness, appeared to be the most relevant parameter to make comparisons under experimental conditions.

According to results, carbamide peroxide with carbopol and carbamide peroxide with natrosol promoted a significant L\* values increase. According to Joiner (2006) the L\* value is a measure of the object lightness and it is quantified on a scale such that a perfect black has an L\* value of zero and a perfect reflecting diffuser an L\* value of 100. It can be assumed that carbamide peroxide gel with natrosol is as efficient as commercially available gel containing carbopol as vehicle. Natrosol used as a vehicle associated with carbamide peroxide did not influence the effectiveness of bleaching treatment. Even more, it can be observed in Table 3 that carbamide peroxide with carbopol or natrosol showed similar values of  $\Delta E$ . The control, carbopol and natrosol groups showed no statistical difference in L\* values before and after treatment, suggesting that carbamide peroxide is the active ingredient responsible for the whitening.

The carbamide peroxide action mechanism is based on its breakdown into hydrogen peroxide and urea. Urea further breaks down into ammonia and carbon dioxide, which accounts for the elevation of the intraoral pH. Hydrogen peroxide breaks down into

water and oxygen and free radicals, which result in oxidation of the pigments in teeth. (Attin et al., 1997).

This study also evaluated the degradation on Icon<sup>®</sup> caused by bleaching gels. It was assessed by hardness Knoop and surface roughness test, widely used to analyse surface degradation of bleaching products (Wang et al.,2011, Turker & Biskin, 2002, De Alexandre et al.,2006). The results of this study showed that there was roughness increase in specimens submitted to bleaching with carbamide peroxide, regardless the vehicle. This results are consistent with the findings of Markovic et al.,2014 and Turker & Biskin, 2003.

After bleaching, specimens showed hardness reduction. It can be explained by the ability of peroxides to induce oxidative cleavage of the polymer chains. Thus, the double bonds of the polymer, which did not react become highly vulnerable, contributing to the decrease of hardness of the resin material (Wattanapayungkul & Yap, 2003). The significant reduction in hardness was expected, since Icon<sup>®</sup> do not have filler in composition and contain a high concentration of monomers to be oxidized by peroxides. Gurgan and Yalcin et al., 2007 demonstrated that the performance of different composites is strongly influenced by different composition, especially due to monomers. Another study (Musange and Ferracane et al.,2004) evaluating the effect of bleaching on experimental hybrid resins associated with non-silanized nanofilled showed a major susceptibility of organic matrixes to bleaching gels.

The degradation caused by the bleaching gels is dependent on the substrate where it is applied and the type and concentration of the whitening. Some authors claimed that simple changes in composition of bleaching gel, for example, differences in pH or the addition of carbopol could contribute to changes in physical properties of the restorative material (Lima et al., 2008). Studies have shown microhardness reduction of human enamel after bleaching agents and in placebo groups treated only with the thickening agent carbopol (Basting et al. 2003; Rodrigues et al. 2005).

Natrosol or carbopol only was not able to promote changes in Knoop hardness of Icon<sup>®</sup> specimens, presenting statistically similar results before and after bleaching.

However, observing the AFM images, Icon<sup>®</sup> samples treated with carbopol only, present higher surface irregularity. Results of studies suggest that changes in dental materials surface may be related to carbopol (Basting et al., 2003; Rodrigues et al., 2005). Carbopol is the most commonly agent used as a thickness agent in the bleaching gels and has an acid nature, with a carboxylic acid derivative. This surface irregularity created by carbopol may jeopardize the material longevity, since it is known that a roughness of 0.2  $\mu\text{m}$  may increase bacterial adhesion (Quirynen et al., 1996). However, this surface irregularity shown by AFM images was not able of reflecting hardness variations. Moreover, more studies should be performed to evaluate the leaching of infiltrants under degradation to assess the actual material loss over time.

When Icon<sup>®</sup> is subjected to artificial saliva (control group), peroxides and natrosol, it can be observed in AFM images a more homogeneous surface. Atomic force microscopy (AFM) is a well-established and documented tool for structural characterization of materials. It offers the opportunity to image the three-dimensional surface topography with high spatial resolution under a wide variety of conditions. This method has a higher capacity to distinguish changes in surface roughness compared to 2D profilometry, but also to reveal more detailed definition of the surface texture compared to SEM image (Kakaboura et al., 2007).

## **5 Conclusion**

Based on the results of this study, it can be stated that the carbamide peroxide associated with carbopol or natrosol is effective in bleaching treatment. Both gels caused similar surface degradation on Icon<sup>®</sup>.

## **Acknowledgements**

The authors wish to thank Drogal for providing experimental gels for this research and Prof. Mônica A. Cotta (Physics Institute, UNICAMP).

## 6 References

- 1) Oltu U, Gurgan S. Effects of three concentrations of carbamide peroxide on structure of enamel. *J Oral Rehabil.* 2000; 27(4): 332-40.
- 2) Wang L, Francisconi LF, Atta MT, Dos Santos JR, Del Padre NC, Gonini A Jr, Fernandes KB. Effect of bleaching gels on surface roughness of nanofilled composite resins. *Eur J Dent.* 2011; 5(2): 173-9.
- 3) Turker SB, Biskin T. Effect of three bleaching agents on the surface properties of three different esthetic restorative materials. *J Prosthet Dent.* 2003; 89(5): 466-73.
- 4) Sharafeddin F, Jamalipour G. Effects of 35% carbamide peroxide gel on surface roughness and hardness of composite resins. *J Dent (Tehran).* 2010; 7(1): 6-12.
- 5) Basting RT, Rodrigues AL Jr, Serra MC. The effects of seven carbamide peroxide bleaching agents on enamel microhardness over time. *J Am Dent Assoc.* 2003; 134(10): 1335-42.
- 6) Rodrigues JA, Marchi GM, Ambrosano GM, Heymann HO, Pimenta LA. Microhardness evaluation of in situ vital bleaching on human dental enamel using a novel study design. *Dent Mater.* 2005; 21(11):1059-67.
- 7) McCracken MS, Haywood VB. Effects of 10% carbamide peroxide on subsurface hardness on enamel. *Quintessence Int.* 1995; 26(1):21-4.
- 8) Kula K, Nelson S, Kula T, Thompson V. In vitro effect of acidulated phosphate fluoride gel on the surface of composites with different filler particles. *J Prosthet Dent.* 1986; 56(2):161-9.



- 9) Paris S, Meyer-Lueckel H, Cölfen H, Kielbassa AM. Resin infiltration of artificial enamel caries lesions with experimental light curing resins. *Dent Mater J.* 2007; 26(4): 582-8.
- 10) Araújo GS, Sfalcin RA, Araújo TG, Alonso RC, Puppim-Rontani RM. Evaluation of polymerization characteristics and penetration into enamel caries lesions of experimental infiltrants. *J Dent.* 2013; 41(11):1014-9.
- 11) Sueliman, M. An overview of bleaching techniques: I. History, chemistry, safety and legal aspects. *Dent Update* 2004; 31(10): 608-16.
- 12) Lima, DANL; Silva, AL; Aguiar, FHB; Liporoni, PCS; Munin, E; Ambrosano, GMB; Lovadino, JR. In vitro assessment of the effectiveness of whitening dentifrices for the removal of extrinsic tooth stains. *Braz Oral Res.* 2008; 22(2): 106-11.
- 13) Joiner, A. The bleaching of teeth: A review of the literature. *J Dent.* 2006; 34(7): 412-9.
- 14) Frysh H, Bowles WH, Baker F, Rivere-Hidalgo F, Guillen G. Effect of pH on hydrogen peroxide bleaching agents. *J Esthet Dent.* 1995; 7:130-3.
- 15) Park J, Eslick J, Ye Q, Misra A, Spencer P. The influence of chemical structure on the properties in methacrylate-based dentin adhesives. *Dent Mater.* 2011; 27(11): 1086-93.
- 16) Sideridou ID, Karabela MM, Bikiaris DN. Aging studies of light cured dimethacrylate-based dental resins and a resin composite in water or ethanol/water. *Dent Mater.* 2007; 23: 1142-9.

- 17) Sulieman M, Addy M, Rees JS. Development and evaluation of a method in vitro to study the effectiveness of tooth bleaching. *J Dent.* 2003; 31(6): 415-22.
- 18) Dietschi D, Benbachir N, Krejci I In vitro colorimetric evaluation of the efficacy of home bleaching and over-the-counter bleaching products. *Quintessence Int.* 2010; 41(6): 505-16.
- 19) Attin T, Kielbassa AM, Schwanenberg M, Hellwig E. Effect of fluoride treatment on remineralization of bleached enamel. *J Oral Rehab.* 1997; 24(4): 282-6.
- 20) Türker SB, Biskin T. The effect of bleaching agents on the microhardness of dental aesthetic restorative materials. *J Oral Rehabil.* 2002; 29(7): 657-61.
- 21) de Alexandre RS, Sundfeld RH, Briso AL, Bedran-Russo AK, Valentino TA, Sundfeld ML. Effect of 10% carbamide peroxide dental bleaching on microhardness of filled and unfilled sealant materials. *J Esthet Restor Dent.* 2006; 18(5): 273-8.
- 22) Markovic L1, Jordan RA, Glasser MC, Arnold WH, Nebel J, Tillmann W, Ostermann T, Zimmer S. Effects of bleaching agents on surface roughness of filling materials. *Dent Mater J.* 2014;33(1):59-63.
- 23) Turker SB1, Biskin T. Effect of three bleaching agents on the surface properties of three different esthetic restorative materials. *J Prosthet Dent.* 2003 May;89(5):466-73.
- 24) Wattanapayungkul P, Yap AU. Effects of in-office bleaching products on surface finish of tooth-colored restorations. *Oper Dent.* 2003; 28(1): 15-9.
- 25) Gurgan S, Yalcin F. The effect of 2 different bleaching regimens on the surface roughness and hardness of tooth-colored restorative materials. *Quintessence Int.* 2007; 38(2): e83-E87.

26) Musanje L, Ferracane JL. Effects of resin formulation and nanofiller surface treatment on the properties of experimental hybrid resin composite. *Biomaterials*. 2004; 25(18): 4065-71.

27) Lima DA, De Alexandre RS, Martins AC, Aguiar FH, Ambrosano GM, Lovadino JR. Effect of curing lights and bleaching agents on physical properties of a hybrid composite resin. *J Esthet Restor Dent* 2008; 20(4): 266-73.

28) Quirynen M, Bollen CM, Papaioannou W, Van Eldere J, van Steenberghe D. The influence of titanium abutment surface roughness on plaque accumulation and gingivitis: shortterm observations. *Int J Oral Maxillofac Implants*. 1996; 11(2): 169–78.

29) Kakaboura A, Fragouli M, Rahiotis C, Silikas N. Evaluation of surface characteristics of dental composites using profilometry scanning electron, atomic force microscopy and gloss-meter. *J Mater Sci Mater Med*. 2007; 18(1): 155-63.

## **CONCLUSÕES GERAIS**

Baseando-se nos resultados obtidos deste estudo, conclui-se que:

- 1) O esmalte infiltrado com Icon<sup>®</sup> apresenta alteração de cor significativa após a pigmentação, quando comparado com esmalte hígido. Entretanto, se ocorrer pigmentação em dentes infiltrados, o tratamento com géis clareadores pode ser realizado com sucesso.
- 2) O peróxido de carbamida associado com carbopol ou natrosol é capaz de promover clareamento dental, porém causam degradação superficial no infiltrante.

## REFERÊNCIAS BIBLIOGRÁFICAS

- 1) Paris S, Meyer-Lueckel H, Mueller J, Hummel M, Kielbassa AM. Progression of sealed initial bovine enamel lesions under demineralizing conditions in vitro. *Caries Res.* 2006; 40: 124-9.
- 2) Kugel G, Arsenault P, & Papas A. Treatment modalities for caries management, including a new resin infiltration system. *Compend Contin Educ Dent.* 2009; 30: 1-10.
- 3) Paris S, Meyer-Lueckel H. Masking of labial enamel white spot lesions by resin infiltration--a clinical report. *Quintessence Int.* 2009; 40: 713-8.
- 4) Paris S, Meyer-Lueckel H, Cölfen H, Kielbassa AM. Penetration coefficients of commercially available and experimental composites intended to infiltrate enamel carious lesions. *Dent Mater.* 2007; 23: 742-8.
- 5) Martignon S, Ekstrand KR, Ellwood R. Efficacy of sealing proximal early active lesions: an 18-month clinical study evaluated by conventional and subtraction radiography. *Caries Res.* 2006; 40: 382-8
- 6) Martignon S, Tellez M, Santamaría RM, Gomez J, Ekstrand KR. Sealing distal proximal caries lesions in first primary molars: efficacy after 2.5 years. *Caries Res.* 2010; 44: 562-70.
- 7) Al-Samadani KH J. Color stability of restorative materials in response to arabic coffee, Turkish coffee and nescafe. *Contemp Dent Pract.* 2013; 14: 681-90.

---

De acordo com as normas da UNICAMP/FOP, baseadas na padronização do International Committee of Medical Journal Editors. Abreviatura dos periódicos em conformidade com o Medline.

- 8) Schulze KA, Marshall SJ, Gansky SA, Marshall GW. Color stability and hardness in dental composites after accelerated aging. *Dent Mater.* 2003; 19: 612–9.
- 9) Omata Y, Uno S, Nakaoki Y, et al. Staining of hybrid composites with coffee, oolong tea, or red wine. *Dent Mater J.* 2006 25: 125–31.
- 10) Awliya WY, Al-Alwani DJ, Gashmer ES, Al-Mandil HB. The effect of commonly used types of coffee on surface microhardness and color stability of resin-based composite restorations. *Saudi Dent J.* 2010; 22: 177-81.
- 11) Guler AU, Yilmaz F, Kulunk T, Guler E, Kurt S. Effects of different drinks on stainability of resin composite provisional restorative materials. *J Prosthet Dent.* 2005; 94: 118-24.
- 12) Anfe TE, Agra CM, Vieira GF. Evaluation of the possibility of removing staining by repolishing composite resins submitted to artificial aging. *J Esthet Restor Dent.* 2011; 23: 260-7.
- 13) Park J, Eslick J, Ye Q, Misra A, Spencer P. The influence of chemical structure on the properties in methacrylate-based dentin adhesives. *Dent Mater.* 2011; 27: 1086-93.
- 14) Sideridou ID, Karabela MM, Bikiaris DN. Aging studies of light cured dimethacrylate-based dental resins and a resin composite in water or ethanol/water. *Dent Mater.* 2007; 23: 1142-9.
- 15) Fontes ST, Fernández MR, de Moura CM, Meireles SS. Color stability of a nanofill composite: effect of different immersion media. *J Appl Oral Sci.* 2009; 17: 388-91.

- 16) Ahmed I, Saif-ul-Haque, Nazir R. Carious lesions in patients undergoing orthodontic treatment. *J Pak Med Assoc.* 2011; 61: 1176-9.
- 17) Paris S, Schwendicke F, Keltsch J, Dörfer C, Meyer-Lueckel H. Masking of white spot lesions by resin infiltration in vitro. *J Dent.* 2013; 41: e28-34.
- 18) Borges A, Caneppele T, Luz M, Pucci C, Torres C. Color stability of resin used for caries infiltration after exposure to different staining solutions. *Oper Dent.* 2014; 39: 433-40.
- 19) Oltu U, Gürgan S. Effects of three concentrations of carbamide peroxide on the structure of enamel. *J Oral Rehabil.* 2000;27: 332-40.
- 20) Attin T, Kielbassa AM, Schwanenberg M, Hellwig E. Effect of fluoride treatment on remineralization of bleached enamel. *J Oral Rehab.* 1997; 24: 282-6
- 21) Wang L, Francisconi LF, Atta MT, Dos Santos JR, Del Padre NC, Gonini A Jr, Fernandes KB. Effect of bleaching gels on surface roughness of nanofilled composite resins. *Eur J Dent.* 2011; 5: 173-9.
- 22) Turker SB, Biskin T. Effect of three bleaching agents on the surface properties of three different esthetic restorative materials. *J Prosthet Dent.* 2003; 89: 466-73.
- 23) Sharafeddin F, Jamalipour G. Effects of 35% carbamide peroxide gel on surface roughness and hardness of composite resins. *J Dent (Tehran).* 2010; 7: 6-12.
- 24) Basting RT, Rodrigues AL Jr, Serra MC. The effects of seven carbamide peroxide bleaching agents on enamel microhardness over time. *J Am Dent Assoc.* 2003; 134: 1335-42.

- 25) Rodrigues JA, Marchi GM, Ambrosano GM, Heymann HO, Pimenta LA. Microhardness evaluation of in situ vital bleaching on human dental enamel using a novel study design. *Dent Mater.* 2005; 21: 1059-67.
- 26) Kula K, Nelson S, Kula T, Thompson V. In vitro effect of acidulated phosphate fluoride gel on the surface of composites with different filler particles. *J Prosthet Dent.* 1986; 56: 161-9.



## APÊNDICE

Neste apêndice estão relacionados os materiais e instrumentos utilizados nos estudos que compõem esta tese.

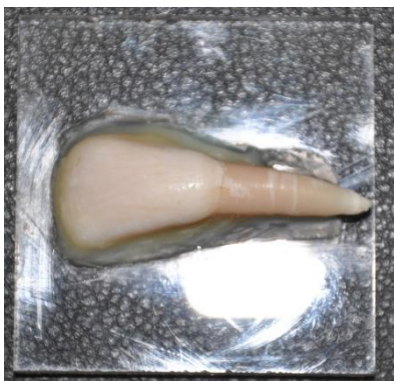


Fig. 1 – Dente bovino

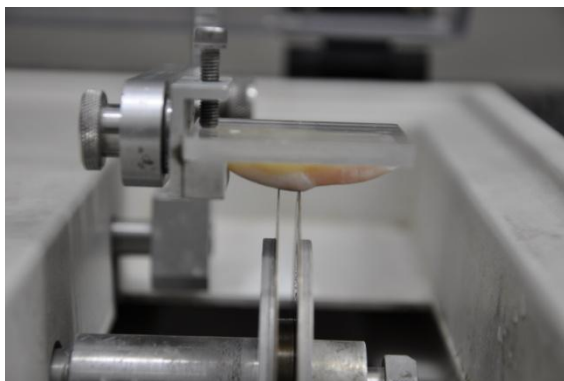


Fig. 2 – Dente bovino na cortadeira mecânica Isomet (Buehler, Illinois, EUA).



Fig. 3 – Aspecto superficial do esmalte bovino cariado



Fig. 4 – Espécime imerso na solução de cárie



Fig. 5 – Aplicação do ácido fosfórico por 60 segundos

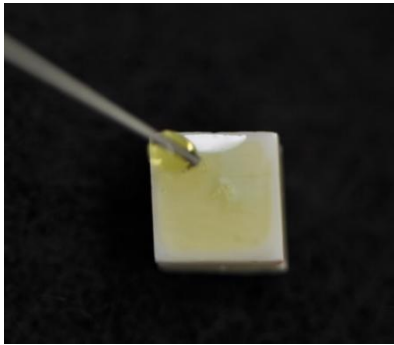


Fig. 6 – Aplicação do infiltrante



Fig. 7 – Icon (DMG)



Fig. 8 – Cabine de luz para mensuração de cor



Fig. 9 – Espectrofotômetro Konica Minolta CM-700d



Fig. 10 – Dispositivo para leitura de cor



Fig. 11 - Dispositivo para leitura de cor acoplado ao espectrofotômetro

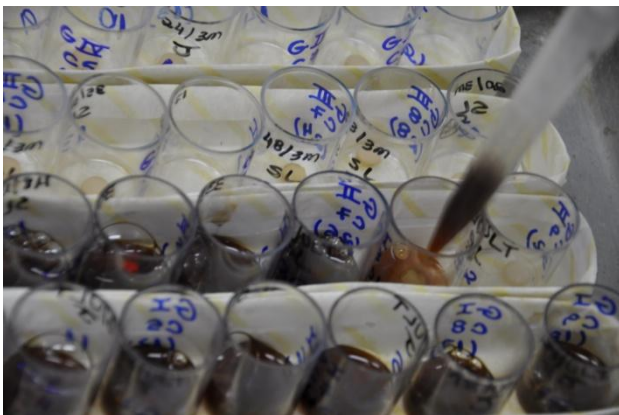


Fig.12 – Imersão dos espécimes em café

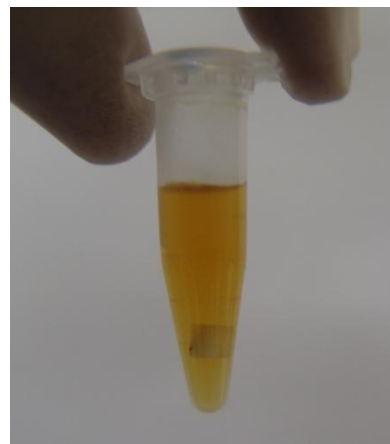


Fig. 13 – Imersão dos espécimes em chá preto



Fig. 14 – Géis clareadores manipulados



Fig. 15 – Durômetro HMV-2 Shimadzu

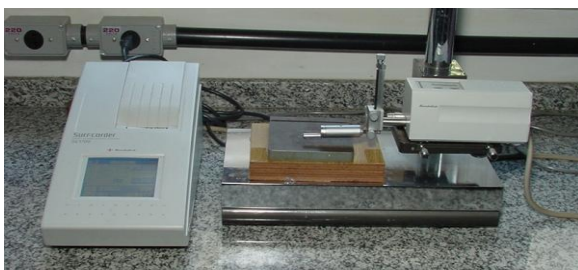


Fig. 16 – Rugosímetro (Surfcorder SE1200, Kosaka Lab.)



Fig. 17 – Microscópio Luz Polarizada (easyScan 2, Nanosurf)



Fig. 18 – Matriz de polivinilsiloxano para confecção dos espécimes de infiltrante

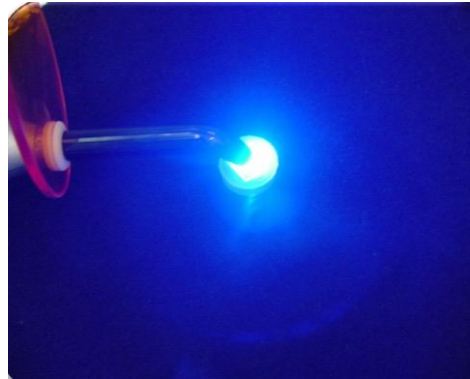


Fig. 19 – Fotoativação do infiltrante

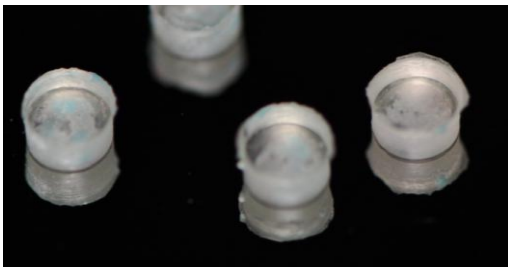


Fig. 20 – Espécimes de infiltrante

## ANEXO

Operative Dentistry Manuscript #14-290-L

↑ ↓ ×

December 18, 2014

Dear Author,

We have received the reviews for your manuscript "Influence of staining solution and bleaching on color stability of resin used for caries infiltration.". On the basis of these reviews I regret to inform you that I cannot accept this article for publication without major revisions. However, once the areas of concern have been addressed, we would invite you to resubmit the corrected manuscript for acceptance consideration.

Please review the referee's comments, you will find them below. They are written to help our colleagues write better papers and to improve their research techniques. I urge you to accept these criticisms in the spirit in which they are offered and to make the appropriate changes in your paper or research methodology. Many valid concerns were expressed and require answers before we can accurately assess your manuscript for publication potential. If you feel some changes are not justified, please defend your position without making the recommended changes. In addition, you should show proof of corrections and answer all reviewer concerns.

At the current time, Operative Dentistry receives a large number of outstanding papers. This makes it necessary for us to be extremely conservative in our acceptance of new manuscripts. Even relatively minor errors in protocols or reporting of the research can prevent acceptance of a paper.

It is obvious that much time and effort was spent in creating your manuscript. We are honored that you would submit this paper for consideration by Operative Dentistry.

We look forward to receiving your corrections for reconsideration. When you are ready to submit the corrections, please only send the latest revised form of the manuscript. Do not send any previous copies! Please do not click on this link until you have all of the requested revisions ready to submit.