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**EFEITO DE AGENTES CLAREADORES SOBRE A
SUPERFÍCIE DAS CERÂMICAS ODONTOLÓGICAS:
AVALIAÇÃO DA MICRODUREZA E RUGOSIDADE**

Guarulhos
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SUPERFÍCIE DAS CERÂMICAS ODONTOLÓGICAS:
AVALIAÇÃO DA MICRODUREZA E RUGOSIDADE**

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Orientador: Prof. Dr. José Augusto Rodrigues
Co-orientador: Prof. Dr. Cesar AG Arrais

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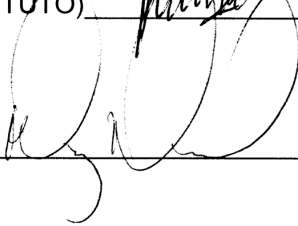
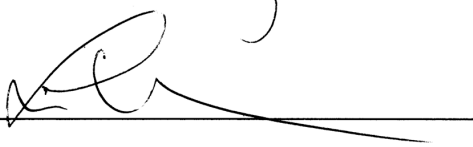

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Guarulhos, 23 de Abril de 2012.

DEDICATÓRIA

Aos meus pais, Francisco e Célia (*in memórian*) e minha tia Maria das Dores (*in memorian*), pelo amor incondicional e pelos ensinamentos sobre a difícil arte de viver com dignidade.

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**Ao mestre cabe aprender sempre, às vezes ensinar.
Ao doutor cumpre a inquietude de questionar e investigar à
exaustão, lançando luz onde descansa a sombra da ciência.**

Sérgio A. M. Ourique

RESUMO

Poucos estudos relatam o efeito de sistemas clareadores sobre as cerâmicas odontológicas ao longo do tempo. Assim, este trabalho teve como objetivo avaliar o efeito de agentes clareadores sobre cerâmicas odontológicas por meio da publicação de três artigos científicos. Corpos-de-prova foram confeccionados utilizando-se diferentes cerâmicas odontológicas. Com o uso de um microdurômetro e penetrador tipo Knoop foi avaliada a microdureza (Capítulo 1), empregando-se um rugosímetro foi determinada a rugosidade superficial das cerâmicas (Capítulo 2). Em seguida foi realizado o tratamento clareador com peróxido de carbamida 10% ou 16% por 6 horas diárias por 21 dias, tendo ainda um grupo controle que permaneceu em saliva artificial. A microdureza e a rugosidade superficial foram avaliadas antes e ao longo da aplicação do clareador, após 18h, 42h, 84h e 126h de tratamento. Na sequência estudou-se o efeito do repolimento prévio ao clareamento na rugosidade superficial das cerâmicas (Capítulo 3). Os dados foram submetidos à Análise de Variância em parcelas subdivididas, não foram observadas diferenças estatísticas significativas nos valores de microdureza ou rugosidade superficiais entre as cerâmicas. Conclui-se que a exposição de cerâmicas aos sistemas clareadores para tratamento caseiro a base de peróxido de carbamida 10% ou 16%, não causam alterações que exijam a substituição das mesmas.

Palavras-Chave: Clareamento de dente, agentes clareadores, cerâmicas odontológicas, microdureza, rugosidade superficial, repolimento.

ABSTRACT

Few studies showed the effect of bleaching systems on dental ceramics throughout time exposure. Thus, the objective of this study was to evaluate the effect of at-home bleaching agents on dental ceramics by means of the publication of three scientific articles. The specimens were manufactured using different dental ceramics. Ceramics microhardness was evaluated with a microhardness tester and a Knoop indenter (Chapter 1) and the surface roughness was determined with a profilometer (Chapter 2). After that 10% or 16% carbamide peroxide were applied for 6 hours daily per 21 days, and a control group remained in artificial saliva. The microhardness and surface roughness were evaluated before and throughout the application of the bleaching agents and after 18h, 42h, 84h and 126h of treatment. Following it was studied the effect of ceramic refinishing before dental bleaching on surface roughness (Chapter 3). The data were submitted to the split plot Analysis of Variance and no statistical significant differences in the values of superficial roughness or microhardness were observed among the groups. It can be concluded that the exposition to at-home bleaching systems based on 10% or 16% carbamide peroxide do not cause alterations on ceramics that demand polishing or replacement.

Keywords: Dental bleaching, bleaching agents, dental ceramics, microhardness, surface roughness, refinishing.

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1. Introdução

O escurecimento dental ocorre por diversos fatores, dentre eles fatores extrínsecos como deposição de pigmentos oriundos da alimentação, que são rapidamente removidos com profilaxia, assim como por fatores intrínsecos relacionados ao envelhecimento fisiológico, traumas e até mesmo adsorção de pigmentos extrínsecos (Goldstain & Garber, 1996).

Como solução mais conservativa para os dentes com escurecimento intrínseco temos o clareamento dental, destacando-se a técnica caseira descrita por Haywood & Heymann, em 1989. Por meio desta técnica resultados efetivos e duradouros são obtidos em torno de 21 dias, com o uso de uma moldeira individual e sistemas clareadores (Haywood & Heymann, 1989; Ritter et al. 2002).

Apesar de proporcionar estética ao sorriso dos pacientes, o tratamento clareador pode causar efeitos colaterais clínicos como sensibilidade e irritação gengival trans-operatória (Rodrigues et al. 2004; Montan et al. 2006); e sub-clínicos ao dente como perda de minerais, alterações da morfologia superficial, com aumento de rugosidade, maior adesão bacteriana e a redução da microdureza (Seghi & Denry, 1992; Shannon et al. 1993; Wandera et al., 1994; Gurgan et al., 1997; Oltu & Gürkan, 2000; Potocnik et al., 2000, Rodrigues et al., 2001; Türkün et al., 2002; Basting et al., 2003; Hosoya et al., 2003; Worschech et al., 2003; Rodrigues et al., 2005; Worschech et al., 2006).

Tais alterações são atribuídas ao pH dos sistemas clareadores que são relativamente baixos, e principalmente aos radicais livres formados durante a reação de clareamento, visto que devido a grande reatividade podem quebrar moléculas alterando a estrutura dental (Seghi & Denry, 1992; Shannon et al. 1993; Wandera et al., 1994; Gurgan et al., 1997; Oltu & Gürkan, 2000; Potocnik et al., 2000, Rodrigues et al., 2001; Türkün et al., 2002; Basting et al., 2003; Rodrigues et al., 2005).

Mesmo frente a estas alterações causadas pela ação do peróxido e de seus radicais livres, o clareamento dental é amplamente indicado, pois os tratamentos convencionais para a resolução da estética de dentes escurecidos envolvem o desgaste da estrutura dental para a restauração

direta com resinas compostas ou indireta com coroas ou facetas cerâmicas.

Por outro lado, os pacientes podem apresentar coroas ou facetas cerâmicas unitárias em alguns dentes confeccionadas antes do escurecimento dental, que devido a estabilidade de cor deste material podem ser conservadas após o tratamento clareador. Porém, como o tratamento clareador caseiro é realizado fora do consultório odontológico os pacientes podem, por descuido ou falta de informação, aplicar os agentes clareadores sobre tais cerâmicas e efeitos indesejados podem ocorrer sobre elas. Poucos trabalhos avaliam o efeito dos agentes clareadores sobre as cerâmicas odontológicas e os resultados são controversos, sendo relatadas alterações superficiais em microscopia eletrônica de varredura (Schemehorn et al., 2004), diminuição de microdureza (Turker & Biskin, 2002; Polydorou et al., 2007), aumento de rugosidade superficial (Moraes et al. 2006) ou mesmo na ausência dessas alterações (Silva et al. em 2006; Duschner et al., 2006; Polydorou et al., 2006).

Devido aos diferentes resultados encontrados, bem como ao pouco número de estudos realizados sobre os efeitos dos clareadores dentais em função do tempo de aplicação sobre os sistemas cerâmicos, este trabalho teve como objetivo avaliar a microdureza e a rugosidade superficial de sistemas cerâmicos após a aplicação *in vitro* de sistemas clareadores caseiros por um período similar a 3 semanas de uso clínico.

2. Proposição

Este trabalho teve como objetivo avaliar a microdureza e a rugosidade superficial de cerâmicas odontológicas submetidas ao tratamento com agentes clareadores.

3. Desenvolvimento

Capítulo 1- *Effect of different concentrations of carbamide peroxide on microhardness of dental ceramics* - Sérgio A.M. Ourique, Jovana P.S. Magdaleno, Cesar A.G. Arrais, José A. Rodrigues

Artigo publicado no periódico *American Journal of Dentistry* (Anexo 1)

Capítulo 2- *Effect of different concentrations of carbamide peroxide and bleaching periods on surface roughness of dental ceramics* - Sérgio A.M. Ourique, Claudia Ota-Tsuzuki, Cesar A.G. Arrais, José A. Rodrigues

Artigo publicado no periódico *Brazilian Oral Research* (Anexo 2)

Capítulo 3- *Surface roughness evaluation of in vitro refinished dental ceramics followed by bleaching treatment-* Sérgio Augusto Morey Ourique, Leonardo Colombo Zeidan, César Augusto Galvão Arrais, Alessandra Cassoni, José Augusto Rodrigues

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Capítulo 1- Effect of different concentrations of carbamide peroxide on microhardness of dental ceramics - Sérgio A.M. Ourique, Jovana P.S. Magdaleno, Cesar A.G. Arrais, José A. Rodrigues

Artigo publicado no periódico *American Journal of Dentistry* (Anexo 1).

Abstract: the objective of this study was to evaluate the effect of 10% and 16% carbamide peroxide bleaching agents on microhardness of dental ceramics after different periods of bleaching treatment. Fifteen specimens with 5x3x1mm³ were created with four dental ceramics following manufacturers' instructions: IPS Classic (Ivoclar-Vivadent); IPS d.Sign (Ivoclar-Vivadent); EX3 (Noritake); VMK-95 (Vita). A microhardness tester with a Knoop diamond with a 100g load was used to evaluate the baseline microhardness values of all ceramics. Afterwards, the specimens were submitted to 6-hour daily bleaching treatments with 10% or 16% carbamide peroxide (Whiteness- FGM) for 21 days, while control groups from each ceramic system were maintained in artificial saliva. The microhardness of all groups was evaluated at 18h, 42h, 84h, and 126h of bleaching treatment. The mean value of 5 indentations performed at each specimen in each time was obtained and all data were submitted to two-way repeated measures ANOVA and Tukey's post-hoc test ($\alpha=0.05$). No significant differences in ceramic microhardness were observed among either bleaching intervals or bleaching treatments. Ceramic restorations are not affected by carbamide peroxide 10 or 16% gel during bleaching treatment.

Keywords: Esthetics; Bleach; Peroxide; Carbamide Peroxide; Hardness; Ceramics; Porcelain; Demineralization; at-home bleaching; dental bleaching.

Clinical relevance statement: This study provided evidence that at-home bleaching systems do not cause detrimental effects on dental ceramics.

Introduction

The best treatment for discolored vital teeth is the dental bleach. The most indicated bleaching technique is the at-home performed, which presents

effective results in few weeks¹. Firstly described by Haywood & Heymann in 1989, the so-called nightguard dental bleaching involves the day or night use of a tray with carbamide peroxide from two to eight hours a day^{1,2}.

Although the wide use of at-home bleaching, this technique may lead to clinical side effects due to the reactive nature of the hydrogen peroxide, so patients may experience dentin sensibility and or gingival irritation^{1,3,4,5}. Microscopically, several alterations are also expected in the enamel morphology due to mineral loss, and surface roughening⁶⁻¹⁵.

Such alterations on tooth tissues are related to the low pH of hydrogen peroxide and to its decomposition into H⁺ free radicals, which are extremely instable and reactive⁶⁻¹⁷. Although conventional dental ceramics are considered the most inert of all dental materials used for dental restorations, the surfaces of dental porcelains can exhibit surface deterioration in contact with acidulated fluoride gels or solutions²⁶. Also, selective leaching of alkali ions and dissolution of the glass network of ceramic may occur by the diffusion of free radicals of H⁺ or H₃O⁺. As hydrogen peroxide releases a great amount of free radicals that may potentially affect dental porcelain exposed accidentally or not to bleaching gel during treatment, the aim of the present study was to evaluate the effect of 10% and 16% carbamide peroxide bleaching agents on microhardness of dental ceramics after different time periods of bleaching treatment.

Materials and Methods

The microhardness of four dental ceramics EX-3 (Noritake Kizai Co., Limited - Aichi, Japan), IPS Classic (Ivoclar Vivadent AG- Schaan, Principality of Liechtenstein), IPS d.Sign (Ivoclar Vivadent AG - Schaan, Principality of Liechtenstein) and VMK 95 (Vita Zahnfabrik - Bad Säckingen, Germany) were evaluated in a research protocol including a factorial design to test the effects of 3 surface treatments: 10% carbamide peroxide (Whiteness FGM); 16% carbamide Peroxide- (Whiteness FGM); and no treatment (control group); at 5 periods of treatment: 0h (before treatment); 18h; 42h; 84h; and 126h.

Fifteen specimens with 5x3x1mm³ of each ceramic were prepared according to manufacturers' instructions and had their surfaces sequentially

polished with diamond polishing pastes of 6, 3, 1, and 0.5 μm and polishing cloths with mineral oil lubricant (top, Gold and Ram, Arotec Ind Com Ltda, Cotia - Brazil).

Microhardness test was performed by a single evaluator prior to and after the bleaching treatment with Knoop indenter with load of 200g applied for 5 s. As recommended by Siew¹⁸, five indentations were evaluated at each interval. The 0h indentations were performed at a distance of 30 μm between each other in the center of the ceramic specimens. In the following intervals, the five indentations were performed 100 μm distant from and on the left of the previous indentations.

The 15 ceramic specimens were randomly divided in three groups according to the surface treatments, having five specimens each (n=5). The respective treatment agent was applied for 6 hours a day during 21 days, corresponding to 126h of treatment. Specimens were covered with 0.03 ml of the bleaching agent and a drop of artificial saliva artificial^{6,7,8,10}, excepting the untreated specimens, which received only the artificial saliva. The specimens were placed in vacuum-formed custom trays^{8,14} and were stored in a closed plastic container at 37°C.

The indentation lengths from each specimen in each interval were measured in micrometers, and transformed into Knoop hardness number (KHN). The mean of the five Knoop hardness (KHN) values obtained from each specimen either before or following the treatment were statistically analyzed by 2-way repeated measures ANOVA with and Tukey's post-hoc test at 5% level of significance within each ceramic^{6,7,8}.

Results

The mean KHN values of each ceramic before and after the treatment with the respective standard deviations are shown in Table 1. No significant difference in KHN values was observed between the control group and bleached groups, as well as between groups treated with 10% carbamide peroxide and those treated with 16% carbamide peroxide, regardless of time. Moreover, no significant difference in KHN values was observed among time intervals, regardless of treatment.

Table 1 – Mean KHN values of each ceramic (SD) at each evaluation interval.

Ceramic (Lot number)	Surface treatment	0h	18h	42h	84h	126h
EX-3 (Lot: 008494)	Control	491.3(12.6)	500.8(4.5)	499.0(10.2)	505.4(15.9)	500.7(1.9)
	PC10%	515.2(34.3)	510.7(23.0)	504.4(18.0)	508.7(6.4)	509.0(13.3)
	PC16%	517.0(10.2)	520.5(17.8)	516.2(19.9)	527.6(23.0)	520.4(39.3)
IPS d.Sign (Lot: K33292)	Control	484.5(9.5)	499.6(16.3)	503.3(12.9)	516.7(7.1)	505.3(9.9)
	PC10%	491.3(41.8)	511.1(21.7)	516.7(21.4)	517.1(19.9)	515.9(16.6)
	PC16%	513.8(35.3)	510.3(17.4)	498.2(23.3)	503.5(17.9)	511.5(16.3)
VMK 95 (Lot: 26590)	Control	534.2(30.4)	524.5(15.7)	524.4(12.8)	531.5(15.8)	524.0(8.9)
	PC10%	533.6(19.0)	532.7(8.6)	529.0(8.3)	532.0(24.8)	538.8(18.2)
	PC16%	524.5(22.5)	523.7(32.1)	524.6(20.4)	530.6(21.9)	536.9(14.4)
IPS Classic (Lot: K02827)	Control	499.4(4.6)	515.1(12.0)	509.4(27.5)	495.6(11.0)	492.4(10.0)
	PC10%	483.7(4.4)	498.5(20.4)	494.1(20.5)	498.8(13.1)	501.8(19.6)
	PC16%	494.9(20.3)	489.4(20.3)	484.9(12.8)	489.0(9.0)	491.9(13.3)

Discussion

Chemical durability is the main property expected from ceramics for intra-oral use, since dental prostheses must stand to degradation in the presence of a wide range of solutions with variable pH²⁶. The integrity of a ceramic avoids possible side-effects such as increased plaque adhesion, release of potentially toxic species as a result of wear, release of radioactive components, and increased abrasion of opposing dental structures²⁶.

The ceramics evaluated in the current study did not show statistical differences in microhardness values after 126 hours of exposure to carbamide peroxide at the concentrations of 10% or 16%, demonstrating to be inert *in vitro* to dental bleaching. The bleaching protocol used in the present study was similar to that of others studies, which aimed to evaluate *in vitro* the effect of bleaching systems on the enamel surface microhardness through time^{6,8,9,10,14}.

Results similar to the those from the current study were observed in several studies, in which no significant changes in microhardness values were found when ceramics were treated with 15% carbamide peroxide for 56 hours¹⁹, 6.5% hydrogen peroxide for 14 hours²⁰, 38% hydrogen peroxide for 30 minutes¹⁹ or 45 minutes²¹.

Despite the high ceramic stability, some degradation in ceramic materials was expected in the present study because of the interaction of free radicals released from the bleaching gels with the ceramic glass network, leading to the loss of alkali metal ions from the glass surface¹⁷. The loss of alkali ions from ceramic material could also occur due to the low pH of bleaching gels, which could also probably decrease microhardness, but such effect was not observed in the four different commercial brands of dental ceramic from the beginning throughout the 126 h of bleaching treatment.

The 126 h of treatment was chosen to simulate 21- day nightguard bleaching treatment and most patients achieve best results within this period. On the other hand, bleaching treatment may be extended to longer treatment periods in patients with severe discoloration, as bleaching detrimental effects are time dependent, more intense mineral loss is expected on enamel and dentin in extended treatments. A time-dependent effect of bleaching treatment on ceramic microhardness may also be suggested if the results of 126 hours of treatment from the present study are compared with those from Turker & Biskin²¹ of 240 hours of treatment. These authors showed a statistically significant decrease in ceramic microhardness after 240 hours of treatment with 10% or 16% carbamide peroxide. Therefore, it can be supposed that the ceramic material may suffer some degradation after long period of bleaching treatment. In addition, Turker & Biskin in the next year, performed surface spectral analyses in ceramics treated for 240 hours with 10% carbamide peroxide and found a decrease in the SiO₂ content, which is the main component of the matrix and for this reason its lower content would affect the surface microhardness²². However, the same authors demonstrated that the bleaching gels affected only surface roughness, then the small amount of released SiO₂ was not considered to be of clinical significance²². Also, some studies showed alterations on ceramic surface after bleaching treatment by scanning electron microscopy and roughness profiles, but concluded that these alterations were clinically insignificant^{26,29,31}.

The degradation of dental ceramics generally occurs because of chemical attack, mechanical forces or a combination of these effects²⁶. In the current study only the chemical attack of ceramics by 10% or 16% hydrogen peroxide was considered, but different results could be found if mechanical

forces was employed since it could weak the structure by creating surface flaws and increase the susceptibility of ceramic to sequential bleaching attack, then more studies are needed to evaluate this factor.

With this regard, the present study showed that ceramic dental materials were not affected by 10% or 16% carbamide peroxide treatment, so there is no need for ceramic replacement in clinical situations where ceramic restorations were accidentally exposed to bleaching gels, once color, form and function are clinically acceptable.

Conclusion

Within the limitations of the current study, the microhardness of the evaluated dental ceramics was not affected by treatment with 10% or 16% carbamide peroxide for 126 hours.

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Capítulo 2- Effect of different concentrations of carbamide peroxide and bleaching periods on surface roughness of dental ceramics – Sergio

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Especialidade: Dentística

Abstract

The objective of this study was to evaluate the effect of 10% and 16% carbamide peroxide bleaching agents on surface roughness of dental ceramics after different periods of bleaching treatment. Fifteen specimens with 5x3x1mm were created with three dental ceramics following manufacturers' instructions: IPS Classic (Ivoclar-Vivadent); IPS d.Sign (Ivoclar-Vivadent); VMK-95 (Vita). A profilometer was used to evaluate the baseline surface roughness (Ra values) of all ceramics by 5 parallel measurements with 5 cut off of 0.25 mm (λ_c), and a speed of 0.1 mm/s. Afterwards, all specimens were submitted to 6-hour daily bleaching treatments with 10% or 16% carbamide peroxide (Whiteness- FGM) for 21 days, while control groups from each ceramic system were stored in artificial saliva. The surface roughness of all groups was evaluated at 18h, 42h, 84h, and 126h of bleaching treatment. The mean value of 5 parallel measurements performed on each specimen in each time was obtained and all data were submitted to two-way repeated measures ANOVA and Tukey's post-hoc test ($\alpha=0.05$). No significant differences in ceramic surface roughness between untreated and bleached ceramic surfaces, regardless of bleaching intervals or bleaching treatments. Ceramic restorations are not affected by carbamide peroxide 10 or 16% gel during bleaching treatment. This study provided evidence that at-home bleaching systems do not cause detrimental effects on dental ceramics surface roughness.

Descriptors: Esthetics; Tooth Bleaching; Hydrogen Peroxide; Peroxides; surface properties; Ceramics; Porcelain.

Introduction

In the last years, dental bleaching has become popular and much requested by patients willing to improve the color of their teeth. The most useful and effective bleaching technique is the one performed at-home, which can bleach all teeth in two weeks with few side-effects such as dental sensitivity¹. This technique was firstly described by Haywood & Heymann in 1989 as nightguard dental bleaching, but nowadays this technique may be performed from one to eight hours a day at-home involving the day or night use of a tray with a bleaching agent^{1,2}.

The most commonly used dental bleaching agent is carbamide peroxide. The reaction of carbamide peroxide releases, hydrogen peroxide and free radicals, which are responsible for dental bleaching^{3,4}. Despite the wide approval of at-home bleaching technique, the use of peroxides may lead to clinical side effects due to the reactive nature of hydrogen peroxide, so patients may experience dentin sensitivity and/or gingival irritation^{1,3,4,5}. Microscopically, several changes on the enamel surface morphology are also observed due to enamel mineral loss and surface roughening⁶⁻¹⁶.

The prolonged use bleaching agents, which release H⁺ free radicals that are extremely unstable and reactive, and their acidic pH are described as the main cause of side-effects^{6-14,17}. Similarly, bleaching agents may cause structural alterations on restorative materials that impair their physical properties and may lead to premature failure¹⁸⁻²⁴. Although conventional dental ceramics are considered the most inert of all dental restorative materials, the surfaces of dental porcelains can exhibit surface deterioration in contact with acidulated fluoride gels or other solutions²⁵. Also, the contact and possible diffusion of free radicals of H⁺ or H₃O⁺ produced by bleaching agents¹⁷ may selectively leach alkali ions and cause the dissolution of the ceramic glass network²⁵. Then, the prolonged exposure of hydrogen peroxide may potentially affect dental porcelain exposed to at-home bleaching treatment and produce alterations on its surface. Moreover, an increase in surface roughness above the threshold of Ra = 0.2 micron may result in an increase in plaque accumulation, thereby increasing the risk of both

secondary caries and periodontal inflammation²⁶ or affecting the ceramic aesthetics by changing the texture of the ceramic restoration.

The hypothesis of the present study is that the surface roughness of ceramic may be modified by exposure to 10% and 16% carbamide peroxide bleaching agents used to at-home treatment in a period of 126h. Then, the aim of the present study was to evaluate the effect of 10% and 16% carbamide peroxide bleaching agents on surface roughness of dental ceramics after different time periods of bleaching treatment.

Materials and Methods

The surface roughness of three dental ceramics (Table 1) one fluorapatite glass-ceramic IPS d.Sign (Ivoclar Vivadent AG - Schaan, Principality of Liechtenstein) and two feldspathic ceramic IPS Classic (Ivoclar Vivadent AG- Schaan, Principality of Liechtenstein), and VMK 95 (Vita Zahnfabrik - Bad Säckingen, Germany) were evaluated in a research protocol including a factorial design to test the effects of 3 surface treatments: 10% carbamide peroxide (Whiteness FGM, Joinville, SC-Brazil; pH=6.0); 16% carbamide Peroxide- (Whiteness FGM, Joinville, SC-Brazil; pH=6.0); and no treatment (control group); at 5 periods of treatment: 0h (before treatment); 18h; 42h; 84h; and 126h.

Table 1 – Type, chemical characterization*, commercial brand, and lot of ceramics.

Ceramic (Lot number)	Type	Chemical characterization*
IPS d.Sign (Lot: K33292)	<i>Fluorapatite-leucite glass-ceramic</i>	SiO ₂ ; BaO; Al ₂ O ₃ ; CaO; CeO ₂ ; Na ₂ O; K ₂ O; B ₂ O ₃ ; MgO; ZrO ₂ ; P ₂ O ₅ ; F; Li ₂ O; TiO ₂ ; SrO; ZnO; and pigments
VMK 95 (Lot: 26590)	<i>Feldspathic ceramic</i>	Al ₂ O ₃ ; BaO; B ₂ O ₃ ; CaO; Fe ₂ O ₃ ; MgO; SiO ₂ ; TiO ₂ ; ZrO ₂ ; CeO ₂ ; Li ₂ O; K ₂ O; Na ₂ O; Glycerine; Butylene Glycol; Tin Oxide.
IPS Classic (Lot: K02827)	<i>Feldspathic ceramic</i>	SiO ₂ ; BaO; Al ₂ O ₃ ; CaO; CeO ₂ ; Na ₂ O; K ₂ O; B ₂ O ₃ ; MgO; ZrO ₂ ; P ₂ O ₅ ; TiO ₂ ; and pigments

* Material Safety Data Sheet; Abbreviations: SiO₂: Silicon Oxide; BaO: Barium Oxide; Al₂O₃: Aluminum oxide; CaO: Calcium Oxide; CeO₂: cerium dioxide; Na₂O: Sodium Oxide; K₂O: Potassium Oxide, B₂O₃: Boron Oxide; MgO: Magnesium Oxide; ZrO₂: Zirconium Oxide; P₂O₅: Phosphorus pentoxide F: Fluor, Li₂O: Lithium Oxide; TiO₂: Titanium Dioxide; SrO: Strontium oxide; ZnO: Zinc oxide; Fe₂O₃: Iron Oxide.

Fifteen specimens with 5x3x1mm of each ceramic were prepared according to manufacturers' instructions and had their surfaces sequentially polished with diamond polishing pastes of 6, 3, 1, and 0.5 μm and polishing cloths with mineral oil lubricant (top, Gold and Ram, Arotec Ind Com Ltda, Cotia - Brazil).

Surface roughness was evaluated by a single blinded evaluator prior to and after the bleaching treatment. A profilometer (TR200, Time Group Inc, Beijing, China) was used to scan, with a microneedle, the surface roughness employing the parameter surface roughness average (Ra). Five points were initially marked in order to ensure repeatable measurements with the profilometer. From these points, five parallel measurements in longitudinal direction were performed on the surface of each specimen, with a cut off of 0.25 mm (λc), and a speed of 0.1 mm/s. The surface roughness was recorded and the mean roughness value (Ra expressed in μm) was determined for each specimen before and after treatment.

The 15 ceramic specimens were randomly divided in three groups according to the surface treatments (n=5). The respective treatment agent was applied for 6 hours a day during 21 days, corresponding to 126-hour treatment. Specimens were covered with 0.03 ml of the bleaching agent, placed in vacuum-formed custom trays, with a drop of artificial saliva^{8,13} and were stored in a plastic container at 37°C⁶⁻⁸, excepting the untreated specimens, which were stored only with the artificial saliva drop in the vacuum-formed custom tray to mimic oral conditions.

After each 6 hours periods of bleaching exposure, the specimens were washed with distilled water to remove the residual carbamide peroxide gel, and stored in a plastic container for the remaining day period in relative humidity at 37°C.

Surface roughness was measured at 18 h, 42 h, 84 h, and 126 h after the beginning of the experiment after wash and dry the specimens. The mean of the five measurements of surface roughness values obtained from each specimen either before or following the treatment were statistically analyzed by 2-way repeated measures ANOVA and Tukey's post-hoc test at 5% level of significance within each ceramic⁶⁻⁸.

Results

The mean Ra values of each ceramic before and after the treatment with the respective standard deviations are shown in Table 2. No significant difference in Ra values was observed between the control group and bleached groups, as well as between groups treated with 10% carbamide peroxide and those treated with 16% carbamide peroxide, regardless of time. Moreover, no significant difference in Ra values was observed among time intervals, regardless of treatment.

Table 2 – Mean of surface roughness (Ra) values of each ceramic, and standard deviation (SD) at each evaluation interval.

Ceramic (Lot number)	Surface treatment	0h	18h	42h	84h	126h
IPS d.Sign	Control	0.035±0.001	0.037±0.002	0.036±0.003	0.033±0.004	0.036±0.002
	PC10%	0.036±0.003	0.033±0.002	0.035±0.002	0.033±0.002	0.034±0.003
	PC16%	0.031±0.002	0.033±0.003	0.034±0.004	0.033±0.001	0.032±0.004
VMK 95	Control	0.073±0.002	0.070±0.004	0.074±0.002	0.074±0.002	0.073±0.003
	PC10%	0.074±0.003	0.075±0.004	0.073±0.003	0.074±0.003	0.074±0.003
	PC16%	0.074±0.003	0.072±0.004	0.072±0.003	0.074±0.005	0.074±0.002
IPS Classic	Control	0.075±0.002	0.077±0.002	0.076±0.001	0.076±0.002	0.075±0.002
	PC10%	0.075±0.004	0.078±0.004	0.077±0.003	0.076±0.004	0.076±0.003
	PC16%	0.080±0.004	0.0786±0.004	0.077±0.002	0.079±0.003	0.080±0.002

No significant difference in surface roughness was noted among groups ($p>0.05$)

PC: carbamide peroxide

Discussion

The main property expected from ceramics is the chemical durability in the mouth, since dental prostheses must stand to degradation in the presence of a wide range of solutions with variable pH²⁵. The ceramics need avoid possible intra-oral challenges and side-effects such as release of potentially toxic substances and radioactive components as a result of wear, increased abrasion of opposing dental structures and increased plaque adhesion²⁵.

This study tested the effect of dental bleaching agents on surface roughness of ceramic specimens with initial roughness average lower than 0.2 micron, a condition that leads to bacterial accumulation similar to that observed on the least rough surface²⁶. The ceramics evaluated in the current study did not show significant differences in roughness values after 126 hours

of exposure to carbamide peroxide at the concentrations of 10% or 16% in comparison to the values before bleaching treatment, demonstrating to be inert *in vitro* to dental bleaching and rejecting the hypothesis of the study. Therefore, an accidental exposure of dental ceramics to bleaching agents does not increase surface roughness that may increase the risk for both secondary caries and periodontal inflammation.

The bleaching protocol used in the present study was similar to that from other studies, which aimed to evaluate *in vitro* the effect of bleaching systems on the enamel surface roughness overtime^{6,8,9,13}. Although an increase in roughness has been observed in composite resins and glass ionomers after bleaching treatment¹⁸⁻²⁴, no alteration on ceramic surfaces was observed after bleaching in the current study²³, so the impact of bleaching agents on the surface roughness may be considered material-dependent, as also demonstrated by Polydorou et al (2006).

Ceramic stability against bleaching agents was observed in several studies, which showed no significant changes in microhardness values after treatment with 15% carbamide peroxide for 56 hours²³, 6.5% hydrogen peroxide for 14 hours²⁷, 38% hydrogen peroxide for 30 minutes²³ or 45 minutes²⁴.

The 126-h bleaching treatment was chosen to simulate 21-day nightguard bleaching treatment as most patients achieve the best results within this period^{4,5}. Although this period may be considered optimum, bleaching treatment may be extended to longer treatment periods in patients with severe discoloration. As detrimental effects of bleaching treatment are time dependent, more mineral loss is expected on enamel and dentin surfaces in extended treatments^{1,6,24}. Therefore, despite the absence of changes in surface roughness in the three different commercial brands of dental ceramic from the beginning throughout the 126 h of bleaching treatment, it is possible that some degradation in ceramic materials could occur after longer exposure, over than 126h, due to the interaction of free radicals released from the bleaching gels with the ceramic glass network, leading to the loss of alkali metal ions from the glass surface. However, only further evaluation comprising longer exposure to bleaching agents would confirm such speculation. Although no significant difference in roughness

was observed during 126-h bleaching treatment, a time-dependent effect of bleaching treatment on ceramic microhardness should not be discarded. According to Turker & Biskin, a statistically significant decrease in ceramic microhardness was observed after 240 hours of treatment with 10% carbamide peroxide²⁰. Furthermore, a spectral analysis of ceramic surfaces exhibited a decrease in the SiO₂ content, which is the main component of the matrix²¹. Thus, its lower in content would affect other properties as the surface microhardness, although the study found no significant difference in roughness values.

Also, Polydorou et al (2006)²³ showed that alterations may be concentration-dependent, as polished ceramic surfaces exposed to 38% hydrogen peroxide exposure for 45 minutes showed slight changes, while no significant difference were noted when the ceramic surfaces were exposed to 15% carbamide peroxide exposure for 56 hours.

However, other authors demonstrated that the bleaching gels affected surface roughness of dental ceramic. Moraes et al. (2006) observed a statistically significant increase in the surface roughness of ceramic material after 21 days of daily application of 10% carbamide peroxide and a weekly application of 35% found, although no alterations in roughness were observed throughout 7 and 14 days of bleaching. According to the authors, these results are related to a leach of any component from porcelain matrix as a function of continuing peroxide application. However, the Ra values observed in the study were within the clinically acceptable range (Ra value of 0.22 to 0.24) and the alterations would probably be clinically insignificant. In addition, some studies showed alterations on ceramic surface after bleaching treatment by scanning electron microscopy, but the authors described these alterations as clinically insignificant^{28,30}.

The degradation of dental ceramics generally occurs because of chemical attack, mechanical forces or a combination of these effects²⁵. In the current study, only the chemical attack of ceramics by 10% or 16% hydrogen peroxide was considered, but different results could be found if mechanical forces were applied since it could weaken the structure by creating surface cracks and increase the susceptibility of ceramic to sequential bleaching

attack. For this reason, further studies are required to evaluate this clinical challenge.

With this regard, the present study showed that ceramic dental materials were not affected by 10% or 16% carbamide peroxide treatment, so there is no need for ceramic polishment or replacement in clinical situations where ceramic restorations were accidentally exposed to bleaching gels, once color, shape and function are clinically acceptable.

Conclusion

Within the limitations of the current study, the surface roughness of the evaluated dental ceramics was not affected by treatment with 10% or 16% carbamide peroxide for 126 hours.

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Capítulo 3- Surface roughness evaluation of in vitro refinished dental ceramics followed by bleaching treatment- Sérgio Augusto Morey Ourique, Leonardo Colombo Zeidan, César Augusto Galvão Arrais, Alessandra Cassoni, José Augusto Rodrigues

Artigo redigido para envio para publicação

Abstract

Aim: The objective of this study was to evaluate the effect of refinishing process on dental ceramics roughness followed by bleaching treatment with 16% carbamide peroxide.

Materials & Methods: Fourteen specimens of 5x3x1mm were produced with two dental ceramics following manufacturers' instructions: IPS d.Sign (Ivoclar-Vivadent); and VMK-95 (Vita). A profilometer was used to evaluate the baseline surface roughness (Ra values) of all ceramics acquiring 3 profiles with five 0.25 mm cut off (λ_c) at 0.1 mm/s. All specimens were submitted to surface treatments with a diamond bur (91-126 μm -grit) to simulate an occlusal adjustment followed by the refinishing procedures with fine (2135F – 37-44 μm -grit) and extra fine (2135FF – 20-40 μm -grit) diamond burs; and with polishing with abrasive cups and paste (OptraFine – Ivoclar Vivadent). After refinishing, the ceramics were divided into a bleached (BL) and a non-bleached subgroups (NB). BL groups were bleached for 6-hour daily with 16% carbamide peroxide (Whiteness- FGM) for 21 days, while NB groups were stored in artificial saliva. The surface roughness was evaluated after each surface treatment and data submitted to 2-way ANOVA and Tukey's test.

Results: There were no statistical significant differences on surface roughness between ceramics regarding surface treatments. The adjustment of dental ceramics with diamond burs drastically increases the surface roughness. The solely treatment with fine and extra fine diamond bur did not reduce the ceramic surface roughness.

Conclusion: Acceptable surface roughness was obtained after refinishing with polishing abrasive cups and paste. The 16% carbamide peroxide treatment was not able to alter the refinished ceramic surface roughness.

Clinical Significance: Ceramic refinishing is properly obtained after sequential polishment with diamond burs to abrasive cups and paste.

Keywords: Laboratory research; Dental; Dental bleaching; Hydrogen peroxide; Surface properties; Ceramics.

Introduction

Ceramic systems have become increasingly popular due to their esthetic properties including conventional metal-ceramic, reinforced ceramics and metal free alumina and zirconia-based materials.

Dental ceramics are considered the most inert of all dental restorative materials, and the main property expected from ceramics is the chemical durability in the mouth, since dental prostheses must stand to degradation in the presence of saliva and a wide range of transitory solutions with variable pH.¹

As an indirect restorative material, the ceramic prostheses are manufactured out of buccal cavity and cemented in the prepared tooth after subjected to a superficial glaze treatment. However, occlusal adjustment of ceramic restorations with high granulation diamond burs may be necessary to correct interferences after cementation. These final adjustments may result in loss of ceramic glaze,^{2,3} which raises some concerns because these materials requires to be refinished.

Ceramics prostheses must be adequately polished to be less susceptible to biofilm and bacterial accumulation, and reduce the potential of wearing opposing occlusal surfaces.³⁻⁸ Also, the mechanical and physical strength of a ceramic restoration can be impaired by refinishing process due microcracks formation and can be more susceptible to later catastrophic fractures.⁹⁻¹¹

This way, the superficial roughness of adjusted ceramic must be reduced with intraoral polishing techniques to achieve an acceptable smoothness and preserve the material as inert as possible.³ Special attention for selection of adequate materials and instruments must be taken because polishing is usually a multistage process. The first stage starts with a rough abrasive and each subsequent stage uses a finer abrasive until the desired finish is achieved. There are a lot of polishing kits, rubber cups and discs in the market but the correct decreasing sequence of abrasive size must be respected.

If occlusal adjustment of a ceramic restoration has to be made after cementation there is always need for a careful intraoral polishing with polishing kits and discs.⁸ The polishing techniques researches showed that the use of a refinishing kit followed by polishing paste or polishing stick application may create surfaces as smooth as glazed specimens. Polishing kits and discs were found more effective than the polishing pastes used alone or combined with Sof-lex discs, resulting in improved surface smoothness.¹²

To describe the overall texture of a surface it is common to use a profilometer and state the results by the parameter “roughness average” (Ra) that refers to the arithmetical average value of all absolute distances of the roughness profile from the center line within the measuring length.⁸ Then, an adequate polishing technique is able to progressive reduce the length of fissures, cracks and flaws caused by diamond burs and also reduce the Ra value

In addition, the prolonged exposure of fissures and cracks on ceramic surface to saliva and other substances as fluorides and bleaching agents may induce progressive flaws.¹³⁻¹⁹ Bleaching agents are composed by high oxidant molecules which release H⁺ free radicals that are extremely unstable and reactive, and their acidic pH are described as the main cause of the detrimental dental side-effects.²⁰⁻²⁶ Although, the effects on dental ceramics are still controversial, studies showed that bleaching agents may cause structural alterations on dental enamel and restorative materials that impair their physical properties and may lead to premature failure.^{14-19,27}

This way it may be supposed that refinishing procedures may induce fractures on ceramic surfaces that could be more severe if treated with bleaching agents impairing mechanically the durability and esthetics results.

This study tested two hypotheses. The first hypothesis is that a diamond bur adjusted roughness surface of a ceramic may be refinished with fine and extra fine diamond burs followed by abrasive cups and diamond pastes. The second hypothesis is that treatment with 16% carbamide peroxide bleaching agents used to at-home treatment do not affect the roughness surface of refinished ceramic.

Then, the aim of this study was to evaluate the effect of refinishing process on dental ceramics roughness and the effect of bleaching treatment with 16% carbamide peroxide on refinished ceramics.

MATERIALS & METHODS

Experimental design

The factors under study first hypothesis were “Dental Ceramic” in two levels (Fluorapatite-leucite glass-ceramic - IPS d.Sign; Feldspathic ceramic - VMK 95; n=14 per group) and “Refinishing” treatment in four levels (Baseline; Adjustment procedure; Refinishing with fine and extra fine diamond burs; Refinishing with abrasive cup/paste; and carbamide peroxide) evaluated by repeated measurements. To study the second hypothesis the ceramics were divided in two subgroups (n= 7 per group) the study factor was bleaching treatment; submitted or not to the bleaching treatment (IPS d.Sign NB; IPS d.Sign BL; VMK 95 NB, VMK 95 BL). The response variable was surface roughness (Ra) in μm .

Specimens' preparation

Fourteen specimens with 5x3x1mm of each ceramic, IPS d.Sign (Ivoclar Vivadent AG - Schaan, Principality of Liechtenstein) and VMK 95

(Vita Zahnfabrik - Bad Säckingen, Germany) were prepared according to manufacturers' instructions and had their surfaces sequentially polished by metallographic technique with diamond polishing pastes of 6, 3, 1, and 0.5 μm and polishing cloths with mineral oil lubricant (Top, Gold and Ram, Arotec Ind Com Ltda, Cotia - Brazil), and the baseline surface roughness measurement was performed.

Surface roughness test

A profilometer (TR200, Time Group Inc, Beijing, China) was used to scan, with a microneedle, the surface roughness employing the parameter surface roughness average (Ra) in μm . Surface roughness was evaluated by a single blinded evaluator prior to and after each surface treatment. Three points were initially marked in order to ensure repeatable measurements of the profiles. From these points, two perpendicular and one transversal profiles were obtained on the surface of each specimen, with a cut off of 0.25 mm (λc), and a speed of 0.1 mm/s. The surface roughness was recorded and the mean roughness value (Ra expressed in μm) was determined for each specimen before and after treatment.

Surface refinishing treatment

A single blinded operator performed the surface treatments with the specimens fixed in wax in the same position. The treatments with rotatory instruments were performed with manual pressure with horizontal movements from left to right side of the specimen for 20 seconds.

Four surface treatments were performed. The first treatment aimed to simulate the clinical adjusts of an oclusal surface with a diamond bur. This treatment was performed with a 2136 diamond bur (KG Sorensen, Barueri, SP, Brazil/ 91-126 μm -grit) at high speed under a constant water spray coolant, and the surface roughness was measured.

To verify the refinished with fine (F) and extra fine (FF) diamond burs, the ceramic specimens were refinished with a fine 2135F diamond bur (Vortex, Sao Paulo, SP, Brazil) with a granulation of 37-44 μ m-grit followed by an extra fine 2135FF diamond bur (Vortex, Sao Paulo, SP, Brazil) with a granulation of 20-40 μ m-grit.

After that, the surface roughness was evaluated and the specimens were polishing with abrasive cups and paste (OptriFine, Ivoclar Vivadent AG - Schaan, Principality of Liechtenstein). The ceramic specimens were treated with the diamond finisher F cup followed by the diamond polisher P cup, and the diamonds polishing paste HP (granulation of 2-4 μ m) with nylon brushes, followed by the surface roughness evaluation.

One representative specimen with surface refinishing treatment of each ceramic was observed by scanning electron microscopy with 70x of magnification (SEM - FEI; Quanta 600F, Nederland, NE).

Bleaching treatment

After all refinished procedures, ceramics specimens were divided in two subgroups VMK 95 NB, VMK 95 BL, IPS d.Sign NB, and IPS d.Sign BL. IPS d.Sign BL and VMK 95 BL were challenged by 16% carbamide Peroxide- (Whiteness FGM, Joinville, SC-Brazil; pH \approx 6.0) to simulate an in vitro bleaching treatment, and the specimens of groups VMK 95 NB and IPS d.Sign NB were kept in artificial saliva for 21 days containing calcium and phosphate at a known degree of saturation (1.5 mmol/L Ca, 0.9 mmol/L PO₄), to mimic the remineralizing properties of saliva, and 50 mmol/L KCl, 20 mmol/L tri-hydroxymethylaminomethan buffer at pH 7.0.

The bleaching agent was applied for 6 hours a day during 21 days, corresponding to 126-hour treatment. Specimens were covered with 0.03 ml of the bleaching agent, placed in vacuum-formed custom trays, with a drop of artificial saliva and were stored in a plastic container at 37°C.²⁷ After each 6 hours periods of bleaching exposure, the specimens were washed with distilled water to remove the residual carbamide peroxide gel, and stored in a

plastic container for the remaining day period with artificial saliva at 37°C. After the 21 days of treatment the surface roughness was evaluated.

Statistical analysis

To analyze the surface refinishing treatment the factors “Dental Ceramic”, “Refinishing” and the interaction between them were analyzed by split plot 2-way ANOVA and Tukey’s test. The effect of bleaching treatment was independently evaluated for each ceramic by T test.

RESULTS

No statistical significant interaction between “Dental Ceramic” and “Refinishing” factors was observed ($p>0.05$). No statistical significant differences on surface roughness was observed between the dental ceramics regardless of surface treatment ($p>0.05$). Statistical significant differences were observed in the factor “Superficial Treatment”. Also, the two ceramics roughened with diamond burs showed similar surface morphology (Fig. 1B and 2B). There was a statistical significant increase in the surface roughness of ceramics after adjustment procedure (Tab. 2), with the highest numbers of pits and more altered surface (Fig. 1B and 2B) when compared to baseline (Fig. 1A and 2A).

The refinishing with fine and extra fine diamond burs statically reduced the surface roughness after adjust procedure (Tab. 2), but the surface roughness was still higher than baseline value with less shallow pits than adjusted one (Fig. 1C and 2C).

The refinished procedure with abrasive cups and paste statistically reduced the surface roughness obtained with refinished with fine and extra fine diamond burs at a level statistically similar to baseline values (Tab. 2). Pits and fissures were removed. The means and standard deviations are described in Table 2 and are graphically represented in graph 1.

The T test showed no statistical significant differences between bleached and non bleached groups for both studied refinished ceramics. The means and standard deviations are described in Table 3 and are graphically represented in graph 1.

DISCUSSION

Dental ceramic has found an increased number of applications in recent years, it is used in metal-ceramic and all-porcelain crowns and bridges for the restoration of anterior and posterior teeth.²⁸ Ideally, porcelain restorations should maintain their glazed surface, but it is very frequent the need to perform an adjustment before cementation or soon after cementation. The adjustment with diamond burs produced an irregular surface, leaving easily identifiable fissures (Figs. 1B and 2B).

This procedure break the glazed surface that could lead to the initiation of microcracks and, under further wear and in the presence of moisture, to subsequent, more pronounced destruction of the ceramic.⁷ Also, to avoid abrasive wear of the opposing dentition, and plaque accumulation the best finish and least abrasive surface need to be achieved by ceramic refinishing. Commercial porcelain refinishing kits are claimed to restore the surface finish on porcelain after adjustments in circumstances that preclude laboratorial reglazing.³⁰

In the present study, specimens of two ceramic systems were produced and submitted to a metallographic polishment to produce a smooth surface (Figs. 1A and 2A) with roughness average (Ra) approximately of 0.2 μm (Table 2). This roughness average is close to a glazed ceramic¹³ and a condition that leads to bacterial accumulation similar to that observed on the least rough surface.³¹ This baseline value was considered as the gold standard to polishment. Although the studied ceramic had different compounds, there were no significant differences in roughness values between porcelain independent of treatment which may be supposed

attributed to a relation with diamond abrasive particles size and physical properties.

The refinishing procedure using in a decreasing granulation order of abrasive diamond burs (F and FF) statistically reduced the remarkable morphological alterations on ceramic surface caused by diamond burs. However, a non-clinically acceptable rough surface with fewer pits, grooves and undercuts could be observed by scanning electron microscopy (Figures 1C and 2C). In addition, the surface roughness reduction by refinishing only with F and FF diamond burs result in a higher rough surface than baseline control situation due to 20-40 μm diamond grade. Another research showed that a refinishing kit with a grade finer than 15 μm would be more appropriate for porcelain adjustments to permit a surface smoothness comparable to the original glaze.³⁰

After the final polishment with abrasive cups and polishing paste an uniform peeling was achieved (Figure 1D and 2D) with a flat surface and surface roughness non different from baseline control surface accepting the first hypothesis of the study, that a diamond bur adjusted roughness surface of a ceramic may be refinished with fine and extra fine diamond burs followed by abrasive cups and diamond pastes. These results are in agreement with Jung³² whose showed that IPS-Empress ceramic specimens were able to be polished to lower roughness values with a rubber polisher and diamond gel.³²

A study evaluated the effect of two polishing diamond pastes for ceramic polishing applied by four different vehicles a dental rubber cup, Robinson bristle brush, felt wheel, and buff discs and found no significant differences between the two pastes, but among vehicles the rubber cup resulted in the highest roughness average with a mean of 0.255 μm (R_a) the other groups were similar and showed a roughness average ranging from 0.087 to 0.119 μm .² Sasahara et al³³ found that the use of a polishing paste after the sandpaper discs or after the rubber wheel resulted in a reduction of the R_a value for ceramics. Rubber or discs followed by diamond paste were the best surface treatments for porcelains d.sign.³³

These results confirm that finish produced by intermediate components of the proprietary finishing kit did not totally reduce the roughness of the ceramic surface. It was necessary to complete the polishing sequence with diamond paste to achieve a surface which approached roughness characteristics of glazed porcelain.²⁹

Significant correlation was found between the roughness of the surface and the biaxial strength, the smoother the surface, the stronger the sample.¹⁰ Also, cracks in the porcelain originated from flaws are propagated with flexural pressure, resulting in lower flexural strength, which indicates that the increase in surface roughness of the porcelain can be interpreted as a reduction in flexural strength. The larger the surface roughness in the porcelain, the lower the flexural strength.¹¹ Then to achieve a less rough as possible surface also improve the physical and mechanical properties of the dental prosthesis.^{10,11}

By the other hand, when a porcelain-veneered ceramic restoration with a flaw on the surface is placed in the mouth, moisture may hasten the breakdown of bonds between silica atoms over time through a process called slow crack growth. Even if the restorations are not subject to excessive occlusal loading, fracture can occur due to static fatigue.⁹ Also, a lot of transitory fluids may interact with porcelain, including hydrogen peroxide from bleaching gels. According to Turker & Biskin^{14,15}, a significant decrease in porcelain microhardness was observed after 240 hours of treatment with 10% carbamide peroxide, and a spectral analysis of showed a decrease in the SiO₂ content, which is the main component of the matrix.^{14,15} Thus, its lower in content would affect other properties in long term. Some alterations were expected because the contact and possible diffusion of free radicals of H⁺ or H₃O⁺ produced by bleaching agents²⁰ that may selectively leach alkali ions and cause the dissolution of the ceramic glass network.¹ Then, the prolonged exposure of hydrogen peroxide could potentially affect dental porcelain exposed to at-home bleaching as showed by some studies. Since the refinished porcelain lost the glaze treatment it could be potentially affected by hydrogen peroxide based bleaching gels.

However, a stability on surface roughness of refinished ceramic against bleaching agents was observed in the present study and the second study hypothesis may be accepted, treatment with 16% carbamide peroxide bleaching agents used to at-home treatment do not affect the roughness surface of refinished ceramic. At our knowledge no other research evaluated the effect of bleaching treatment on a refinished ceramic, but these results are in agreement with Ourique et al³⁴ that found no statistical differences in the surface roughness of ceramics treated with 10 % or 16% carbamide peroxide for 126-h³⁴; and other studies which showed no significant changes in physical properties after treatment with 15% carbamide peroxide for 56-h, 6.5% hydrogen peroxide for 14-h, 38% hydrogen peroxide for 30 min or 45 min.¹⁷⁻¹⁹

Regardless of the type of ceramic or pretreatment, any adjusted on restoration should be reglazed or subjected to a refinishing sequence.³⁵ Since the ultimate goal of refinishing of a dental porcelain is the attainment of a well-polished surface as a substitute for glazed porcelain,⁷ and based on the results found in this study, it may be suggested that clinical refinishing of roughened ceramic surfaces after occlusal adjustment with diamond burs may be well obtained using fine and extra fine diamond burs followed by abrasive rubber tips and diamond paste.

CONCLUSION

Ceramic refinishing with fine and extra fine diamonds burs are not able to produce a smooth surface, but the following treatment with rubber cups and abrasive paste are efficient to peeling the groves and fissures and create a low roughness surface which may not be rough by bleaching treatment with 16 % carbamide peroxide.

CLINICAL SIGNIFICANCE

When necessary, ceramic restorations must be properly refinished with fine, extrafine, and rubber cups with polishing pate to achieve a smooth surface.

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Figure list

Figure 1- Scanning electron microscopic (SEM) photograph representative of the IPS d.Sign ceramic (70x magnification). A- ceramic surface after metallographic polishment. B- ceramic surface after adjustment with a diamond bur. C- ceramic surface after refinishing with fine and an extra fine diamond bur. D- ceramic surface after refinishing with first and second abrasive cups and after with polishing paste.

Figure 2- Scanning electron microscopic (SEM) photograph representative of the VMK 95 ceramic (70x magnification). A- ceramic surface after metallographic polishment. B- ceramic surface after adjustment with a diamond bur. C- ceramic surface after refinishing with fine and an extra fine diamond bur. D- ceramic surface after refinishing with first and second abrasive cups and after with polishing paste.

Graph 1- Surface roughness (Ra) of each ceramic as a function of surface treatment.

Tables

Table 1 – Ceramic materials used in this study: commercial brand, lot, type, and chemical characterization*.

Ceramic (Lot number)	Bleaching	n	Type	Chemical characterization*
IPS d.Sign (Lot: K33292)	NB	7	Fluorapatite-leucite glass-ceramic	SiO ₂ ; BaO; Al ₂ O ₃ ; CaO; CeO ₂ ; Na ₂ O; K ₂ O; B ₂ O ₃ ; MgO; ZrO ₂ ; P ₂ O ₅ ; F; Li ₂ O; TiO ₂ ; SrO; ZnO; and pigments
	BL	7		
VMK 95 (Lot: 26590)	NB	7	Feldspathic ceramic	Al ₂ O ₃ ; BaO; B ₂ O ₃ ; CaO; Fe ₂ O ₃ ; MgO; SiO ₂ ; TiO ₂ ; ZrO ₂ ; CeO ₂ ; Li ₂ O; K ₂ O; Na ₂ O; Glycerine; Butylene Glycol; Tin Oxide.
	BL	7		

* Material Safety Data Sheet; Abbreviations: SiO₂: Silicon Oxide; BaO: Barium Oxide; Al₂O₃: Aluminum oxide; CaO: Calcium Oxide; CeO₂: cerium dioxide; Na₂O: Sodium Oxide; K₂O: Potassium Oxide, B₂O₃: Boron Oxide; MgO: Magnesium Oxide; ZrO₂: Zirconium Oxide; P₂O₅: Phosphorus pentoxide; F: Fluor; Li₂O: Lithium Oxide; TiO₂: Titanium Dioxide; SrO: Strontium oxide; ZnO: Zinc oxide; Fe₂O₃: Iron Oxide.

Table 2- Surface roughness (Ra) of each ceramic and standard deviations (in brackets) at each evaluation period after surface treatment, and the results of Tukey's test for ceramics.

Surface Treatment	IPS d.Sign NB	IPS d.Sign BL	VMK 95 NB	VMK 95 BL	Ceramics
Baseline	0.142 (±0.018)	0.164 (±0.037)	0.237 (±0.049)	0.280 (±0.093)	0.206 (±0.077) A
Adjustment procedure	2.339 (±0.391)	2.751 (±0.610)	2.134 (±0.635)	2.503 (±0.760)	2.432 (±0.622) C
F/FF diamond burs	0.919 (±0.098)	1.059 (±0.163)	0.876 (±0.141)	0.911 (±0.152)	0.940 (±0.150) B
Abrasive cup/paste	0.337 (±0.052)	0.339 (±0.040)	0.359 (±0.084)	0.317 (±0.025)	0.338 (±0.054) A

Different letters indicate statistical significant differences among surface treatments (line).

Table 3- Surface roughness (Ra) of each ceramic and standard deviations (in brackets) after bleaching treatment.

Bleaching Treatment

Ceramics	NB	BL
IPS d.Sign	0.341 (±0.080)	0.350 (±0.078)
VMK 95	0.321 (±0.080)	0.372 (±0.091)

4. Considerações Finais

A estabilidade química é uma das principais propriedades necessárias para uma cerâmica odontológica. A degradação da cerâmica pode levar a um maior desgaste das estruturas dentais, liberação de componentes radioativos, aumento da adesão bacteriana e liberação de substâncias tóxicas (Anusavice, 1992). Esta pode ocorrer devido a incidência de forças mecânicas ou contato com substâncias químicas (Anusavice, 1992). Mesmo sendo considerado o material restaurador odontológico mais inerte, as cerâmicas podem exibir deterioração superficial quando expostas a soluções ácidas pela dissolução da rede de vidro, como por exemplo pela exposição ao flúor acidulado (Anusavice, 1992; Kukiattrakoon & Thammasitboon, 2007).

De acordo com Anusavice (1992) dois mecanismos dominantes são responsáveis pela corrosão dos vidros de silicato nas cerâmicas sendo esses a liberação seletiva de íons alcalinos e a dissolução da rede de vidro, principalmente dos íons metálicos, que são fortemente influenciados por radicais livres (ácidos) de H^+ e H_3O^+ . Estes radicais livres podem ser liberados como subprodutos dos agentes clareadores que dessa forma podem causar alterações nas cerâmicas odontológicas.

Mesmo considerada segura, é indispensável que a técnica de clareamento caseiro seja corretamente indicada e que os pacientes sejam supervisionados e orientados pelos cirurgiões-dentistas (Haywood & Heymann, 1989), devido ao risco de alterações sub-clínicas que podem ocorrer na micromorfologia do esmalte, podendo levar a redução na microdureza, aumento da rugosidade e formação de trincas e porosidades (Seghi & Denry, 1992; Wandera et al., 1994, Gurgan et al., 1997; Oltu & Gürgan, 2000; Rodrigues et al., 2001; Türkun et al., 2002; Basting et al., 2003; Worschech et al., 2003 Hosoya et al., 2003, Rodrigues et al., 2005; Worschech et al., 2006) que também podem ser observados nos materiais restauradores diretos (Campos et al. 2003; Al-Salehi et al., 2007; Al-Salehi et al., 2006; Gurgan & Yalcin, 2007; Yu et al. 2008) e indiretos como as cerâmicas (Turker & Biskin, 2003; Butler et al., 2004; Schemehorn et al., 2004; Moraes et al. 2006; Polydorou et al., 2006).

Butler et al. em 2004 demonstraram que as cerâmicas de baixa fusão podem sofrer alterações na rugosidade superficial após o tratamento com peróxido de carbamida 10% por 48h.

Moraes et al., em 2006, realizaram um estudo do efeito do peróxido de carbamida a 10% e 35% sobre a rugosidade superficial do esmalte dental, de resinas compostas (micropartículas e microhíbridas) e de uma porcelana feldspática e constataram que o peróxido de carbamida a 10% aplicado diariamente por 3h causou alterações na rugosidade superficial da cerâmica após 21 dias de tratamento. Já o peróxido de carbamida 35% aplicado semanalmente durante 30 minutos pelo mesmo período causou aumento estatisticamente significativo na rugosidade superficial da cerâmica, das resinas e do esmalte dental.

No presente trabalho nenhum grupo demonstrou alterações na microdureza ou rugosidade superficiais durante o tratamento clareador. Polydorou et al. em 2006, demonstraram por microscopia eletrônica de varredura (MEV) que a aplicação de peróxido de carbamida 38% por 45 minutos sobre uma cerâmica não causou alterações superficiais. Schemehorn et al. (2004) avaliaram amostras de uma cerâmica feldspática em MEV após a aplicação de peróxido de hidrogênio 6% por 20 minutos e não notaram alterações na morfologia superficial. Silva et al. em 2006 que relataram não haver alterações significativas na superfície de cerâmicas após o tratamento clareador *in situ*. Turker & Biskin (2003), não observaram aumento na rugosidade superficial de cerâmicas feldspáticas após o tratamento com peróxido de carbamida 10% por 8 horas diárias durante 30 dias, porém, notaram uma perda no conteúdo de óxido de silício (SiO_2) que pode resultar em perda de propriedades físico-mecânicas para a cerâmica.

Turker & Biskin (2002) relataram diminuição na microdureza de cerâmicas feldspáticas após a aplicação de peróxido de carbamida 10 a 16% por 8 horas diárias durante 4 semanas. Tanto Turker & Biskin, em 2002, quanto Moraes et al. em 2006 sugerem que as alterações causadas pelos peróxidos nas cerâmicas feldspáticas são devido a perda de componentes estruturais. Turker (1999) através de uma microanálise por dispersão de raio

X demonstrou uma redução de 4,8% no conteúdo de SiO_2 da superfície de uma cerâmica feldspática após o tratamento com sistemas clareadores caseiros, o que supostamente pode estar relacionado com as alterações descritas.

Em relação às alterações de componentes químicos na estrutura de uma cerâmica feldspática, Turker & Biskin, em 2003, demonstraram uma diminuição média de 1% de SiO_2 e K_2O_2 após o tratamento clareador caseiro, porém, nesse mesmo estudo não encontraram alterações na rugosidade superficial da cerâmica. Divergindo destes resultados, o presente estudo não apresentou diferenças estatisticamente significantes de microdureza após o tratamento clareador com peróxido de carbamida 10% ou 16% das cerâmicas feldspáticas em estudo, mesmo após 126 horas. Resultados similares aos presentes podem ser observados em estudos como o de Polydorou et al. (2006), que avaliaram o efeito do clareamento caseiro com peróxido de carbamida 15% por 56 horas e do peróxido de hidrogênio 38% por 30 minutos na técnica de consultório sobre superfície de uma cerâmica feldspática e não houveram alterações na microdureza superficial.

Corroborando ainda com os resultados do presente estudo estão os obtidos por Duschner et al. (2004) que após o tratamento com peróxido de hidrogênio 6,5% por um período de 14 horas não observaram alteração na microdureza de uma cerâmica feldspática e de Polydorou et al. (2007) que em um estudo *in vitro* também notaram que o clareamento de consultório com peróxido de hidrogênio 38% por 15, 30 e 45 minutos, não alterou a microdureza de uma cerâmica feldspática.

Silva et al., em 2006, relataram ainda que não ocorreram alterações na rugosidade superficial de uma cerâmica feldspática após o tratamento com dois sistemas clareadores, peróxido de carbamida 18% (Colgate Simply-White) e perborato de sódio sobre a rugosidade superficial da cerâmica, os resultados mostraram que não houveram alterações estatisticamente significantes.

Assim, observa-se que apesar de poucos estudos na literatura os resultados ainda são inconclusivos e a transposição destes resultados obtidos *in vitro* para a realidade clínica é uma questão extremamente delicada. Frente ao tratamento com peróxidos, clinicamente as cerâmicas podem ser mais resistentes comparadas a outros materiais restauradores e ao esmalte dental. Apesar dos resultados encontrados na literatura que demonstram alterações de microdureza e rugosidade superficial, deve-se questionar se estas alterações microscópicas podem levar a necessidade da substituição de cerâmicas odontológicas desde que estejam bem adaptadas e em consonância com a cor obtida após o tratamento clareador. Esta questão foi levantada por Turker, em 1999, que observou a perda de SiO₂ e julgou que clinicamente seus efeitos não seriam significativos.

Dessa forma, pode-se concluir através dos resultados obtidos e apoiados pela literatura que a exposição de cerâmicas aos sistemas clareadores a base de peróxido de carbamida 10% ou 16% não causam alterações que exijam a substituição das mesmas.

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Anexo 1 – Artigo publicado no periódico Am J Dent

Research Article

Effect of different concentrations of carbamide peroxide on microhardness of dental ceramics

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ABSTRACT: Purpose: To evaluate the effect of 10% and 16% carbamide peroxide bleaching agents on microhardness of dental ceramics after different periods of bleaching treatment. **Methods:** 15 specimens with 5 x 3 x 1 mm³ were created with four dental ceramics following manufacturers' instructions: IPS Classic (Ivoclar-Vivadent); IPS d.Sign (Ivoclar-Vivadent); EX3 (Noritake); VMK-95 (Vita). A microhardness tester with a Knoop diamond with a 100 g load was used to evaluate the baseline microhardness values of all ceramics. Afterwards, the specimens were submitted to 6-hour daily bleaching treatments with 10% or 16% carbamide peroxide (Whiteness Perfect - FGM) for 21 days, while control groups from each ceramic system were maintained in artificial saliva. The microhardness of all groups was evaluated at 18, 42, 84, and 126 hours of bleaching treatment. The mean value of five indentations performed at each specimen in each time was obtained and all data were submitted to two-way repeated measures ANOVA and Tukey's post-hoc test ($\alpha=0.05$). **Results:** No significant differences in ceramic microhardness were observed among either bleaching intervals or bleaching treatments. Ceramic restorations are not affected by carbamide peroxide 10% or 16% gel during bleaching treatment. (*Am J Dent* 2011;24:57-59).

CLINICAL SIGNIFICANCE: This study provided evidence that at-home bleaching systems do not cause detrimental effects on dental ceramics.

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Introduction

The best treatment for discolored vital teeth is dental bleaching. The most indicated bleaching technique is that performed at-home, which presents effective results in a few weeks.¹ First described by Haywood & Heymann¹² in 1989, the so-called nightguard dental bleaching involves the day or night use of a tray with carbamide peroxide for 2-8 hours a day.

Although at-home bleaching is widely used, this technique may lead to clinical side effects due to the reactive nature of the hydrogen peroxide, so patients may experience dentin sensibility and/or gingival irritation.^{1,3,5} Microscopically, several alterations are also expected in the enamel morphology due to mineral loss and surface roughening.⁶⁻¹⁵

Such alterations on tooth tissues are related to the low pH of hydrogen peroxide and to its decomposition into H⁺ free radicals, which are extremely unstable and reactive.^{6,7,9-16} Although conventional dental ceramics are considered the most inert of all dental materials used for dental restorations, the surfaces of dental porcelains can exhibit surface deterioration in contact with acidulated fluoride gels or solutions.¹⁷ Also, selective leaching of alkali ions and dissolution of the glass network of ceramic may occur by the diffusion of free radicals of H⁺ or H₂O⁺. As hydrogen peroxide releases a great amount of free radicals that may potentially affect dental porcelain exposed accidentally or not to bleaching gel during treatment, the present study evaluated the effect of 10% and 16% carbamide peroxide bleaching agents on microhardness of dental ceramics after different time periods of bleaching treatment.

Materials and Methods

The microhardness of four dental ceramics EX-3,^a IPS Classic,^b IPS d.Sign,^b and VMK 95^c were evaluated in a research protocol including a factorial design to test the effects

of three surface treatments: 10% carbamide peroxide (Whiteness Perfect^d); 16% carbamide peroxide (Whiteness Perfect^e); and no treatment (control group); at five treatment periods: 0 (before treatment), 18, 42, 84, and 126 hours.

Fifteen specimens with 5 x 3 x 1 mm³ of each ceramic were prepared according to manufacturers' instructions and had their surfaces sequentially polished with diamond polishing pastes of 6, 3, 1, and 0.5 μ m and polishing cloths with mineral oil lubricant (top, Gold and Ramf).

Microhardness test was performed by a single evaluator prior to and after the bleaching treatment with Knoop indenter with load of 200 g applied for 5 seconds. As recommended by Siew,¹⁸ five indentations were evaluated at each interval. The 0 hour indentations were performed at a distance of 30 μ m between each other in the center of the ceramic specimens. In the following intervals, the five indentations were performed 100 μ m distant from and on the left of the previous indentations.

The 15 ceramic specimens were randomly divided into three groups according to the surface treatments, having five specimens each (n= 5). The respective treatment agent was applied for 6 hours a day during 21 days, corresponding to 126 hours of treatment. Specimens were covered with 0.03 ml of the bleaching agent and a drop of artificial saliva,^{6-8,10} except the untreated specimens, which received only the artificial saliva. The specimens were placed in vacuum-formed custom trays^{8,14} and stored in a closed plastic container at 37°C.

The indentation lengths from each specimen in each interval were measured in micrometers, and transformed into Knoop hardness number (KHN). The mean of the five Knoop hardness (KHN) values obtained from each specimen either before or following the treatment were statistically analyzed by two-way repeated measures ANOVA and Tukey's post-hoc test at a 5% level of significance within each ceramic.^{6,8}

Table. Mean KHN values of each ceramic (standard deviation) at each evaluation interval.

Ceramic	Surface treatment	0 hours	18 hours	42 hours	84 hours	126 hours
EX-3	Control	491.3 (12.6)	500.8 (4.5)	499.0 (10.2)	505.4 (15.9)	500.7 (1.9)
	PC10%	515.2 (34.3)	510.7 (23.0)	504.4 (18.0)	508.7 (6.4)	509.0 (13.3)
	PC16%	517.0 (10.2)	520.5 (17.8)	516.2 (19.9)	527.6 (23.0)	520.4 (39.3)
IPS d.Sign	Control	484.5 (9.5)	499.6 (16.3)	503.3 (12.9)	516.7 (7.1)	505.3 (9.9)
	PC10%	491.3 (41.8)	511.1 (21.7)	516.7 (21.4)	517.1 (19.9)	515.9 (16.6)
	PC16%	513.8 (35.3)	510.3 (17.4)	498.2 (23.3)	503.5 (17.9)	511.5 (36.3)
VMK 95	Control	534.2 (30.4)	524.5 (15.7)	524.4 (12.8)	531.5 (15.8)	524.0 (8.9)
	PC10%	533.6 (19.0)	532.7 (8.6)	529.0 (8.3)	532.0 (24.8)	538.8 (18.2)
	PC16%	524.5 (22.5)	523.7 (32.1)	524.6 (20.4)	530.6 (21.9)	536.9 (34.4)
IPS Classic	Control	499.4 (4.6)	515.1 (12.0)	509.4 (27.5)	495.6 (11.0)	492.4 (10.0)
	PC10%	483.7 (4.4)	498.5 (20.4)	494.1 (20.5)	498.8 (13.1)	501.8 (19.6)
	PC16%	494.9 (20.3)	489.4 (20.3)	484.9 (12.8)	489.0 (9.0)	491.9 (13.3)

Results

The mean KHN values of each ceramic before and after the treatment with the respective standard deviations are shown in the Table. No significant difference in KHN values was observed between the control group and bleached groups, as well as between groups treated with 10% carbamide peroxide and those treated with 16% carbamide peroxide, regardless of time. Moreover, no significant difference in KHN values was observed among time intervals, regardless of treatment.

Discussion

Chemical resistance is the main property expected from ceramics for intra-oral use, since dental prostheses must withstand degradation in the presence of a wide range of solutions with variable pH.¹⁷ The integrity of a ceramic is determined by its ability to avoid possible side-effects such as increased plaque adhesion, release of potentially toxic species as a result of wear, release of radioactive components, and increased abrasion of opposing dental structures.¹⁷

The ceramics evaluated in the current study did not show statistical differences in microhardness values after 126 hours of exposure to carbamide peroxide at the concentrations of 10% or 16%, demonstrating to be resistant *in vitro* to dental bleaching. The bleaching protocol used in the present study was similar to that of other studies^{6,8-10,14} evaluating the *in vitro* effect of bleaching systems on the enamel surface microhardness through time.

Results similar to those from the current study were observed in several studies, in which no significant changes in microhardness values were found when ceramics were treated with 15% carbamide peroxide for 56 hours,¹⁹ 6.5% hydrogen peroxide for 14 hours,²⁰ 38% hydrogen peroxide for 30 minutes¹⁹ or 45 minutes.²¹

Despite the high ceramic stability, some degradation in ceramic materials was expected in the present study because of the interaction of free radicals released from the bleaching gels with the ceramic glass network, leading to the loss of alkali metal ions from the glass surface. The loss of alkali ions from ceramic material could also occur due to the low pH of bleaching gels, which could also probably decrease microhardness, but such effect was not observed in the four different commercial brands of dental ceramic from the beginning throughout the 126 hours of bleaching treatment.

The 126 hours of treatment was chosen to simulate 21-day

nightguard bleaching treatment and most patients achieve best results within this period. However, bleaching treatments may be extended to longer treatment periods in patients with severe discoloration, and as bleaching detrimental effects are time dependent, more intense mineral loss is expected on enamel and dentin in extended treatments. A time-dependent effect of bleaching treatment on ceramic microhardness may also be suggested if the results of 126 hours of treatment from the present study are compared with those from Turker & Biskin²² of 240 hours of treatment. These authors showed a statistically significant decrease in ceramic microhardness after 240 hours of treatment with 10% or 16% carbamide peroxide. Therefore, the ceramic material may suffer some degradation after long periods of bleaching treatment. In addition, Turker & Biskin²² in the next year, performed surface spectral analyses in ceramics treated for 240 hours with 10% carbamide peroxide and found a decrease in the SiO₂ content, which is the main component of the matrix and for this reason its lower content would affect the surface microhardness.²³ However, the same authors demonstrated that the bleaching gels affected only surface roughness, then the small amount of released SiO₂ was not considered to be of clinical significance.²³ Also, some studies²⁴⁻²⁶ showed alterations on ceramic surface after bleaching treatment by scanning electron microscopy and roughness profiles, but concluded that these alterations were clinically insignificant.

The degradation of dental ceramics generally occurs because of chemical attack, mechanical forces or a combination of these effects.¹⁷ In the current study only the chemical attack of ceramics by 10% or 16% hydrogen peroxide was considered, but different results could be found if mechanical forces were employed since it could weaken the structure by creating surface flaws and increase the susceptibility of ceramic to sequential bleaching attack. More studies are needed to evaluate this factor.

The present study showed that ceramic dental materials were not affected by 10% or 16% carbamide peroxide treatment, so there is no need for ceramic replacement in clinical situations where ceramic restorations were accidentally exposed to bleaching gels, once color, form and function are clinically acceptable.

- Noritake Kikai Co., Ltd, Aichi, Japan.
- Ivoclar Vivadent AG, Schaan, Liechtenstein.
- Vita Zahnfabrik, Bad Säckingen, Germany.
- Whiteness FGM, FGM, Joinville, Santa Catarina, Brazil.
- Anotec Ind Com Lda., Cotia, Sao Paulo, Brazil.

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Anexo 2 – Artigo publicado no periódico BOR



Restorative Dentistry

Effects of different concentrations of carbamide peroxide and bleaching periods on the roughness of dental ceramics

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Abstract: The wide use of dental bleaching treatment has brought concern about the possible effects of hydrogen peroxide on dental tissue and restorative materials. The objective of this study was to evaluate *in vitro* the effect of nightguard bleaching on the surface roughness of dental ceramics after different periods of bleaching treatment. Fifteen specimens of $5 \times 3 \times 1$ mm were created with three dental ceramics following the manufacturers' instructions: IPS Classic (Ivoclar-Vivadent); IPS d.Sign (Ivoclar-Vivadent); and VMK-95 (Vita). A profilometer was used to evaluate baseline surface roughness (Ra values) of all ceramics by five parallel measurements with five 0.25 mm cut off (λ_c) at 0.1 mm/s. Afterwards, all specimens were submitted to 6-h daily bleaching treatments with 10% or 16% carbamide peroxide (Whiteness-FGM) for 21 days, while control groups from each ceramic system were stored in artificial saliva. The surface roughness of all groups was evaluated after 18 h, 42 h, 84 h, and 126 h of bleaching treatment. The surface roughness of each specimen ($n = 5$) was based on the mean value of five parallel measurements in each time and all data were submitted to two-way repeated measures ANOVA and Tukey's post-hoc test ($\alpha = 0.05$). No significant differences in ceramic surface roughness were observed between untreated and bleached ceramic surfaces, regardless of bleaching intervals or bleaching treatments. This study provided evidence that at-home bleaching systems do not cause detrimental effects on surface roughness of dental ceramics.

Descriptors: Esthetics, Dental; Tooth Bleaching; Hydrogen Peroxide; Surface Properties; Ceramics.

Declaration of Interests: The authors certify that they have no commercial or associative interest that represents a conflict of interest in connection with the manuscript.

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Introduction

In recent years, dental bleaching has become popular and often requested by patients wanting to improve their teeth shade. The most useful and effective bleaching technique is the one performed at home, in which bleaching of all the teeth is undertaken over two weeks, with few side effects such as dental sensitivity.¹ This technique was firstly described by Haywood & Heymann in 1989 as nightguard dental bleaching, but today this technique may be performed at home from one to eight hours a day, involving the day or nighttime use of a tray with a bleaching agent.^{1,2}

The most commonly used dental bleaching agent is carbamide per-

■ Effects of different concentrations of carbamide peroxide and bleaching periods on the roughness of dental ceramics

oxide. The reaction of carbamide peroxide with the teeth releases hydrogen peroxide and free radicals, which are responsible for dental bleaching.^{3,4} Despite the wide approval of at-home bleaching techniques, the use of peroxides may lead to clinical side effects due to the reactive nature of hydrogen peroxide, so patients may experience dentin sensitivity and/or gingival irritation.^{1,3-5} Several microscopic changes on the enamel surface morphology are also observed, due to enamel mineral loss and surface roughening.⁶⁻¹⁵

Extremely unstable and reactive H[•] free radicals, released by bleaching agents, and low pH are described as the main cause of the side effects of prolonged use of these products.^{6-24,16-17} Similarly, bleaching agents may cause structural changes on restorative materials that may compromise their physical properties and lead to premature failure.¹⁴⁻²⁴ Although conventional dental ceramics are considered the most inert among all dental restorative materials, their surfaces can exhibit surface deterioration in contact with acidulated fluoride gels or other solutions.²⁵ In addition, the contact and possible diffusion of free radicals of H[•] or H₂O[•] produced by bleaching agents¹⁷ may selectively leach alkali ions and cause dissolution in ceramic glass networks.²⁵ Thus, prolonged exposure to hydrogen peroxide through at-home bleaching treatment may potentially affect dental porcelain and may produce alterations on the porcelain's surface.¹⁶ Moreover, an increase in surface roughness greater than the threshold of Ra = 0.2 μm may result in an increase

in plaque accumulation, thereby increasing the risk of both secondary caries and periodontal inflammation²⁶ or affecting ceramic aesthetics by changing the ceramic texture.¹⁶

The aim of the present study was to evaluate the effect of 10% and 16% carbamide peroxide nightguard bleaching agents on the surface roughness of dental ceramics after different time periods of bleaching treatment. Thus, the hypothesis of the present study was that the surface roughness of ceramic might be modified by exposure to 10% and 16% carbamide peroxide bleaching agents used in at-home treatment for a period of 126 h.

Methodology

The surface roughness of three dental ceramics (Table 1) – one fluorapatite glass-ceramic, IPS d.Sign (Ivoclar Vivadent AG - Schaan, Liechtenstein), and two feldspathic ceramics, IPS Classic (Ivoclar Vivadent AG - Schaan, Liechtenstein), and VMK 95 (Vita Zahnfabrik - Bad Säckingen, Germany) – were evaluated in a research protocol, including a factorial design to test the effects of three surface treatments: 10% carbamide peroxide (Whiteness FGM, Joinville, Brazil; pH ≈ 6.0); 16% carbamide peroxide (Whiteness FGM, Joinville, Brazil; pH ≈ 6.0); and no treatment (control group); at five periods of treatment: 0 h (baseline), 18 h, 42 h, 84 h, and 126 h.

Fifteen specimens with 5 × 3 × 1 mm of each ceramic were prepared according to the manufacturers' instructions and their surfaces were sequentially

Table 1 - Ceramic materials used in this study: commercial brand, lot, type, and chemical characterization*.

Ceramic (Lot number)	Type	Chemical characterization*
IPS d.Sign (Lot: K33292)	Fluorapatite-leucite glass-ceramic	SiO ₂ ; BaO; Al ₂ O ₃ ; CaO; CeO ₂ ; Na ₂ O; K ₂ O; B ₂ O ₃ ; MgO; ZrO ₂ ; P ₂ O ₅ ; F; Li ₂ O; TiO ₂ ; SrO; ZnO; and pigments
VMK 95 (Lot: 26590)	Feldspathic ceramic	Al ₂ O ₃ ; BaO; B ₂ O ₃ ; CaO; Fe ₂ O ₃ ; MgO; SiO ₂ ; TiO ₂ ; ZrO ₂ ; CeO ₂ ; Li ₂ O; K ₂ O; Na ₂ O; glycerin; butylene glycol; tin oxide
IPS Classic (Lot: K02827)	Feldspathic ceramic	SiO ₂ ; BaO; Al ₂ O ₃ ; CaO; CeO ₂ ; Na ₂ O; K ₂ O; B ₂ O ₃ ; MgO; ZrO ₂ ; P ₂ O ₅ ; TiO ₂ ; and pigments

* Material Safety Data Sheet; Abbreviations: SiO₂: Silicon Oxide; BaO: Barium Oxide; Al₂O₃: Aluminum Oxide; CaO: Calcium Oxide; CeO₂: Cerium Dioxide; Na₂O: Sodium Oxide; K₂O: Potassium Oxide; B₂O₃: Boron Oxide; MgO: Magnesium Oxide; ZrO₂: Zirconium Oxide; P₂O₅: Phosphorus Pentoxide; F: Fluorine; Li₂O: Lithium Oxide; TiO₂: Titanium Dioxide; SrO: Strontium Oxide; ZnO: Zinc Oxide; Fe₂O₃: Iron Oxide.

polished with diamond polishing pastes of 6, 3, 1, and 0.5 μm and polishing cloths with mineral oil lubricant (Top, Gold and Ram, Arotec Ind. Com. Ltda., Cotia, Brazil).

Surface roughness was evaluated by a single blinded evaluator prior to and after all bleaching periods. A profilometer (TR200, Time Group Inc., Beijing, China), with a microneedle, was used to scan the specimen surfaces to determine the parameter of average surface roughness (Ra). Five points were initially marked to ensure repeatable measurements. From these points, five parallel measurements in a longitudinal direction were performed on each specimen surface, with a 0.25 mm cut off (λ_c) at 0.1 mm/s. Surface roughness was recorded, and mean roughness (Ra expressed in μm) was determined for each specimen before and after each treatment period.

Fifteen ceramic specimens were randomly divided into three groups according to surface treatments ($n = 5$). The respective treatment agent was applied for six hours a day over 21 days, corresponding to 126 hours of treatment. Specimens to be bleached were covered with 0.03 ml of the respective bleaching agent, were placed in vacuum-formed custom trays with a drop of artificial saliva,^{4,13} and were stored in a plastic container at 37 °C.⁴⁻⁸ Specimens from control groups were stored only with artificial saliva drops in the vacuum-formed custom tray to mimic oral conditions.

After bleaching exposure, specimens were

washed with distilled water to remove residual carbamide peroxide gel and were stored in a plastic container for the remaining day period in relative humidity at 37 °C.

Surface roughness was measured at 18 h, 42 h, 84 h, and 126 h after the beginning of the experiment, after the specimens were washed and dried. Data of each ceramic material were statistically analyzed by two-way repeated measures ANOVA and Tukey's post-hoc test at a 5 % level of significance within each period, using statistical software (SAS 8.0 for Windows; SAS Institute Inc., Cary, USA).

Results

Mean Ra values of each ceramic material before and after treatment, with the respective standard deviations, are shown in Table 2. No significant differences in Ra values were observed between the control group and bleached groups, as well as between groups treated with 10% carbamide peroxide and those treated with 16% carbamide peroxide, regardless of time. Moreover, no significant differences in Ra were observed among time intervals, regardless of treatment. Figure 1 shows the surface roughness of each ceramic treated with 10% or 16% carbamide peroxide as a function of time.

Discussion

Ceramics are expected to be chemically stable in the mouth, as dental prostheses must withstand degradation in the presence of a wide range of solutions

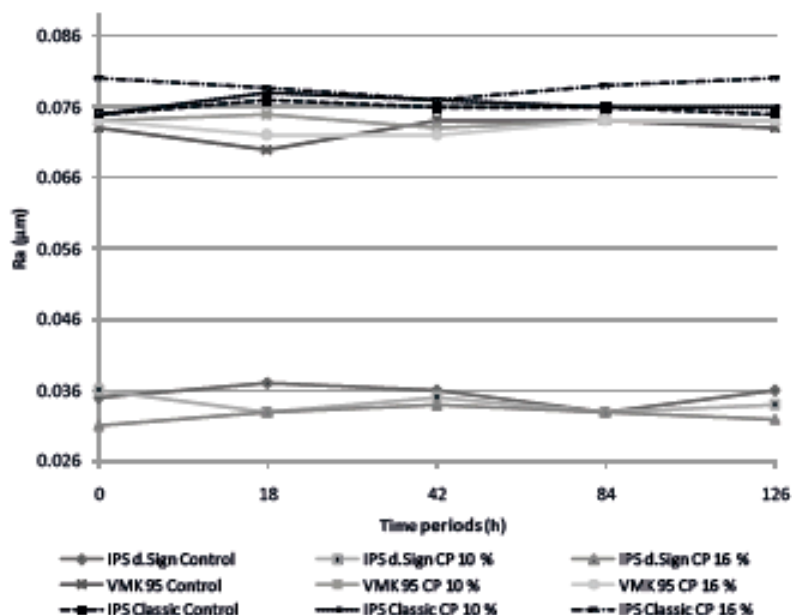
Table 2 - Surface roughness (Ra) of each ceramic and standard deviation (SD) at each evaluation period.

Ceramic	Surface treatment	0 h	18 h	42 h	84 h	126 h
IPS d.Sign	Control	0.035 ± 0.001	0.037 ± 0.002	0.036 ± 0.003	0.033 ± 0.004	0.036 ± 0.002
	CP 10%	0.036 ± 0.003	0.033 ± 0.002	0.035 ± 0.002	0.033 ± 0.002	0.034 ± 0.003
	CP 16%	0.031 ± 0.002	0.033 ± 0.003	0.034 ± 0.004	0.033 ± 0.001	0.032 ± 0.004
VMK 95	Control	0.073 ± 0.002	0.070 ± 0.004	0.074 ± 0.002	0.074 ± 0.002	0.073 ± 0.003
	CP 10%	0.074 ± 0.003	0.075 ± 0.004	0.073 ± 0.003	0.074 ± 0.003	0.074 ± 0.003
	CP 16%	0.074 ± 0.003	0.072 ± 0.004	0.072 ± 0.003	0.074 ± 0.005	0.074 ± 0.002
IPS Classic	Control	0.075 ± 0.002	0.077 ± 0.002	0.076 ± 0.001	0.076 ± 0.002	0.075 ± 0.002
	CP 10%	0.075 ± 0.004	0.078 ± 0.004	0.077 ± 0.003	0.076 ± 0.004	0.076 ± 0.003
	CP 16%	0.080 ± 0.004	0.0786 ± 0.004	0.077 ± 0.002	0.079 ± 0.003	0.080 ± 0.002

No significant difference in surface roughness was noted among the groups or treatment periods ($p > 0.05$); CP: carbamide peroxide.

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Figure 1 - Mean surface roughness (Ra) of each ceramic as a function of time.



with variable pH levels.²⁵ Otherwise, ceramics could release potentially toxic substances and radioactive components and exhibit increased wear, abrasion of opposing dental structures, and increased plaque adhesion after exposure to such intra-oral challenges.²⁵

This study tested the effects of dental bleaching agents on the surface roughness of ceramic specimens with standardized initial roughness averages less than 0.2 µm, a condition that leads to bacterial accumulation similar to that observed on the least rough surface.²⁶ Therefore, any possible change in roughness due to *in vitro* treatment would be detected by contact profilometry.^{11,12,16,26} No significant differences in ceramic roughness were observed after 126 hours of exposure to 10% or 16% carbamide peroxide in comparison to the baseline values, demonstrating that all products were inert *in vitro* to dental bleaching, so the hypothesis of this study was rejected. Our results corroborate those of Zavaneli *et al.*,²⁷ who found no alterations on ceramic surfaces treated with 10% or 15% carbamide peroxide for 126 h. Therefore, accidental exposure of dental ceramics to bleaching agents does not change their surface roughness to values capable of

increasing the risk for both secondary caries and periodontal inflammation.

The bleaching protocol used in this study was similar to that used in other studies, which aimed to evaluate *in vitro* the effect of bleaching systems on enamel surface roughness over time.^{6,8,9,13} Although an increase in roughness has been observed in composite resins or glass ionomers after bleaching treatment,^{18-24,27} no alteration in ceramic surfaces was observed after bleaching in the current study,²³ so the impact of bleaching agents on surface roughness may be considered material-dependent, as also demonstrated by previous studies.^{16,20-23} In these studies, the chemical stability of ceramics against bleaching agents was observed after treatment with 15% carbamide peroxide for 56 h,²³ 16% carbamide peroxide for 126 h,²⁷ 10% or 15% carbamide peroxide²⁷ and 38% hydrogen peroxide for 30 minutes²³ or 45 minutes, respectively.²⁴

The 126-hour bleaching protocol was chosen to simulate 21-day nightguard bleaching treatment, as most patients achieve the best results within this period.^{4,5} Although this period may be considered optimal, bleaching treatment may be extended to longer treatment periods in patients with severe dis-

coloration. As the detrimental effects of bleaching treatment are time-dependent, more mineral loss is expected on enamel and dentin surfaces in extended treatments.^{3,6,24} Therefore, despite the lack of changes in ceramic surface roughness from the beginning throughout 126 h of bleaching treatment, it is possible that overexposure to bleaching agents for longer than 126 h might lead to some degradation in ceramics due to the interaction between free radicals released from the bleaching gels and the ceramic glass network, which leads to the loss of alkali metal ions from the glass surface. However, only further evaluation, comprising longer exposure to bleaching agents, would confirm such speculation. Thus, a time-dependent effect of bleaching treatment on ceramic roughness should not be discarded because only one study observed a statistically significant decrease in auto-glazed ceramic roughness after treatment with 35% and 15% carbamide peroxide for 56 h followed by acid fluoride gel treatment for 30 h, which was probably due to a mild etching of the ceramic caused by a carbamide peroxide agent with the additive effect of fluoride gel.¹⁴ Furthermore, an energy-dispersive x-ray microanalysis of ceramic surfaces exhibited a decrease in SiO₂ content, which is the main matrix component.²¹ Thus, its lower content would affect other properties, such as surface microhardness, although the study found no significant difference in roughness. In addition, Polydorou *et al.* (2006)²⁴ showed that the effect of bleaching on surface texture was material- and time-dependent, as polished ceramic surfaces exposed to 38% hydrogen peroxide for 45 minutes showed slight changes in surface texture evaluated by scanning electron microscopy, while no significant difference was noted when ceramic surfaces were exposed to 15% carbamide peroxide for 56 h.²⁴

However, other authors have demonstrated that bleaching gels affected the surface roughness of dental ceramics. A statistically significant increase occurred in the surface roughness of ceramic material after 21 days of daily application of 10% carbamide

peroxide and a weekly application of 35%, although no alterations in roughness were observed over seven and 14 days of bleaching. According to the authors, these results were related to the leaching of components from the porcelain matrix as a function of continuing peroxide application. However, all Ra values were within the clinically acceptable range (Ra values of 0.22 to 0.24), and the changes would most likely be clinically insignificant. In addition, scanning electron microscopy analyses showed surface changes on ceramic surfaces after bleaching treatment, but the authors described them as clinically insignificant.²⁸⁻³⁰

The degradation of dental ceramics generally occurs because of chemical attacks, mechanical forces or a combination of these effects.²⁵ In the current study, only a chemical attack of ceramics by 10% or 16% hydrogen peroxide was considered, but different results might be found if mechanical forces were applied because they could weaken the structure by creating surface cracks and increasing ceramic susceptibility to sequential bleaching attacks. For this reason, further studies are required to evaluate this clinical challenge.

In this regard, the present study showed that ceramic dental materials were not affected by 10% or 16% carbamide peroxide treatment, so there is no need for ceramic polishing or replacement in clinical situations in which ceramic restorations are accidentally exposed to bleaching gels, provided that shade, shape, and function are clinically acceptable.

Conclusion

Within the study limitations, the surface roughness of all evaluated dental ceramics was not affected by treatment with 10% or 16% carbamide peroxide for 126 hours.

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Anexo 3 – Esquema de confecção dos corpos-de-prova.

