



VERA LÚCIA SCHMITT



Alterações nas características da superfície de compósitos restauradores submetidas a diferentes técnicas de acabamento e polimento

*Tese apresentada à Faculdade de
Odontologia de Piracicaba da
Universidade Estadual de
Campinas, para a obtenção do
título de Doutor em Materiais
Dentários.*

PIRACICABA

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Profa. Dra. REGINA MARIA PUPPIN RONTANI

Profa. Dra. FABIANA SCARPARO NAUFEL

Prof. Dr. JULIO KATIUDE UEDA

Prof. Dr. SIMONIDES CONSANI

Prof. Dr. LOURENÇO CORRÊA SOBRINHO

Dedicatória

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Só é digno do pódio quem usa as derrotas para alcançá-lo.

Só é digno da sabedoria quem usa as lágrimas para irrigá-la.

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Seja um sonhador, mas una seus sonhos com disciplina,

Pois sonhos sem disciplina produzem pessoas frustradas.

Seja um debatedor de idéias. Lute pelo que você ama".

(Augusto Cury)

RESUMO

Inúmeras alterações foram feitas na fabricação das resinas compostas fotoativáveis, especialmente na tentativa de obter melhor estabilidade de cor, adequada resistência ao desgaste e lisura de superfície clinicamente aceitável. Neste contexto, fabricantes têm, promovido alterações no tamanho das partículas de carga e incorporado a maior quantidade de componentes inorgânicos no interior da matriz resinosa. Além da composição do material, os procedimentos de acabamento e polimento podem também influenciar na qualidade superficial da resina composta, relacionando-se diretamente com rugosidade. Superfícies rugosas predispõem a restauração a maior retenção de biofilme bacteriano, facilitando o desenvolvimento de lesões cáries, manchamentos além de interferir no seu brilho e estética finais. Neste contexto, esta tese propôs a avaliar qualitativamente a topografia de superfície de resinas compostas microhíbridas e nanoparticuladas, em função de diferentes combinações entre técnicas de polimento e resinas compostas. Com objetivo didático, esta tese foi dividida em 3 partes, como descrito a seguir. Parte 1: avaliar a influência que diferentes técnicas de acabamento e polimento exercem sobre a rugosidade da superfície de uma resina composta nanoparticulada. Parte 2: comparar a estabilidade de cor e a característica de superfície promovida por técnicas de acabamento e polimento que empregam um ou múltiplos passos sobre resinas microhíbridas e nanoparticuladas, correlacionando com a geometria e composição das partículas inorgânicas das resinas compostas. Parte 3: aferir de forma quantitativa e qualitativa, por meio da rugosidade superficial e microscopia eletrônica de varredura, respectivamente, o efeito de técnicas de acabamento e polimento de resinas compostas nanoparticuladas, nanohíbridas e microhíbridas. Há uma relação direta entre os sistemas de acabamento e polimento e a qualidade superficial das resinas. Sistemas de múltiplos passos tendem a ser melhores em comparação aos de passo único, qualquer que seja o tipo de resina composta empregada. Já a forma e o tamanho das partículas de carga exercem influência sobre a lisura e manchamento, sendo que resinas composta nanoparticuladas apresentam melhor lisura e estabilidade de cor em relação às micro-híbridas. A lisura e o manchamento são materiais e polimento dependentes.

ABSTRACT

Several changes were made in the light-cured composites resins manufacture, especially trying to obtain better color stability, adequate wear resistance and clinically acceptable surface smoothness. About this, manufacturers have predominantly reduced the diameter of charged particles, promoting changes in its geometry and also incorporating a bigger amount of inorganic components within the resin matrix. Besides the material composition, the procedures of finishing and polishing may influence in the composite resin surface quality, relating directly with roughness. Rough surfaces predispose to the restoration a greater retention of bacterial biofilms, facilitating the development of secondary caries, discoloration and staining as well as interfering in their brilliance and aesthetic. In this context, this thesis intends to qualitatively evaluate the surface topography of microhybrid and nanoparticle composite resins, in different combinations for polishing techniques of resin composites. With instructional purposes, this thesis was divided into 3 parts, as described below. Part 1: assessing the influence that different finishing and polishing techniques have on the surface roughness of a nanoparticle composite resin. Part 2: compare the color stability and the surface feature promoted by finishing and polishing techniques of single or multiple steps in nanoparticulate and microhybrid resins, correlating with composite resins geometry and inorganic particles composition. Part 3: assess in a quantitatively and qualitatively way, through surface roughness and electron microscopy scanning, respectively, the effect of different finishing and polishing techniques of hybrid and microhybrid composite resins. There is a direct relationship between finishing and polishing systems and resins surface quality. Systems of multiple steps tend to be better when compared to single step systems, regardless to the type of the resin used. The shape and diameter of charged particles influence on the smoothness and staining, nanoparticulate resins present better surface smoothness and color stability in relation to micro-hybrid resins. Smoothness and staining are materials and polishing dependents.

SUMÁRIO

Introdução	01
Capítulo 1	05
Capítulo 2	19
Capítulo 3	34
Conclusão	53
Referencias	54
Apêndices	56
Anexos	78

INTRODUÇÃO GERAL

A crescente demanda estética no consultório odontológico tem impulsionado e estimulado o desenvolvimento de pesquisas que visam a melhorar a qualidade dos materiais estéticos. O advento dos compósitos ativados por luz visível tem contribuído significativamente para suprir o anseio tanto de profissionais como de pacientes, pois sua utilização permite reproduzir de maneira cada vez melhor a forma, a cor e a textura natural dos dentes.

No desenvolvimento destes novos materiais restauradores, ao longo dos anos, inúmeras alterações foram feitas, especialmente na tentativa de obter melhor estabilidade de cor, adequada resistência ao desgaste e lisura de superfície clinicamente aceitável.^{23 24} Neste contexto, fabricantes têm, predominantemente, incorporados maior quantidade de componentes inorgânicos no interior da matriz resinosa, reduzindo o tamanho das partículas de carga promovendo alterações em sua geometria.¹⁴

Atualmente, os materiais mais utilizados nos procedimentos restauradores diretos são resinas compostas microhíbridas com tamanho de partícula de 0,1 a 5 μm e tamanho de médio 0,7 μm . Uma das mais recentes inovações em tecnologia de resinas compostas é a aplicação da nanotecnologia, que acrescenta muitas vantagens ao material, como menor contração de polimerização, melhores propriedades mecânicas, comportamento óptico favorecido, melhor brilho, lisura de superfície e estabilidade de cor, bem como desgaste diminuído.^{14,17}

Segundo Hörsted-Binslev e Mjör, em 1988⁸, a descoloração dos compósitos pode ocorrer por três mecanismos: 1. descolorações externas devido ao acúmulo de biofilme bacteriano; 2. alterações na superfície ou sub-superfície da resina devido a suave penetração e reação de agentes corantes com a camada superficial da resina composta, pelo fenômeno de adsorção e 3. descolorações intrínsecas devido a reações físico-químicas nas porções profundas da restauração, pelo fenômeno de absorção.

A estrutura da matriz orgânica bem como as características das partículas de carga exercem um impacto direto na lisura de superfície da resina composta e na susceptibilidade de manchamento. Matrizes resinosas hidrófilas tendem a absorver mais água e são mais facilmente manchadas em comparação a matrizes mais hidrófobas, uma vez que a água constitui-se em veículo de penetrabilidade de corantes.^{6; 13} Partículas maiores tendem a produzir superfícies mais rugosas e mais difíceis de serem polidas, comprometendo a lisura superficial.¹¹

Além do efeito da composição do material, os procedimentos de acabamento e polimento influenciam na qualidade superficial da resina composta, relacionando-se diretamente com descoloração e rugosidade.^{20; 9; 16} Acabamentos refere-se ao desgaste ou redução de uma restauração, para se obter características anatômicas ideais. O polimento, por sua vez, relaciona-se à diminuição de irregularidades e micro-trincas da superfície criadas pelos instrumentos de acabamento.²

Adequado acabamento e polimento das resinas compostas são procedimentos clínicos que potencializam o resultado estético, bem como a longevidade da restauração.^{8, 23} Superfícies rugosas predisõem a restauração a maior retenção de biofilme bacteriano, facilitando o desenvolvimento de lesões cariosas secundárias,¹⁹ descolorações e manchamentos²³ além de interferir no seu brilho e estética finais.^{8; 23} A rugosidade aumentada da superfície favorece também a deposição de agentes químicos provenientes de bebidas ou alimentos, que uma vez retidos no biofilme bacteriano previamente formado, difundem-se pela resina podendo afetar o polímero final formado, induzindo à degradação.⁵ Além disso, rugosidades de superfície de 0,3 micrômetros podem ser detectadas pelos lábios ou pela língua, enquanto superfícies mais lisas favorecem conforto ao paciente.¹⁰

Os procedimentos de acabamento e polimento requerem o uso sequencial de instrumentos, geralmente com diminuição gradual das partículas abrasivas, com o objetivo de promover uma superfície lisa e brilhante.¹⁰ Os conjuntos de discos altamente flexíveis à base de poliuretano impregnados com óxido de alumínio são os materiais mais utilizados para esta finalidade.²⁴

No entanto, recentemente, foram introduzidos no mercado odontológico, borrachas abrasivas sintéticas de silicone, com o objetivo de proporcionar à superfície da resina composta lisura e brilho adequado, porém reduzindo-se os passos e o tempo clínico necessários ao acabamento e polimento.¹² Os fabricantes denominam estes sistemas como polimento de “um passo”, em função de se utilizar apenas um instrumento. Este conceito de polimento vem ao encontro das necessidades dos profissionais, no que tange à obtenção de uma superfície lisa e clinicamente aceitável, utilizando-se de apenas um instrumento e em um menor tempo.^{3, 4, 10; 12, 24}

Entretanto, a literatura científica não é unânime em relação à efetividade dos diferentes sistemas para acabamento e polimento de resinas compostas. Enquanto alguns estudos demonstram que o passo determinante, para se alcançar adequada lisura superficial nos procedimentos de acabamento e polimento, consiste na utilização de brocas multilaminadas previamente ao uso de discos ou borrachas abrasivas,¹⁸ outros reportam que os sistemas de polimento de “um passo” são efetivos,^{12; 3,4} enquanto outros preconizam ainda, emprego dos sistemas de polimento de múltiplos passos.²²

Considerando que um dos fatores que contribui para o sucesso ou o fracasso das restaurações estéticas depende, além da estabilidade da cor do material, principalmente da compatibilidade de cor entre o material restaurador e o dente, pesquisas têm sido realizadas com o intuito de tornar esta compatibilidade mais precisa, por meio do desenvolvimento de aparelhos de mensuração objetiva da cor.^{15, 25, 1}

A avaliação quantitativa de alterações mínimas de cor deve ser realizada com dispositivos padronizados, uma vez que o exame visual não tem a acuidade suficiente e é de baixa reprodutibilidade. Para isso, a partir da década de 90, muitos instrumentos baseados em computadores têm entrado no mercado.⁴ Especificamente em 2002, o aparelho VITA Easyshade (Vident, Brea, CA, USA)²¹ foi lançado no comércio, sendo sua tecnologia baseada na espectrofotometria. Um espectrofotômetro mede secções precisas do espectro de luz visível, baseando-se na reflexão pelo corpo de comprimentos de onda específicos. Este método de mensuração do comprimento de onda refletido pelo corpo, traduzindo-se

em valores expressos em unidades de ΔE , é considerado pela literatura especializada como mais preciso quando comparado às medidas obtidas com colorímetros, uma vez que não é influenciado pela luminosidade do ambiente. Os valores de ΔE podem ser utilizados para representar as alterações de cor do material restaurador após um determinado tratamento ou períodos de tempo.²¹

Em função da variedade de resinas compostas e de sistemas de polimento disponíveis no mercado odontológico, torna-se importante avaliá-los, especialmente na verificação de qual sistema de polimento promove o melhor efeito na lisura de superfície de uma determinada resina composta. Além disso, torna-se importante estabelecer qual a relação existente entre as características da rugosidade superficial dos compósitos restauradores resinosos e a manutenção da cor ao longo do tempo.

Dessa maneira, o objetivo geral desta tese é avaliar o efeito que diferentes técnicas de acabamento e polimento exercem sobre a rugosidade superficial e estabilidade de cor de resinas compostas micro-híbridas; nanohíbridas e nanoparticuladas, tendo ainda como objetivos específicos: (1) avaliar as alterações na rugosidade superficial de uma resina específica (Z 350 (3M/ESPE), em função de cinco diferentes técnicas de acabamento e polimento; (2) determinar a influência da geometria e composição das partículas inorgânicas da resina composta na rugosidade de superfície e estabilidade de cor; (3) avaliar *in vitro* as diferenças na estabilidade de cor de resinas compostas micro-híbridas; nanohíbridas e nanoparticuladas, após a imersão em solução corante de café, em função de técnicas de polimento de um e múltiplos passos; (4) analisar qualitativamente a topografia superficial de diferentes resinas compostas, em função dos sistemas de polimento, por meio de microscopia eletrônica de varredura. Com finalidade didática, esta Tese foi dividida em 3 capítulos, correspondentes aos objetivos descritos, de acordo com portaria aprovada pela Resolução CCPG/002/06, que aprovou a apresentação de tese na forma alternativa.

Effect of finishing and polishing techniques on the surface roughness of a nanoparticle composite resin.

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ABSTRACT

Objective: To evaluate the superficial roughness of a resin Filtek Z350 3M ESPE, after different finishing and polishing techniques. **Material and method:** 60 specimens of 7x2 mm were made and distributed in 6 groups (n=10), in accordance with the technique employed: G1- polyester strip; G2- multilaminated burs; G3- diamond burs 3195F and 3195FF; G4- Diamond pro (FGM); G5- Sof-Lex Discs (3M ESPE); G6- Robinson brushes + pumice paste 20 s, and felt disc with of 2-4 μ m diamond paste for 30 s. Artificial saliva was used to storage for a period of 7 days 37°C. After the finishing and polishing techniques, surface roughness (Ra) was measured with Surf-Corder rugosimeter SE 1700. Data were submitted to one-way ANOVA and Tukey test ($p < 0,05$). **Results:** The G3 presented the highest value of roughness (0.61). The G5 resulted lowest roughness values surface (0.15), but were not statistically significantly different from G1, G4 and G6. **Conclusion:** According to the present results, Z350 composite resin presented the lower surface roughness when used polishing systems (Sof-Lex, Diamondpro and Robinson brushes associated with pumice paste and felt discs with diamond paste). Diamond burs (G3) promoted the highest surface roughness mean on the composite surface. It can be evidenced that the polishing systems were the most efficient in providing the surface smoothness.

Keyword: Dental polishing. Composite resin. Nanotechnology.

Introduction

Due to the vast utilization of light activated composite resins since their marketing, they have been constantly improving.³ One of the most significant improvements regarding composite resins is related to the employment of nanotechnology. The new composites, named nanocomposites, have advantages such: lower polymerization shrinkage, improved mechanical properties, favored optical behavior, better brightness, extended maintenance of surface smoothness, better color stability and lower wear.^{12,14} The Filtek Z350 3M ESPE composite, one of those nanoparticle composites, presents zirconia and silica particles,¹ with approximate size between 5-20 nm and pre-polymerized nanoclusters ranging from 0.6 to 1.4 micrometers.²²

The organic matrix structure and the characteristics of fillers exert direct influence on surface roughness of composite resins and on staining susceptibility. Besides the effect of composite composition and the material conversion degree, finishing and polishing procedures might influence the surface quality of composite resins and are related to roughness and staining.^{10,19}

Clinical procedures including finishing and polishing of composite resins improve aesthetic results and restoration longevity. Rough surfaces predispose restorations to increase bacterial biofilm accumulation, facilitating the development of secondary caries,¹⁸ discoloration and staining,²⁰ besides interfering with final brightness and aesthetics.^{9, 20} A major surface roughness also increases the absorption of chemical components from beverages and food, which once retained within the previously formed bacterial biofilm, diffuse into the composite possibly affecting the final formed polymer, inducing degradation.⁷

Finishing and polishing procedures require sequential employment of instruments presenting gradual decrease in particles abrasiveness, aiming to a brighter and smoother

surface.¹¹ Scientific literature is not unanimous regarding the effectiveness of the different systems used for finishing and polishing of composite resins. While some reports state the previous usage of multi blade burs to abrasive discs or rubbers bur is a determinant step to achieve adequate surface smoothness,¹⁷ others report the effectiveness of “one step” polishing systems.^{5,6,13} Moreover, Heintze et al. show a considerable decrease on the mean surface roughness after 20 seconds of polishment to practically every restorative materials, being greater periods of time unnecessary once it will not result in sustainable improvements on surface smoothness.⁸ The aim of this study was to evaluate the surface roughness provided by different finishing and polishing techniques in a nanocomposite. The hypothesis tested of this study is that different finishing and polishing techniques provide different roughness surface on Filtek Z350 3M ESPE composite resin.

Material and methods

Sixty specimens of Filtek Z350 composite resin (3M/ESPE) were used (n=10). The characteristics of Filtek Z350 are described on Table 1.

TABLE 1 – Characteristics of Filtek Z350.

Composite resin	Composition	Type	Shade	Amount of particles	Batch #
Filtek Z350 3M ESPE, St Paul, MN, USA	Matrix: Bis-GMA, Bis – EMA, TEGDMA, UDMA Filler Particle: silica nanofillers (5-75 nm), zirconia / silica nanoclusters (0.6-1.4 µm)	Nanoparticle composite	A3	78.5 wt.% 59 vol.%	8NW

TABLE 2 – Finishing and polishing systems and their respective compositions.

Product	Manufacturer	Composition	Batch #
Carbide multi blade bur	KG Sörensen SP.	Pressed carbide	2976511
Fine diamond bur	KG Sörensen SP.	30 µm diamonds	39520308
Ultrafine diamond bur	KG Sörensen SP.	20 µm diamonds	061027
Diamond Pro Discs	FGM – Joinville- SC.	Al ₂ O ₃ Discs 20 µm (G) 10 µm (M) 5 µm (F) 0.3 µm(XF)	2011
Sof-Lex Discs	3M ESPE, St. Paul, MN, USA.	Al ₂ O ₃ Discs 29 µm (M) 14 µm (F) 5 µm (SF)	081720027
Felt discs Diamond	FGM– Joinville- SC.	Natural or artifical felt	2011
Polishing paste Diamond Excel	FGM– Joinville- SC.	diamond (2 to 4 µm)	141207

The procedures were performed in special room according to the *American Dental Association* specification #27 (ADA) for direct composite resin restorations.

A metallic matrix mould with 2 mm in height and 7 mm in diameter was used to fabricate the specimens. Composite resin was inserted into the mould and covered with clear polyester matrix strips and pressed with glass slides. The composite resin was then light cured following the manufacturer's instructions using a halogen light device (Optilux 501, Kerr Corp., Orange, CA.). The curing tip was positioned perpendicular to specimens'

surface. The power output density employed was 620mW/cm^2 , frequently monitored by means of a radiometer.

The specimens were stored at 37°C and 100% humidity for 7 days prior to finishing and polishing procedures. The specimens were divided into 6 groups ($n= 10$) according to the polishing systems as described on Table 3.

TABLE 3 – Distribution of groups within the study according to polishing systems.

Groups	Material	Technique
Group 1	Polyester strip matrix No finishing or polishing	Direct contact with surface
Group 2	Multi blade carbide bur	Conventional rotation, mean time of 30 s.
Group 3	Fine and Ultrafine diamond bur	Conventional rotation, mean time of 30 s – 15 s each.
Group 4	Sof-Lex system	Intermittent employment for 15 s for each grain in low speed, washed with water spray and dried with air before the next disc in the sequence.
Group 5	Diamond Pro - FGM sequential discs	Intermittent employment for 15 s for each grain in low speed, washed with water spray and dried with air before the next disc in the sequence.
Group 6	Bristle brush with pumice and paste Diamond Excel felt disc.	Bristle brush with pumice for 15 s, washed with water spray and dried with air followed by 15 s,. with and paste Diamond Excel felt disc. Final wash and dry with air syringe.

Only one operator performed the polishment of specimens in order to reduce technique variability. Soon after the polishment of specimens was accomplished, their surface roughness was immediately determined.

The roughness (Ra) in micrometers was carried out using a profilometer device (Kosaka Lab. SE 1700), with a cut off in 0,25 mm and a 0,1mm/s speed. Three consecutive measurements in different areas on the polished surface were conducted and an average was achieved.

Statistical analysis of the results

Data were submitted to one-way ANOVA, with significance level at 5%, followed by Tukey test ($P < 0.05$) for individual comparisons between groups.

Results

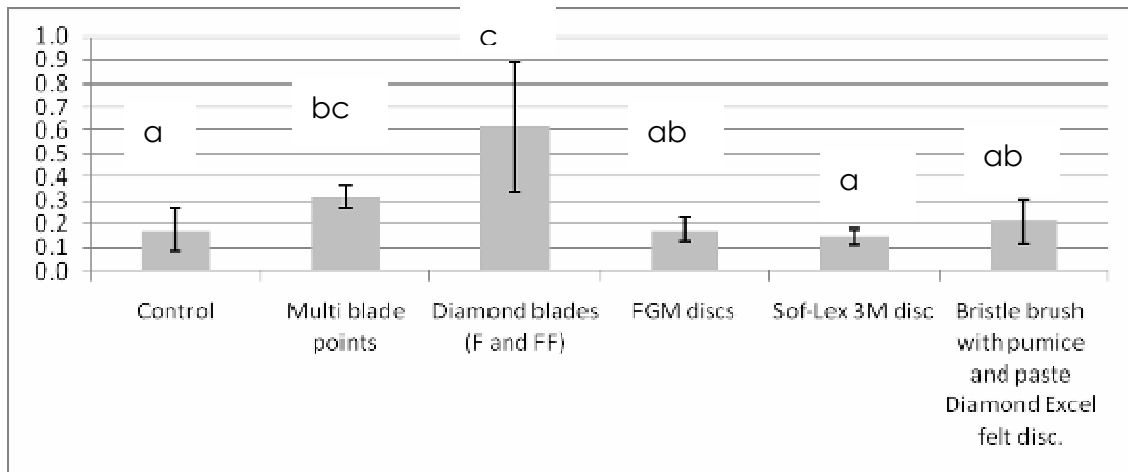
The mean values, standard deviation and statistical comparisons for surface roughness (μm) analyses are shown on Table 4.

Table 4 – Means of surface roughness values (μm) and standard deviation (SD) for each experimental condition.

<i>Finishing /Polishing systems</i>	<i>Control</i>	<i>Multi blade bur</i>	<i>Diamond Point</i>	<i>Diamond Pro - FGM sequential discs</i>	<i>Sof-Lex disc 3M</i>	<i>Bristle brush with pumice and paste Diamond Excel felt disc.</i>
Mean (SD)	0.18 (0.09) a	0.32 (0.05)bc	0.61 (0.28) c	0.18 (0.05) ab	0.15 (0.03)a	0.21 (0.09)ab

Different letters represent statistic significant differences

FIGURE 1 – Roughness means according to experimental groups. Black vertical lines means standard deviation. Different small letters mean statistically significantly difference between means.



A one-way ANOVA test indicated significant effects for finishing/polishing techniques ($p < 0.05$). Tukey's test ascertained that the use of Diamond bur resulted in more rougher surfaces, than FGM discs, bristle brush, Sof-Lex and Control groups ($p < 0.05$). The smoothest surfaces were achieved with Sof-Lex discs and did not differ from the control group ($p < 0.05$). The technique in which Filtek Z350 3M ESPE composite resin was polished with Felt plus Al₂O₃ and Diamond Pro - FGM sequential discs did not differ significantly from the Multi blade bur ($p > 0.05$). Multi blade bur did not differ significantly from the Diamond bur ($p > 0.05$).

Discussion

The surface roughness of composite resin is dependent on the microstructure created by the sequence of physical procedures used to modify this surface. In this study the tested hypothesis was partially accepted. Different finishing/polishing techniques provided different roughness values.

The use of clear polyester strips over the last increment of composite resin restorations is an usual step and it is used in order to avoid the oxygen inhibition layer on the resin surface. However, the obtained surface is rich in organic matrix brought about from the material, leading to a relatively unstable surface. The use of finishing and polishing techniques are essential to favor the chemical stability and to improve the mechanical properties of the resin composite surface.¹⁷ Although these procedures could, depending on the polishing system and material used, increase the surface roughness of the polished surface in different degrees. For the present study, the greater surface smoothness was obtained using sequential Sof-Lex system discs, being similar the surfaces obtained with the use of clear polyester strips, in control group.¹⁹

The geometric structure of the filler particles content of Filtek Z350 3M ESPE might be a possible explanation for these results. Furthermore, the micromorphology of composite resin surfaces after finishing and polishing is strongly influenced by fillers quantity, geometry and size. As the tested material is a nanoparticle composite resin, the fillers are round, smaller and more homogeneous distributed, leading to less wear and if it occurs, it will be more homogeneous.¹⁷ Composite resins presenting smaller fillers provide “protection” to resin matrix and consequently a better clinical performance with less wear and improved polishing.⁷ Several authors¹⁶ state Sof-Lex discs provide smoother surfaces and may be indicated when necessary.

Other discs may also provide good results as reported by Mondelli,¹⁵ where the FGM sequential discs presented adequate polishing, being similar to Sof-Lex Pop discs, similar results were found in this study. The numeric differences in mean surface roughness is not due to type or size of impregnated abrasive particles, once according to the manufacturer’s instructions, the Diamond Pro FGM discs present smaller aluminum oxide abrasive particles in comparison to the Sof-Lex discs.

According to the results, it can be observed that Group 3 (diamond polishing bur) presented the highest roughness, being statistically significant different from the other groups, except G2. In relation to the finishing method, the use of 30 and 20 μm diamonds

promotes the highest surface roughness. The diamond bur present the highest hardness among the finishing and polishing instruments. Observing that characteristic, the diamond bur are highly efficient, wear resistant,¹⁰ but difficult the surface leveling for the final polishing. By this reason, those bur should be only used for polishing in cases where extensive removal of composite resin is required.¹²

The present observation that surface finishing of Filtek Z350 3M ESPE composite using tungsten carbide, presents numerical values smaller than in comparison with the diamond bur is in agreement with previous data from the study of Jung et al. (2007).¹²

The association of polishing pastes after the use of abrasives presented lower mean roughness than the Diamond Pro - FGM sequential discs, but greater than the 3M Sof-Lex polishing system. The smaller mean dimension of particles is found in polishing systems like the Diamond Excel paste FGM (0.2 to 0.4 μm). Theoretically, this Diamond Excel paste FGM should have promoted the best surface polishing in association with felt discs, providing smooth and bright surfaces. However, this fact was not observed in the present study. According to Costa et al. (2007),⁴ this lack of correlation could be explained by the quality of abrasive used in each system, the differences in composition and the physical properties, such hardness. Hardness is expected to influence the surface polishing more than the dimension of abrasive particles.¹⁰

Bollen *et al.* (1997),² stated that surface roughness greater than 0.2 (Ra) may lead to bacterial colonization over the restoration and risk of secondary caries. Ra values lower than 0.2 μm were obtained in the present study for the control, Sof-Lex and FGM sequential discs groups and Bristle brush with pumice and paste Diamond Excel felt disc, when performing polishing on Filtek Z350 3M ESPE composite resin.

Conclusions

According to the present results, Z350 composite resin presented the greater surface smoothness with control and when the Sof-lex and FGM flexible discs, and Bristle brush with pumice and paste Diamond Excel felt disc were used. Multi blade points and 30 and 20 μm diamond burs polishing didn't promote and adequate smoothness

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Effect of the polishing procedures on color stability and surface roughness of microhybrid and nanofilled composite resins.

Abstract

Objectives: to evaluate the effect of polishing procedures (single and multiple steps) on color stability and surface roughness of a microhybrid and a nanofilled composite resins. **Methods:** specimens were fabricated (7 mm x 2 mm) in a metallic mold covered matrix with a Mylar strip and distributed into 6 groups (n=10) according to composite resin (Filtek Supreme XT and Amelogen plus) and polishing procedure (no polishing, PoGo, Sof-Lex: G1. Filtek Supreme XT + PoGo; G2. Filtek Supreme XT + Sof-Lex; G3. Filtek Supreme XT + no polishing; G4 Amelogen + PoGo; G5 Amelogen + Sof-Lex.; G6 Amelogen. + no polishing. The initial color values (baseline) were evaluated using the CIELab scale, using the L*, a* e b* coordinates and ΔE^* using the equation $E\Delta ab = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$, with a Vita Easyshade device. The specimens were submitted to different polishing procedures according to the manufactures' instructions. The surface roughness was evaluated using Surf-Corder (Kosaka Lab. SE 1700). Next, all the specimens were stored in coffee solution at 37°C for 7 days. Following, the final color measurement and roughness were determined. Data were submitted to two-way ANOVA and Tukey's tests with significance level at 5% (p<0.05). **Results:** Multiple steps polishing procedure using Sof-Lex resulted in lower staining. Amelogen showed the highest roughness values than Filtek Supreme on baseline and final evaluations regardless the polishing technique. Filtek Supreme polished with PoGo showed the lowest roughness values. All groups presented discoloration after storage in coffee solution, regardless the polishing technique. **Conclusion:** Multiple steps polishing technique provided lower degree of discoloration for both composite resins. Amelogen showed greater roughness, regardless storage in coffee solution. The final surface texture is material and technique dependent. The best results for roughness and staining resistance were obtained by the association of Filtek Supreme and Sof-Lex polishing procedures.

Keyword: Dental polishing. Composite resin. Nanotechnology.

Introduction

Tooth-colored restorations using resin composites have been widely used in comparison to metallic restorations even for posterior teeth, and they have been relatively successful. Patients and clinicians have defined resin composites as the choice material for aesthetic restorations because their adequate strength, excellent initial aesthetics, moderate cost compared to ceramics and adhesion to tooth structure. However, due to intrinsic properties of this type of material, they are prone to staining and wear.²⁴

Surface roughness is the major contributor for extrinsic discoloration of composite resin restorations. This property is closely related to the organic matrix, inorganic filler composition of the composite and finishing and polishing procedures. Rough surface greater than 0.2 μm provides higher chances of biofilm accumulation, leading to staining and/or discoloration of the restoration's body or margins.¹

Composite resin discoloration can occur by three ways: I) extrinsic discoloration due to biofilm accumulation on the restoration surface; II) surface or subsurface changes with slight penetration and reaction of dye agents on the superficial layer of composite resin; III) intrinsic discoloration due to physic-chemical reactions inside the body of the restoration.¹⁰

Moreover, the matrix structure as well the features of inorganic fillers have a direct effect on surface smoothness of composite resin restorations and on the staining ability. Hydrophilic matrices are more susceptible to water absorption, dye penetration and staining than hydrophobic ones. Similarly, the filler type and size (glass, pyrogen silicon and others) are also closely related with staining.²

In order to measure objectively the color alterations on composite resin restorations, some methods have been experienced, among them the espectrophotometry, which make it possible the study of several parameters related with color stability of composite resins. Vita Easyshade spectrophotometer (Vident, Brea, CA, USA) can measure special sections of visible light spectrum based on the body light reflexion of wavelength specifics. This method about reflected wavelength

by a body are changed in values expressed in ΔE^* units. The ΔE^* values can be used in order to represent the color alterations provided by the composite resin after treatment or period of time.²²

In order to provide color stability, wear resistance and surface smoothness, inorganic fillers have been changed in size and shape.^{24, 25} During mastication, wear leads to dislodgment of filler particles. Due to dislodged particles, holes are present on the surface of restoration exposing the organic matrix to oral environment. In addition, these dislodged particles might cause more abrasion to the restoration. Also, the larger and harder the filler, the more wear and degradation might be observed.⁴

Nanotechnology has recently been used on composite resin production. The new material represents an evolution on balance of aesthetics and mechanical properties, allowing them to be used in anterior and posterior restorations. Among the advantages of using this material it can be pointed out: lower polymerization shrinkage, improved mechanical properties, favoured optical behaviour, greater brightness, surface smoothness, better color stability and decreased wear.^{15, 18}

However, not only the material type and composition are responsible for maintaining the smoothness, but also the finishing and polishing procedures. These procedures require a sequential using of less abrasive instruments, favouring a smoothness and brightness surface.¹¹ In order to carry out those procedures, some sets of highly flexible discs polyurethane-based and impregnated with aluminum oxide have been used²⁵. However, recently, there have been marketed abrasive silicon rubbers in order to provide a smoothness and brightness surface on composite resins. Indeed, the time and clinical steps were reduced.¹³ The manufacturers call these systems as one step systems, since they use only one device.^{5,6,13} Although, there are no consensus on literature concerning the effectiveness of different finishing and polishing procedures and systems used to finish and polish composite resins. Once some studies have demonstrated that the main procedure to reach adequated smoothness on composite resin surface using the multilayered burs before the using of discs or abrasive rubbers,²⁰ others reported that the one steps polishing systems are effective.^{5,6,13}

In this way, it is important to evaluate the effect of different polishing systems on different composite resin, concerning the roughness and the surface roughness of composite resin and color maintenance on time.

Therefore, the tested hypothesis at the present study are that composite resin with different filler types submitted to different types of polishing procedures produce different results of surface roughness and staining. The objective was to evaluate the effect of the polishing procedures, single and multiple steps systems on (1) the color stability and (2) the surface roughness of a nanofilled and a microhybrid composite resin submitted to storage in coffee solution for 7 days.

Materials and Methods

The manufacturers and the composition of tested composite resins and polishing systems are presented on Table 1.

Table 1 – Composition and manufacturers of the materials used in the study

Material (Shade)	Manufacturer	Composition	Filler average	Filler loading
Filtek Supreme XT (A2)	3M ESPE, St Paul, MN, USA	Matrix: Bis-GMA, UDMA, Bis-EMA, and TEGDMA Filler: (zirconia/silica)	nanofillers of silicon (5-75 nm), zircon/silicon nanoclusters (0.6-1.4 µm) – Nanofiller	78.5% wt 59.5% v
Amelogen plus (A2)	Ultradent Inc., South Jordan, UT, USA	Matrix: Bis GMA and dissolvent Filler: silicon dioxide, silicon, silicate particles	Microhybrid	76% w 61 % v
PoGo	Dentsply Caulk Milford, DE, USA	Cured composite of urethane dimethacrilate, fine diamond powder, silicon dioxide 7µm, Al ₂ O ₃	7µm	
Sof-Lex Discs	3M ESPE, St Paul, MN, USA	Al ₂ O ₃ flexible discs	29µm (M), 14µm (F), 5µm (SF)	

Specimen preparation

Cylindrical specimens (7 mm in diameter and 2 mm in height) were fabricated for each group (n=10), according to composite resin and polishing procedures. The composite resin was inserted in the metallic matrix and covered with clear strip and pushed with a glass plate. The specimen was then light cured following the manufacturer's instructions using a halogen light system (Optilux 501, Kerr Corp., Orange, CA.). The curing tip was positioned perpendicular to specimens' surface. The power output density used was 620 mW/cm^2 , frequently monitored by means of a radiometer. The specimens were stored at 37°C , immersed in water distilled for 24 h before the first testing. Table 2 presents the groups' distribution.

Table 2 – Groups distribution according to composite resin and polishing procedure.

GROUP	COMPOSITE RESIN + TREATMENTS
1	Filtek Supreme XT + PoGo
2	Filtek Supreme XT + Sof-Lex pop on
3	Filtek Supreme XT + mylar strip
4	Amelogen + PoGo
5	Amelogen + Sof-Lex pop on
6	Amelogen + mylar strip

Baseline color evaluation

The color of specimens was measured at baseline with a VITA Easyshade (Vident, Brea, CA, USA) spectrophotometer, using the CIELAB scale and the L^* , a^* e b^* . ΔE^* was determined using

the following equation: $\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$. The measurement was performed three times for each specimen. The device was calibrated after the measurement of each specimen.

The specimens were submitted to different polishing systems and procedures, strictly following the manufacturer's instructions. In order to reduce the technique variability, only one operator performed this step.

After the polishing procedure, each specimen was evaluated according to surface roughness using the Surf-Corder (Kosaka Lab. SE 1700) and Ra as a parameter.

Following the baseline measurements, the specimens were immersed in coffee solution (Nescafe, Nestlé, Switzerland, Batch - 91591210B) for 7 days. The coffee solution was the choice as it is one of the most consumed drinks in Brazil and world-wide. Fifteen grams of powder coffee were added to 500 mL boiled water and filtered after 10 min. The coffee manufacturer states that the average time for consumption of one cup of a drink is 15 min, and among coffee drinkers, the average consumption of coffee is 3.2 cups per day. Therefore, the 7 days storage time simulated 10.080 minutes of consumption of the drink over about seven-month period. The solution was then inserted in the eppendorfs with the specimens⁶ and daily renovated.

Before the color was measured, the specimens were washed in distilled water for 1 min and dried with tissue paper. The final color of all specimens was measured as described for the baseline. The effects of discoloration are expressed in ΔE^* units and calculated from the ΔL^* , Δa^* e Δb^* averages using the following equation: $\Delta E^* = [(\Delta L_0^* - \Delta L_1^*)^2 + (\Delta a_0^* - \Delta a_1^*)^2 + (\Delta b_0^* - \Delta b_1^*)^2]^{1/2}$.

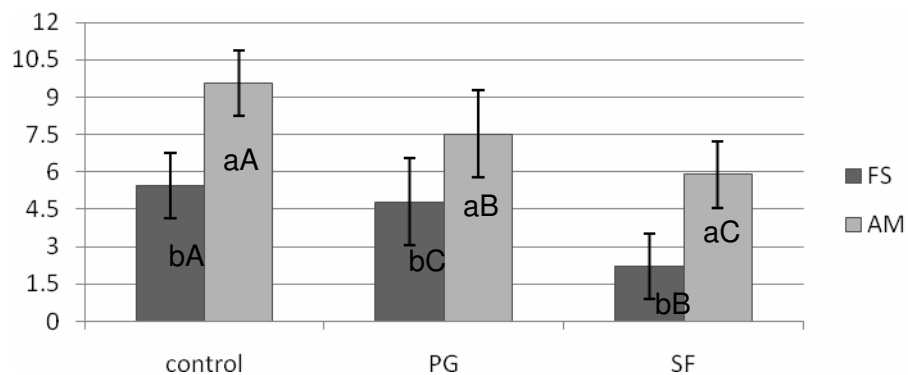
According to Lee et al., 2007,¹⁴ $\Delta E^* < 1$ relates to color alterations not detected by human eye; $\Delta E^* < 3.3$ – clinically acceptable color alterations; and $\Delta E^* > 3.3$ – clinically not acceptable color alterations, resulting in need of restoration replacement due to aesthetics.

The data were submitted to two way ANOVA and Tukey's tests with significance level at 5% .

Results

ANOVA factorial test determined a significant interaction between the studied factors (composite resin and polishing procedures – $P < 0.01$). After 7 days of storage in coffee solution, it was observed greater values of color changes for Amelogen, regardless the polishing procedure (figure 1).

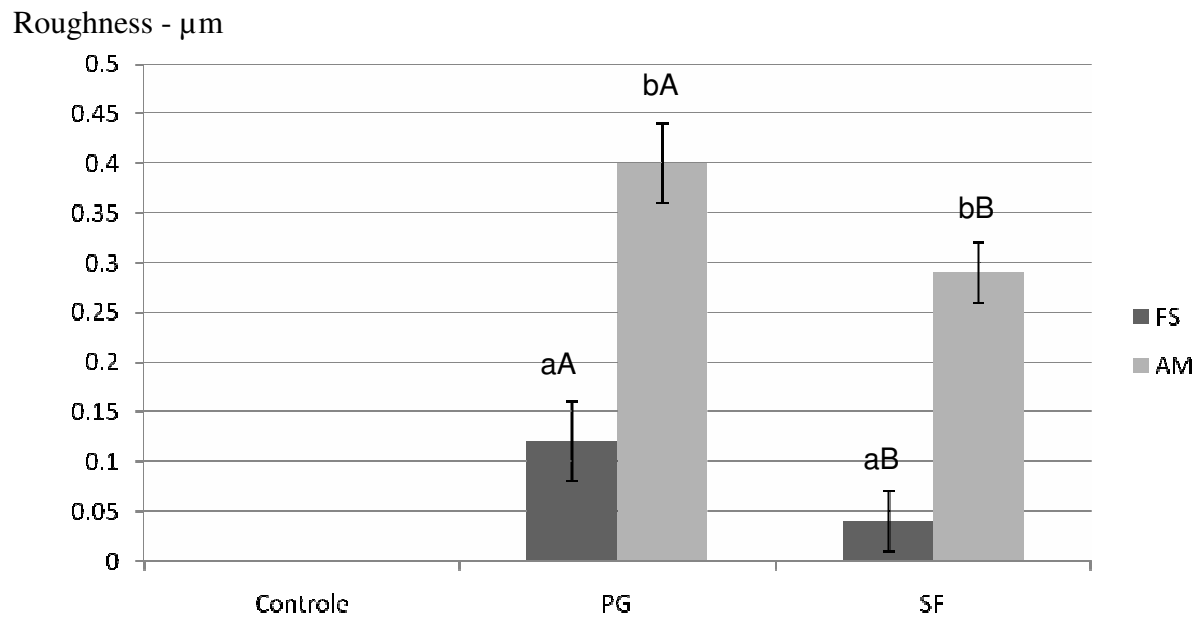
Figure 1 – Average and Standard Desviation values concerning Delta E regarding composite resins (Filtek Supreme (FS) and Amelogen (AM)), polishing procedures and coffee storage time (baseline and 7days-storage)



Different small letters mean statistically significantly difference between averages ($p < 0.05$); Different capital letters mean statistically significantly difference between averages ($p < 0.05$)

The higher color changes were observed when Amelogen was not polished compared to the other treatments and composite resins. Filtek Supreme XT showed low color alteration and the best results were found when it was polished with Sof-Lex. Control groups (3 and 6) showed the highest level of staining, although there was no statistically difference when Filtek Supreme XT was polished with PoGo.

Figure 2 – Average values between baseline and final evaluation of surface roughness concerning polishing procedures and composite resins.



Different small letters mean statistically significantly difference between averages ($p > 0.05$); Different capital letters mean statistically significantly difference between averages ($p > 0.05$)

Immersion in coffee solution provided higher roughness values for Filtek XT polished with PoGo, while for Amelogen, the highest values were observed for the non polished specimens. Concerning polishing procedures, coffee solution provided lower roughness values for both resins Sof-Lex polished samples. (Fig 2)

Discussion

Changes in color of composite resins provided by extrinsic factors are attributed to contamination like coffee, tea, nicotine and beverages. Low periods of immersion, like 7 days, are sufficient to produce staining and color changes to composite resins.^{3,6,19}

Quantitative evaluation of minimal color change by means of visual assessment is not possible or even useful most of times, besides presenting low reproducibility. However, standardized devices can be used for such measurements. Evolution in electronic optics and informatics make the electronic techniques for color selection more adequate for daily usage.^{6,8,19} By this reason, in this study was used the VITA Easyshade system (Vident, Brea, CA, USA). This spectrophotometer measures precise sections of the visible light spectrum, within 400 to 700 nm, based on the reflection of specific body wavelengths, and translating them in values expressed in ΔE^* unities. These systems are more precise, according to the literature, in comparison to measurements obtained from colorimeters, once they are not influenced by the environment luminosity. ΔE^* values can be used to represent color alterations of restorative materials undergoing determined treatment or certain periods of time.^{6,19,22}

The hypotheses tested in this study were accepted. Different polishing procedure systems and different composite resin produced different levels of polishing and different staining after 7 days in coffee solution immersion.

Concerning ΔE^* value, the use of clear (polyester) strips for nanofilled (Filtek supreme) and microhybrid (Amelogen plus) composites, considering the, resulted in greater staining values, since resin matrix emerges to the surface,^{5,6,13} which is highly rich in organic components.^{6,17,19} Moreover, resin matrices tend to absorb more water and are more prone to staining, once water is the vehicle for dyes penetration.^{6,17,19}

It was also observed for both composite resins a decrease in ΔE^* values in relation to the polishing procedures. The highest values were observed when no polishing was performed, followed by one step polishing and finally the multiple step polishing. ΔE^* values observed in the present study revealed that the lower roughness after polishing, the greater the resistance to staining of the

composite resins. In the present study, the microhybrid resin composites were the smoothest surfaces against matrix.^{17,25} These surfaces against matrix were smoother than polished surfaces because the unpolished surfaces are composed of more polymer matrix than fillers.

Different polishing methods of finishing a direct composite resin restoration influence the resistance to color and brightness alterations of the restoration⁹. It is clinically important to determine the procedures to be used in order to obtain a smooth surface, with lower time and number of used instruments¹⁹. This study used single or multiple steps techniques, and it could be observed that the size and geometry of particles exert a direct impact on the surface smoothness and staining resistance.⁶ The combination of nanofillers in nanocluster formulations reduces the interstitial space among fillers, increasing the filler percentage and improving the physical properties. The increase in polishing maintenance in comparison to composites presenting only nanoclusters is also observed, justifying the lowest roughness values for nanofilled composites observed in the present study.

The lowest surface roughness values were observed for the nanofilled composite when the multi steps polishing technique was used. A possible explanation for this observation is the composition and way of usage of the aluminum discs. As they were used in decreasing abrasiveness level, they promote uniform wear and whatever polishing of the surface, regardless the type of composite resin. However, for the nanofilled composites, this effect is increased once the wear occurs due to individual breakdown of the nanofillers, preserving the nanoclusters.^{7,20} The preservation of nanoclusters is possible as function of the strong chemical interaction between nanocluster and resin matrix.¹⁵

Staining is directly related to the resin phase of composites.⁶ Urethane dimethacrylate (UDMA) seems to be more stain resistant than bis-GMA.⁸ However, the resin system of Filtek supreme consist primarily of; bis-GMA, UDMA, and bis-EMA. In these restorative systems, the majority of TEGDMA, a somewhat hydrophilic monomer, has been replaced with a blend of UDMA and bis-EMA.⁴.According to the manufacturers, Filtek supreme composite resins impart a greater hydrophobicity to the composite resin. The low staining susceptibility of Filtek supreme may be related to a low water sorption rate due to the use of hydrophobic resins.⁸

The multiple steps technique demonstrated to be most effective in obtaining a smoother surface, even for the microhybrid composite resin. This fact can be explained by the operationalization of using these materials, as they are usually structured in sequential order of

using with abrasiveness decreasing, favoring the final surface texture. This scenario does not occur with the one step materials.^{19,24}

Studies report that aluminum oxide flexible discs are the best instruments to generate low roughness in resin surfaces. Lu et al., 2003,¹⁴ Turkun & Turkun, 2004¹⁹ e Venturini et al., 2006²¹ demonstrated that aluminum oxide discs are capable of providing smooth surfaces, and this fact is related to their capacity to reduce fillers and matrix evenly. This justifies the multiple steps systems evaluated during the present study were more effective in providing smoother surfaces for both, microhybrid and nanofilled composites.¹² The present results corroborate with those found by Watanabe et al., 2005,²³ who demonstrated that surface finishing with multiple steps systems was superior to one step systems.

The single step system - PoGo, was used in the present study with no surface pre treatment. This system presented higher surface roughness values in comparison to the Sof-Lex discs, regardless the evaluated composite resin. Similar results were obtained by Yap et al., 2004.²⁵ Although the manufacturer recommends the used of the Enhance system prior to PoGo, Jung, et al., 2007¹² while evaluating the surface of microhybrid and nanofilled composite resins, polished with Enhance/POGO association, observed no beneficial results on the composite surface quality with the pre treatment with the Enhance system.

On the other hand, Turkun & Turkun., 2004¹⁹ investigated the surface roughness of microhybrid and nanofilled composite resins when polished with Sof-Lex discs and the POGO system. They used medium, fine and ultra-fine Sof-Lex discs for 30 seconds each for each of the composite resin samples, and the PoGo discs for 30 seconds, using a light rotation movement. They observed that PoGo system promoted a smooth finishing for all the samples in a shorter period of time in comparison to the Sof-Lex discs, and revealed that PoGo system saves time in comparison to multiple steps systems. The multiple steps systems provided the greater staining resistance for both, the nanofilled and microhybrid composite resins.

Conclusion

Based on the results of the present study, it was concluded that the multiple steps polishing technique promoted greater staining resistance, for both the nanofilled and microhybrid composites, and provided the lowest values of surface roughness.

The final surface texture is material and technique dependent. The best results for roughness and staining resistance were obtained from the association of nanofilled composites and multiple steps polishing procedures.

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Single and multiple-step polishing techniques on color and surface roughness stability of microhybrid, nanohybrid and nanofilled composite resins: a quantitative and qualitative analysis.

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ABSTRACT:

Objective: to verify the influence of single and multiple-steps polishing techniques on (1) color stability and (2) surface roughness (Ra) of different composite resins. Methods: 120 specimens (7x2mm) were accomplished according to composite resin and finishing/polishing technique (n=10): G1.Filtek Supreme XT + clear strip; G2. Filtek Supreme XT + PoGo; G3. Filtek Supreme XT + Sof-Lex; G4. Amelogen + clear strip; G5 Amelogen + PoGo; G6.Amelogen + Sof-Lex;G7.Tetric EvoCeram + clear strip; G8.Tetric EvoCeram + PoGo; G9.Tetric EvoCeram + Sof-Lex; G10.Ceram X Duo + clear strip; G11.Ceram X Duo + PoGo; G12.Ceram X Duo + Sof-Lex. The initial color was determined by CIELAB scale with L*, a* and b* coordinates and ΔE^* determined by the equation $\Delta ab=[(L\Delta)^2+(a\Delta)^2+(b\Delta)^2]^{1/2}$, using a Vita Easyshade spectrophotometer device. Next, the roughness of all specimens was determined (Ra).The specimens were submitted to different polishing procedures following manufacturer instructions, followed by a new roughness assessment. All specimens were stored for 7 days in coffee solution at 37°C. After that, final color assessment was performed. Two specimens from each group were evaluated by scanning electron microscopy (SEM).Roughness and color data were submitted to ANOVA and Tukey tests($p<0.05$).Results: All tested materials presented color alterations after coffee solution storage; the highest ΔE values were observed for clear strips and all composite resins, following this sequence: G4 (9h) > G7 (5.8f) = G10 (5.7ef) > G1 (3.7c), followed by the G6 (5.9f) > G9 (3.1bc)=G12 (2.1ab)>G3 (1.7a). G3 after 7 days coffee solution storage presented the lowest values for ΔE (1.704). The lowest roughness values were observed when the clear strips were used regardless material type. Between the polishing techniques employed, the Sof-Lex system provided the lowest surface roughness values for all tested composite resins. Among composite resins the highest roughness values were found for Amelogen (G5–0.49 and G6–0.35), regardless polishing technique. Conclusion: All tested materials presented changes in color after immersion in coffee solution; the highest ΔE values were observed for clear strip and PoGo groups regardless material type. Nanofilled composite resins presented the lowest staining ability. The roughness surface and the staining of composite resins are material and polishing technique dependents.

Keyword: Dental polishing. Composite resin. Nanotechnology.

Introduction

New restorative materials with large application on daily basis dentistry have been launched as a response for the technological improvement of composite resins and for the increased aesthetics request by patients. However, the failure and success of an aesthetic restoration is dependent on chosen material, as well as color stability and material physical-mechanical properties.³⁰

Composite resin's composition and structure and fillers' characteristics present direct impact on surface smoothness and staining susceptibility.^{8, 25} Finishing and polishing procedures may also influence the surface quality of composite resins.

Lately, several modifications on the fabrication of lightcured composite resins were accomplished and the major development has been done to the fillers' system, aiming to decrease the mean size of particles and decrease alterations in geometry, and also to incorporate greater quantities of inorganic components into the resin matrix.^{17, 22} A different mineral and distinct filler shapes have been employed to reduce polymerization shrinkage, to specially achieve better color stability, adequate wear resistance and clinically acceptable surface smoothness, resulting in better aesthetic results.^{17, 22, 30} The type, size and quantity of filler particles influence extrinsic material staining, affecting the surface roughness. The resin matrix and filler particles do not wear at the same rate due to different physical properties. Particle degradation might occur after polishing procedures, leading consequently to irregularities on restoration surface, affecting surface smoothness and favoring surface stain accumulation. These factors are related to initial phases of material staining.

Color stability is an important parameter for modern restorative resin-based materials. Resin matrices have influences on color stability. Hydrophilic resin matrices tend to absorb more water and are easily stained in comparison to hydrophobic matrices, once water constitutes a vehicle for dye penetration.² The type of chosen particle (glass, fumed silica, and others) similarly influences composite resin staining.² The finishing and polishing procedures, besides the effects of material composition and conversion degrees, might influence the surface quality of composite resins, directly in relation to staining and roughness.^{8, 25}

Finishing refers to restoration contouring or reduction in order to reproduce ideal anatomic characteristics. Polishing by its turn, is related to reduction of roughness, microgaps and surface irregularities created by finishing instruments.⁸

Adequate finishing and polishing procedures of composite resins potentiate aesthetics results and restoration longevity. Rough surfaces predispose restorations to increased biofilm retention, leading to development of recurrent caries lesion, discoloration and staining,²⁸ besides interfering with final shine and aesthetics.²⁸

Finishing and polishing procedures require the sequential employment instruments, usually with gradual decrease of abrasive particles, aiming to achieve a shiny and smooth surface.¹⁰ The highly flexible discs impregnated with aluminum oxide are the most employed materials for this purpose.³⁰ Recently however, synthetic silicon abrasive rubber points were launched into market aiming to produce an adequate smooth and shiny resin surface, with reduction of steps and required clinical time for finishing and polishing.¹³ Manufactures name this system as “single-step polishing procedure”, once a single instrument is employed. This polishing concept fulfills dentists’ requirements for obtaining clinically acceptable and smooth surfaces using a single instrument in a shorter period of time.^{5, 6, 10, 30}

However, the literature is not unanimous regarding the effectiveness of different finishing and polishing systems for composite resins. While some studies demonstrate the prior use of multi-blade instruments to discs or abrasive rubber points is a determinant step to achieve surface smoothness,²³ others report the effectiveness of single-step polishing systems.^{5,6,13}

Several methods for color assessment were recently developed, including spectrophotometry and colorimetry, which made possible the study of numerous parameters related to color stability in composite resins.

For this study, a spectrophotometer, which measures precise sections of the visible light spectrum and bases on the specimen body reflection by specific wavelengths, was chosen. This wavelength measurement method reports values of specimen reflection in ΔE^* units. ΔE^* values can be used to represent color changes presented by restorative materials after a certain treatment or period of time.²⁶

According to the variety of composite resins and polishing systems available for dental marketing, it is important to assess them especially to determine which polishing system provides better surface smoothness effect for determined composite resin. Moreover, it is important to determine the relationship between the surface roughness characteristics of composite resins and color maintenance over time.

Facing this issue, the objective of the present study was to evaluate the effect of single-step or multiple-step polishing techniques on (1) color stability and (2) surface roughness of four composite resins.

In the present study, the tested hypotheses are that different dental composite resins according to type, size and compaction of fillers when submitted to different polishing techniques present different patterns of staining and surface roughness.

Material and methods

For this study, a nanofilled composite resin: Filtek Supreme XT (3M ESPE, St Paul, MN, USA), two nanohybrid composite resins: Tetric EvoCeram (Ivoclar Vivadent, Schaan, Liechtenstein) and Ceram X Duo (Dentsply, Konstanz, Germany), and a microhybrid composite resin: Amelogen (Ultradent Inc., South Jordan, UT, USA) were used. Table 1 presents the classification, composition, batch number and manufacturer of the composite resins used in the present study. The employed single-step PoGo (Dentsply) and multiple-step Sof-Lex (3M ESPE Dental Products) polishing systems are listed on Table 2.

TABLE 1: Characteristics of tested composite resins (manufacturer, composition, color, filler size and batch number).

Composite Resin	Composition	Type	Filler quantity	Batch #
Filtek Supreme XT (3M ESPE, St Paul, MN, USA)	Matrix: (Bis-GMA), (TEGDMA, (UDMA), Bis-EMA Filler: silicon nanoparticles (5-75 nm), zirconia/silicon nanoclusters (0.6-1.4 μm)	Nanofilled	78.5 % w 59% v	7EJ
Tetric EvoCeram (Ivoclar Vivadent, Schaan, Liechtenstein)	Matrix: Bis GMA and TEGMA dimethacrylate, additive, initiator, stabilizers, dye Filler: barium glass, ytterbium fluoride, spherical mixed oxides, prepolymers - 0.6 μm	Nanohybrid	82.5% w 68% v	L32850
Ceram X Duo Dentsply, Konstanz, Germany	Matrix: Methacrylate modified polysiloxane, dimethacrylate resin Filler: Barium-aluminum-borosilicate glass (1-1.5 μm), silicon dioxide nanofiller (10 nm)	Nanohybrid	76% w 57% v	0708000 836
Amelogen plus Ultradent Inc., South Jordan, UT, USA)	Matrix: Bis GMA and diluent Filler: silicon dioxide and silicate particles	microhybrid	76% w 61 % v	B3D26

TABLE 2 – Characteristics of used polishing systems (manufacturer, composition and directions)

Product Manufacturer	Composition	Directions
PoGo Dentsply Caulk Milford, DE, USA)	Cured urethane dimethacrylate resin, fine diamond powder, silicon dioxide 7 μm , Al_2O_3	Apply with light intermittent pressure for 40 seconds under moderate speed
Sof-Lex 3M ESPE Dental Products, St Paul, MN, USA	Al_2O_3 Flexible discs 29 μm (M), 14 μm (F), 5 μm (SF)	Apply with light intermittent pressure for 15 seconds under moderate speed

One hundred and twenty specimens were fabricated using a metallic matrix with dimension of 7mm in diameter and 2mm in width. The specimens were divided into 12 groups (N=10) according to the association of composite resin type and polishing system, as described on Table 3. Composite resin was inserted into the matrix and covered by a clear strip and pressed with 2mm thick glass slides. Specimens were lightcured for 40 seconds using a halogen light - Curing Light 2500 (3M/ESPE, Saint Paul - MN, USA), with power density of 500 mW/cm². Its power density was checked with a radiometer before each irradiation. The specimens were stored at 37°C and 100% absolute humidity for 24 hours prior the initial color measurement.

TABLE 3 – Association of composite resins and polishing systems.

GROUPS	CR + TREATMENTS
1	Filtek Supreme + Mylar Strip
2	Filtek Supreme + PoGo
3	Filtek Supreme + Sof-Lex
4	Amelogen + Mylar Strip
5	Amelogen + PoGo
6	Amelogen + Sof-Lex
7	Tetric EvoCeram + Mylar Strip
8	Tetric EvoCeram + PoGo
9	Tetric EvoCeram + Sof-Lex
10	Ceram X Duo + Mylar Strip
11	Ceram X Duo + PoGo
12	Ceram X Duo + Sof-Lex

The initial color of all specimens was determined using a VITA Easyshade spectrophotometer (Vident, Brea, CA, USA), following the CIELAB scale (Commission Internationale de l'Eclairage), with L*, a* and b* coordinates and ΔE^* determined by the equation $\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$. L* refers to shade from 0 to 100 (from black to white), a* refers to colorfulness (-80 to +

80, negative green value and positive red) and b^* (-80 to + 80, negative blue value and positive yellow).

The referred spectrophotometer measures precise sections of visible light at the 400 nm to 700 nm range, based on the reflection of specimen-specific wavelength and translates values reported in ΔE^* , which are not influenced by environment colorfulness. ΔE^* values can be used to represent restorative material color changes underwent after a determined treatment of period of time.^{5, 26} According to Lee et al., 2007,¹⁴ $\Delta E^* < 1$ relates to color alterations not detected by human eye; $\Delta E^* < 3.3$ – clinically acceptable color alterations; and $\Delta E^* > 3.3$ – clinically not acceptable color alterations, resulting in need of restoration replacement due to aesthetics.

The measurement was performed three times per specimen. After each specimen reading, the instrument was calibrated for the next specimen assessment.

Specimens were submitted to the different polishing procedures in low speed following the manufacturer's instructions. One single operator performed the specimens polishing to reduce the technique variability of this phase.

To evaluate polishing effect on specimen surface smoothness, the specimens' surface roughness was assessed at the end of polishing procedures using a surface roughness measuring instrument Surf-Corder (Kosaka Lab. SE 1700). The mean surface roughness (R_a), obtained by three consecutive measurements in different surface locations, was the used parameter.

For the assessment of staining resistance of composite resins, the specimens were immersed in coffee solution (Nescafe, Nestlé, Switzerland, Batch 91591210B) for seven days. Coffee solution was the choice for being an extremely consumed product in Brazil and worldwide. Fifteen grams of coffee were added to 500 mL boiling water and filtered 10min prior to placement in the storage recipients with the specimens (ERGÜCÜ et al, 2008). The solution was changed every 24 hours.

Before color assessment, specimens were washed in distilled water for one minute and dried with absorbent paper. The specimens' final color was assessed following the same protocol described for the initial color measurement.

Data were submitted to 2-way ANOVA and Tukey test for individual comparisons among the different groups, both with level of significance set at 5%, ($P < 0.05$).

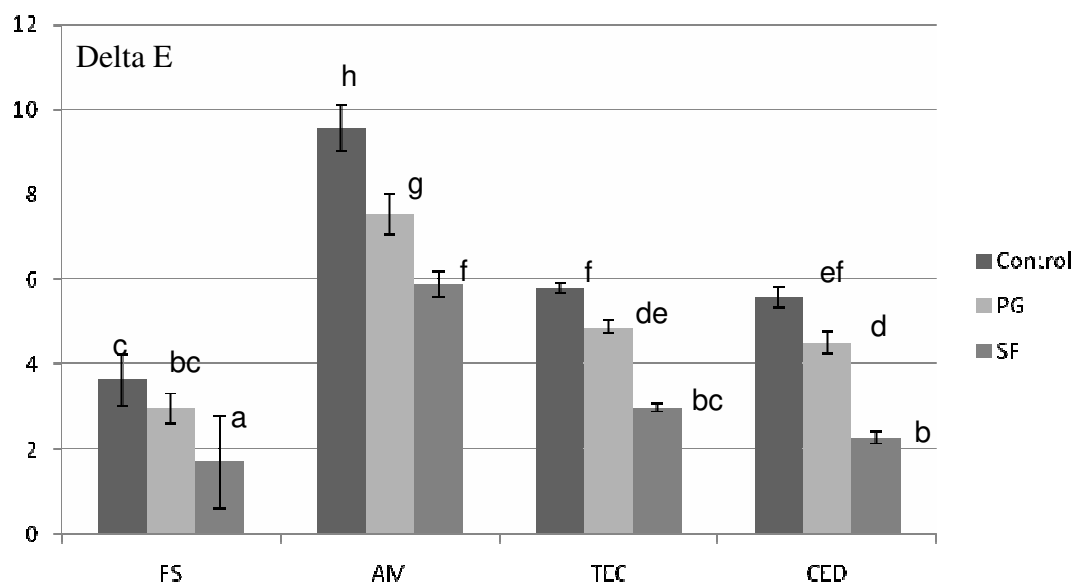
Two specimens from each group were evaluated through scanning electronic microscopy (SEM – JEOL JLM 5600 LV) for the qualitative surface analysis and possible correlations between composite resin and polishing system with surface smoothness. They were gold metalized to assure visualization at 50X magnification and to observe fillers' size and geometry under 1000X magnification.

Results

The statistical analysis (2-way ANOVA) determined the existence of significant interaction among the factors composite resins and polishing techniques ($P < 0.05$), for the studied variables, surface roughness and staining after immersion in coffee solution (Delta E). Multiple comparisons were carried out with Tukey test, which revealed differences among groups.

The mean values and standard deviation for composite resins and polishing systems are presented on figures 1 and 2 and respectively represent the mean values for Delta E and surface roughness (Ra expressed in μm).

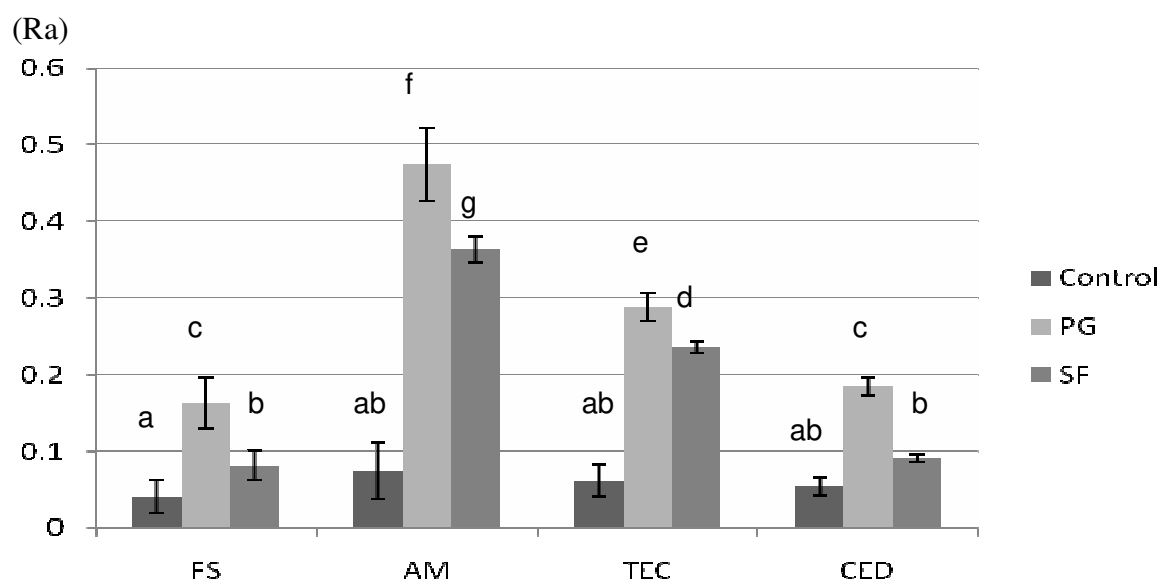
Figure 1– Delta E mean values of the resins Filtek (FS), Amelogen (AM), Tetric Evo Ceram (TEC) and Ceram X Duo (CED). Columns identified by the same small letters do not significantly differ according to Tukey test ($P > 0.05$). The pink band represents Delta E values between 1 and 3.



FS composite resin and CED submitted to the Sof-Lex polishing system and after one week immersed in coffee solution, presented the lowest values for Delta E. All tested materials presented changes in color after immersion in coffee solution; and the highest values for Delta E were observed in the control group for each material type followed by the Pogo and Sof-Lex system.

Figure 2 – Mean surface roughness values (μm) and standard deviation according to employed material type and polishing technique. Columns identified the same small letters do not significantly differ according to Tukey test ($P>0.05$). The critical value for Ra is 0.2.

Final Roughness

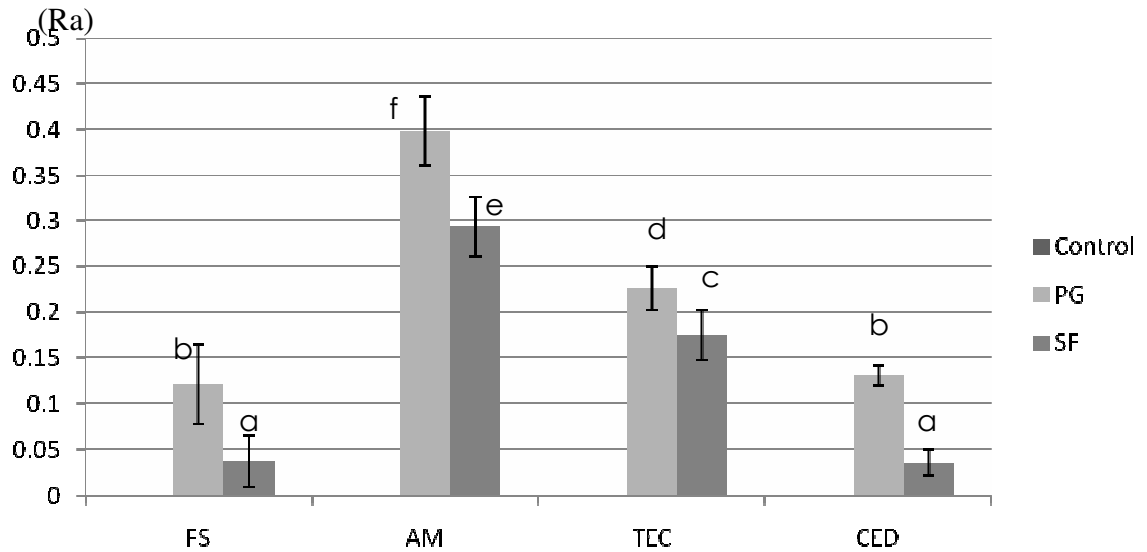


For the roughness values, the lowest values were observed when the clear strip was used regardless material type. Between the polishing techniques employed, the Sof-Lex system presented the lowest surface roughness values for all tested composite resins.

Statistical analysis revealed that regardless polishing technique, Amelogen composite resin presented the highest ΔE^* values and roughness after treatment.

Figure 3 – Surface roughness variation according to employed material type and polishing technique. Columns identified by the same letters do not significantly differ according to Tukey test ($P>0.05$).

Roughness -Variation



Multiple comparisons using Tukey test determined that Filtek Supreme (FS) and Ceram X Duo (CED) specimens presented the lowest roughness variation when polished with the Sof-Lex (SL) system, those being statistically different from Tetric EvoCeram (TEC) and Amelogem (AM). (Figure 3)

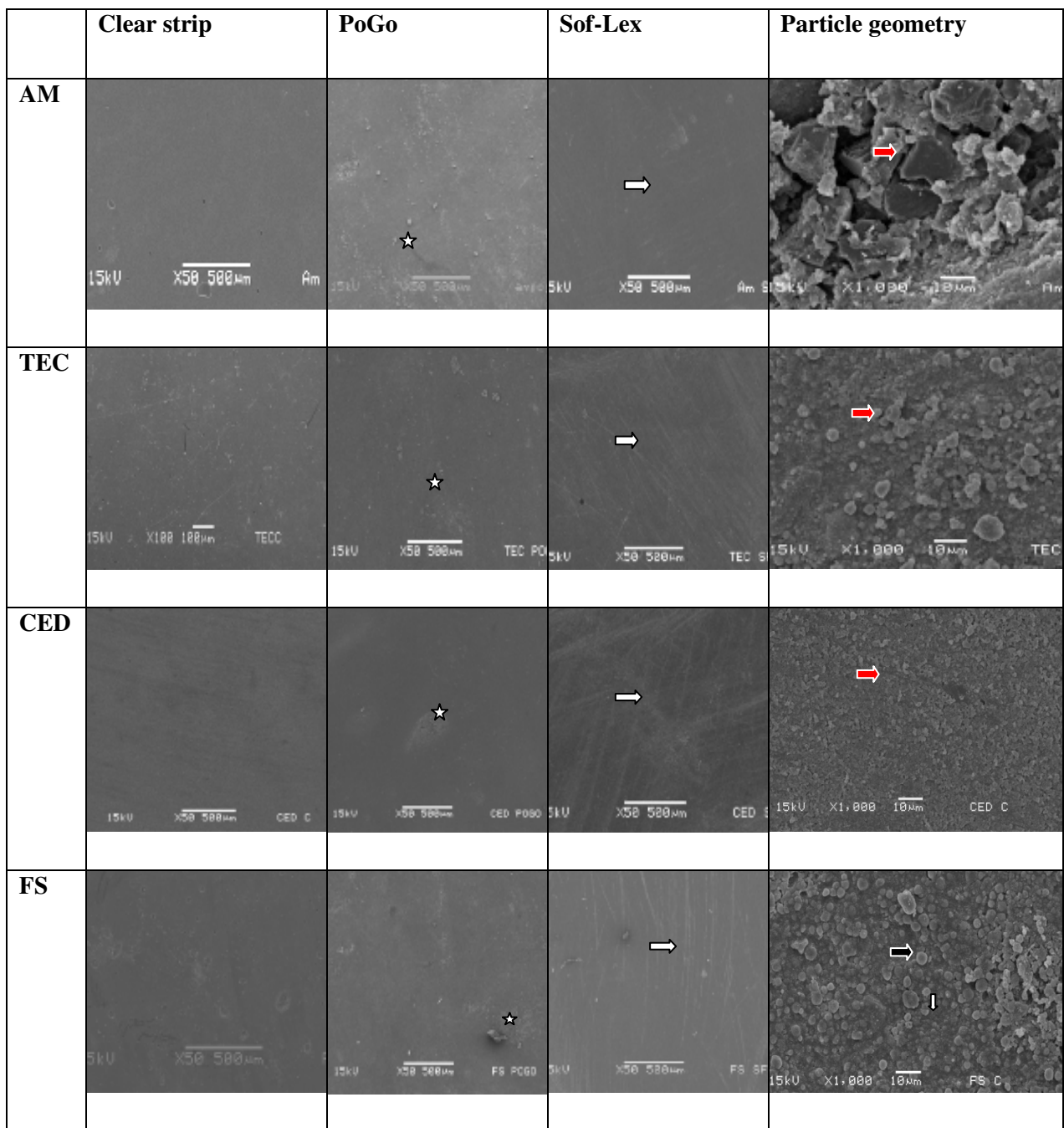


Figure 4– Microphotographs of Filtek supreme (FS); Tetric EvoCeram (TEC); Ceram X Duo (CED) and Amelogen (AM) composite resins are disposed in the rows. Columns are represented by control (clear strip); after polishing with Sof-Lex discs; after polishing with PoGo system (original SEM 50X) and composite resin particles geometry on the last column (original SEM 1000X). Observe the surface smoothness promoted by the clear strip (column 1); the irregular surface promoted by PoGo system (stars); the more uniform surface promoted by Sof-Lex system (column 3) (50X magnification), besides some noticed scratches (with arrows). The last column presents a greater magnification (1000X) of the composite resins after polishing and the removal of the organic matrix and exposure of fillers can be observed. Note the size, shape and distribution of fillers. Amelogen – microfillers with borders predominantly irregular and accentuated. TEC, CED and FS with spherical fillers and compaction characteristics, allowing even filler distribution (red arrows). FS – last column – note the presence

Discussion

Finishing and polishing procedures have been associated to lower bacterial plaque retention, lower marginal discoloration and consequent increased longevity for aesthetic restorations.

The tested hypotheses of this study were proved once significant interactions between different composite resins and polishing techniques were observed.

Lower surface roughness values were observed for the association of Filtek Supreme composite resin and Sof-Lex multiple-step polishing system. This observation could probably be attributed to material composition and influenced by filler type, particles size and quantity, particles distribution within organic matrix (Fig. 4), which interfere with the mechanical properties and polishing degree of composite resins.³⁰ For the nanocomposites, the nanometer-sized particles combination reduces interstitial space among fillers, leading to improvements on surface physical characteristics.^{1, 24}

According to polishing procedures, lower surface roughness values were observed for the nanofilled composite resins with the multiple-step polishing procedure (Fig. 3). A possible explanation for this fact relies on composition and employment directions of the aluminum oxide discs. As they are employed in decreasing grit order, they promote even surface wear and polishing, regardless the composite resin type. This effect is potentiated for the nanofilled composite resins, once wear occurs by individual breakage of nanometer-sized particles, preserving the nanoclusters as reported by Turssi et. al (2005), and resulting in surfaces with defects smaller than the light wavelength¹⁷.

The fabrication of nanofilled composite resins occurs by associating small particles in a more favorable thermodynamic pattern leading to formation of aggregates and clusters.¹⁷ This might explain the low surface roughness observed for the Filtek Supreme specimens when submitted to the Sof-Lex polishing system. This nanocluster preservation is possible due to strong chemical interaction between nanocluster and resin matrix.

According to the present results, smoothest surfaces were obtained when the clear strip was used, regardless the type of tested composite resins, due to the emergence of resin organic matrix.^{28, 30} This matrix emergence, besides promoting better surface texture, might lead to faster degradation of restoration surface due to the characteristics of the formed polymer. This surface is also limited by the anatomy complexity and restorative procedures, being the functional occlusal adjustment necessary in almost every restoration.⁴

Inorganic particles of composite resins are more difficultly abraded than the resin matrix, and they might be lost during finishing and polishing procedures. This will result in a filler positive relief on the surface;²³ thus the larger the particles the larger the relief, reflecting on the roughness (Fig. 4).

A relationship between filler size and surface roughness can be observed, as the microhybrid composite resin Amelogem, composed by inorganic particles with bigger size and uneven borders, presented greater surface smoothness results with the two tested polishing systems, being this observation in agreement with reported results.²¹ When the particles size is reduced, and nanomer-sized are incorporated to matrix, an improvement in surface smoothness is expected, as observed in the present study results for the composite resins Ceram X Duo and Tetric EvoCeram. The relationship between filler size and surface roughness can be also observed when comparing the nanohybrid composites among them. As an example, Ceram X Duo presents smaller sized particles in comparison to Tetric EvoCeram and thus, presented lower surface roughness regardless the employed polishing system.

The results showed that Sof-Lex™ discs promote smoother surfaces than PoGo polishing system. The effects of finishing and polishing systems show surface roughness was polishing system and composite resin dependent. Researches show that Sof-Lex™ aluminum oxide flexible discs are the best instruments for promoting low composite resin surface roughness.¹⁶ Uctasli, (2007)²⁴ demonstrated aluminum oxide discs are able to propitiate smooth surfaces, and this fact is related to the ability of those discs to evenly reduce filler and matrix, favoring their employment for composites presenting smaller sized fillers.^{11, 16, 20, 25, 27}

The abrasive particles of a composite resin finishing system should be harder than the material particles in order to be efficient. On the contrary, the polishing agent would only remove soft matrix and leave highly leveled fillers on the surface. Aluminum oxide is harder than silicon oxide and usually harder than fillers employed for composite resin formulations.²⁰ According to

Jefferies, (2007),⁸ fine aluminum oxide particles are hard enough to polishing ceramic and composite resin.

Contrasting with the present results, Turkun LS, Turkun M. 2004 e Yap. et. al. in 2004 observed that one-step polishing systems are as efficient as multiple-step systems for composite resin polishing.

Color determination in dentistry has been done for many years through color guides to visually identify colors;^{9, 14} however, previous studies observed this visual assessment as being inconsistent and lacking in reliability.³ Instrumental methods using electronic optics and informatics, have been developed to eliminate uncontrolled variables during the color determination process, leading to a more adequate technique of electronic color selection for daily basis use,⁵ once they are able to detect subtle color changes.¹⁸ According to Yap et al, 1999,²⁹ in cases where ΔE^* value is greater than 3, the computerized analysis is better than naked eye.

ΔE^* values in the control groups for all tested composite resins showed more intense staining and the color ΔE^* crescent degree was 3.62 for Filtek supreme; 5.57 for Ceram X Due; 5.78 for Tetric EvoCeram and 9.54 for Amelogem. According to Ergücü et al., 2008⁵, all Mylar-finished specimens showed the most intense staining due to the outermost resin layer. Therefore, removal of this resin layer by finishing and polishing procedures would produce a harder, more stain resistant and, hence, more esthetically stable surface. Moreover, surfaces present higher quantities of organic matrix as a result of pushing the material with clear strips.¹⁹

A decrease in ΔE^* values were observed in relation to polishing procedures for all tested composite resins, indicating the removal of the most superficial non cured composite layer by finishing and polishing procedures results in a surface more staining resistant and consequently a more aesthetically acceptable surface. These results are in agreement with Ergücü et al., 2008⁵.

Differences in chemical structures of composite resins, such as types of employed oligomers or monomers, concentration/type of activators, initiators and inhibitors, oxidation of carbon-carbon double bonds, size/type of fillers and bonding system might influence color stability^{5,7}.

All tested composite resins presented detectable color changes after immersion in coffee solution. The effect of the staining solution was material dependent according to the observed results Ergücü et al., 2008⁵. An association between filler size and color change could be detected

in the present study, as the Filtek Supreme nanocomposite presented the lowest staining values, followed by the nanohybrid and microhybrid composite resins. This observation is in agreement with Kawaguchi et al., 1994¹², who detected that composite resins with large-sized inorganic fillers are more prone to water sorption and color alterations.

Moreover, color might be related to polymer and filler/matrix phase, or even to polymer filler interface. The filler/matrix interface performs an important water uptake role for composite resins^{2, 14}, being color stability directly related to hydrophilic characteristics of composite resin matrix, which will contribute to greater staining. Thus, as higher the composite resin water uptake, the higher the volume of formed polymer and consequently there will be greater spaces for water molecules to diffuse within the polymeric structure^{7, 15}. A mixture of UDMA and bis-EMA provides a higher hydrophobicity to composite resins and consequently higher stability, confirming the present results obtained for Filtek Supreme. On the other hand, Tetric EvoCeram presents Bis-GMA and TEGDMA in its composition, and those monomers are more hydrophilic, leading to more water absorption, fact confirmed by the present ΔE^* values. Furthermore, the present tested nanohybrid composite Tetric EvoCeram presented higher ΔE^* values than Ceram X Duo. This fact may be attributed to the presence of ytterbium trifluoride on Tetric EvoCeram's composition. This component provides a certain fluoride release and according to Buchalla, 2002², this component may be soluble and influence on the optical characteristics of the material.

Conclusion

Based on the present results, it can be concluded that both the surface roughness and staining (ΔE^*) are influenced by the composite resin type and by the polishing technique. Multiple-step polishing technique is more favorable in achieving surface smoothness and staining resistance regardless resin type. Nanofilled composite resin presented greater staining resistance. Surface smoothness and composite staining are material and polishing technique dependent.

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Conclusão Geral da tese

Sistemas de polimento múltiplos passos tendem a ser melhores em comparação aos de passo único, independentemente do tipo de resina empregada

A resina nanoparticulada estudada apresenta melhor lisura após qualquer que seja o sistema de polimento empregado.

Há uma relação direta entre a composição da resina e a resistência ao manchamento

A lisura e o manchamento são materiais e polimento dependentes

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APÊNDICE 1 - Ilustrações dos materiais empregados na confecção dos espécimes da metodologia do capítulo 1, 2 e 3

Confecção dos espécimes

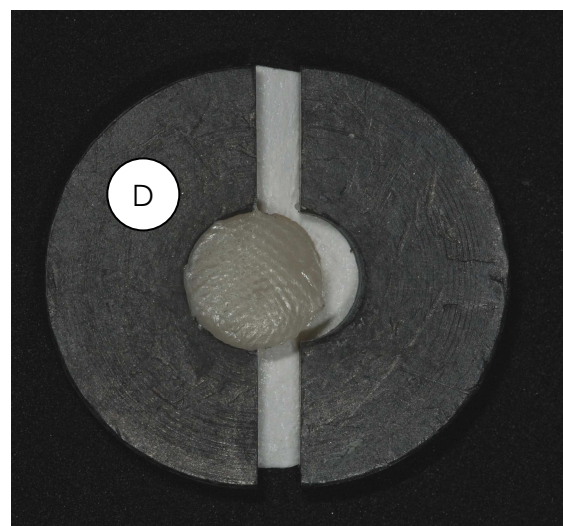
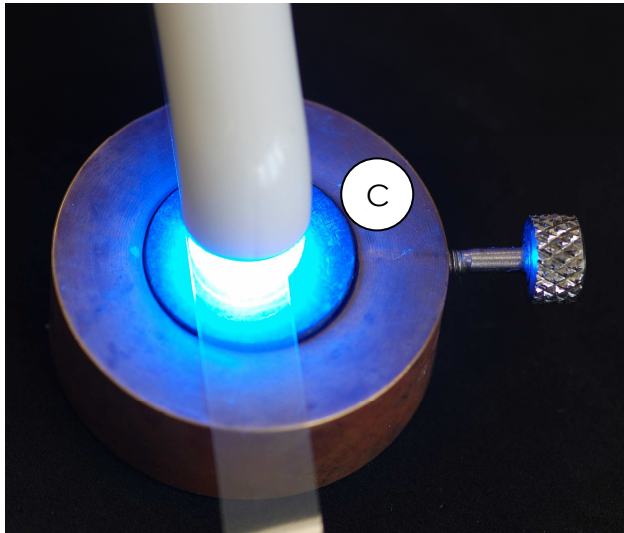
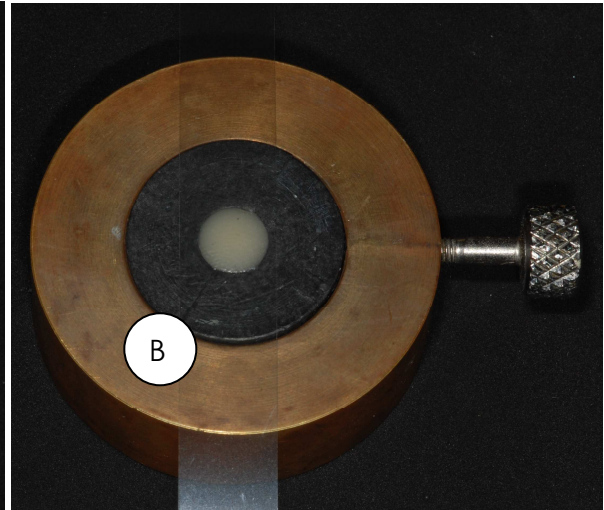
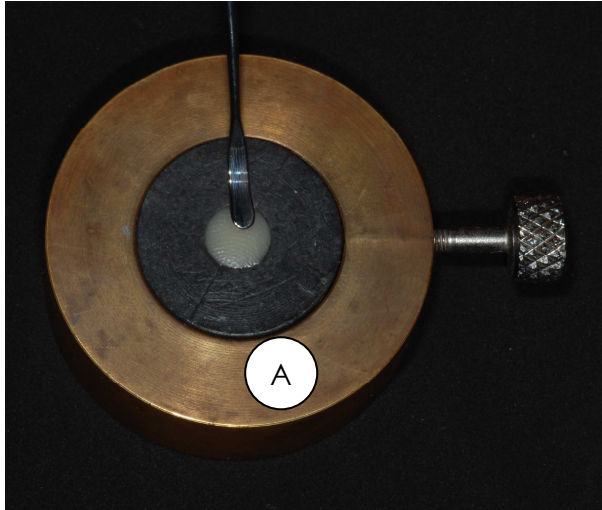
A – Matriz utilizada para confecção dos espécimes.

B - Inserção da resina na matriz e coberta com tira de poliéster.

C –Fotoativação com o aparelho de luz halógena Optilux 501, Kerr Corp.,Orange, CA

D - Remoção do espécime da matriz.

E - Espécimes de resina composta



APÊNDICE 2 –Ilustrações dos materiais empregados na metodologia do capítulo 1

- A - Ponta diamantada fina e ultrafina KG Sörensen
- B - Fresa multilaminada carbide KG Sörensen
- C - Disco de feltro – DIAMOND - FGM
- D - Discos seqüências, - Discos Diamond Pro -FGM.
- E - Pasta diamantada. - Diamond Excel
- F - Discos seqüências da soflex 3M - ESPE
- H - Escova Robson com pedra pomes-SS White
- I – Resina Composta Filtek Z350 (3M ESPE, St Paul, MN, USA
- J – Distribuição dos grupos experimentais

GRUPOS		RC + Sistema de polimento
1	Z350 + tira de poliéster	
2	Z350 + Ponta multilaminadas carbide	
3	Z350 + Ponta diamantada fina e ultrafina	
4	Z350 + Discos seqüências da FGM,	
5	Z350 + Discos seqüências da soflex 3M	
6	Z350 + Escova Robson com pedra pomes e disco de feltro com pasta diamantada.	



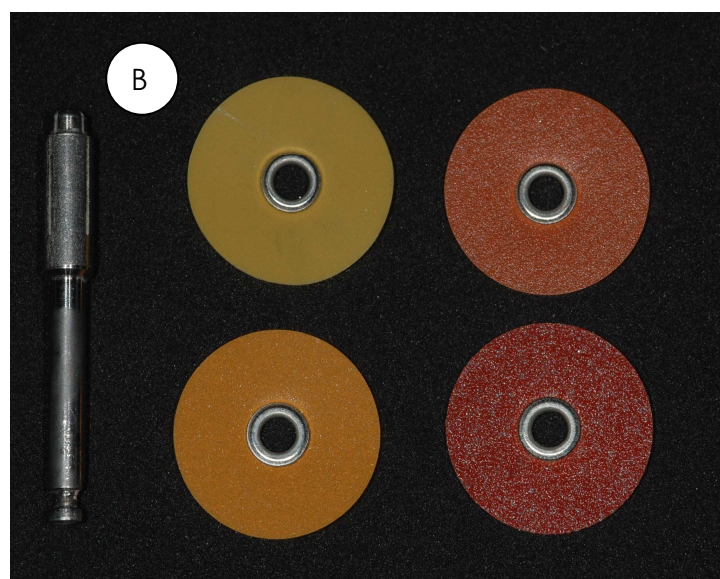
APENDICE 3 – Ilustrações dos materiais empregados e na metodologia do capítulo 2 e 3

Material utilizado

A – Resina compostas utilizadas no estudo, Filtek Supreme XT, 3M ESPE; Tetric Evo Ceram, Ivoclar Vivadent; Ceram X Duo, Dentsply; Amelogen, Ultradent

B - Os sistemas de acabamento e polimento utilizados: sistema de “múltiplos passos” Discos Sof-Lex 3M ESPE

C - Os sistemas de acabamento e polimento utilizados: sistema de “um passo” PoGo, Dentsply



APENDICE 4 – Ilustrações da metodologia empregada no capítulo 2 e 3

Polimento dos espécimes

A - Polimento da amostra da resina sistema de acabamento e polimento multipassos Sof-lex 3M/ESPE

– Granulação maior (vermelho claro)

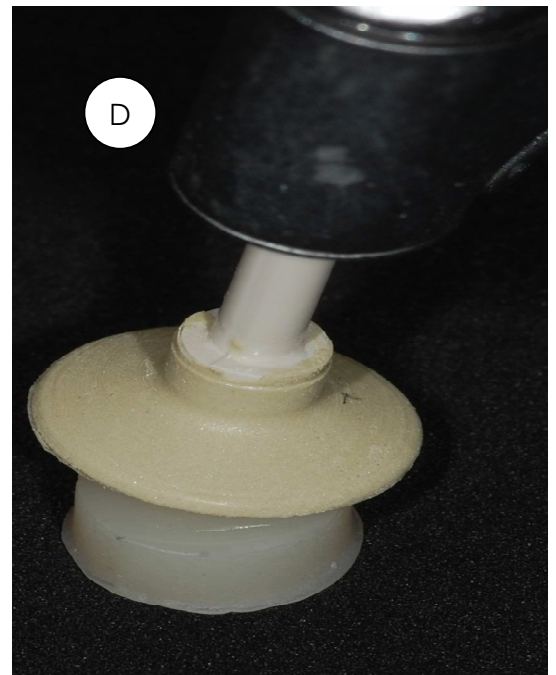
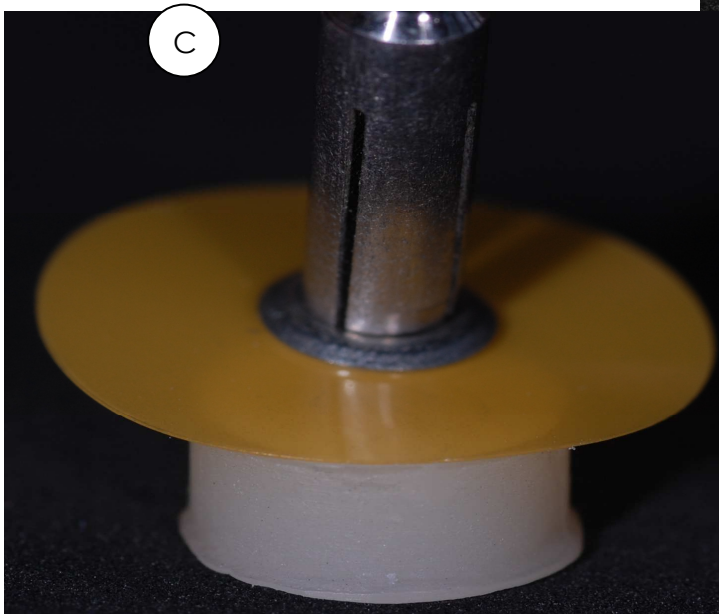
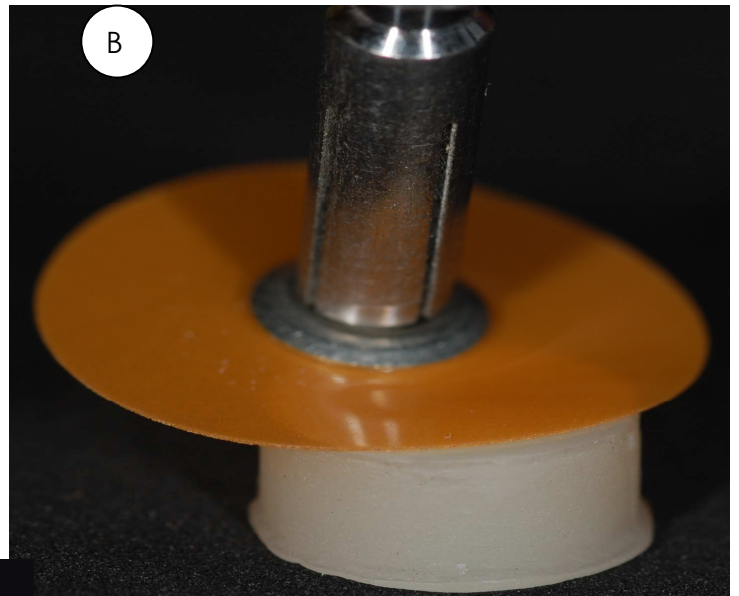
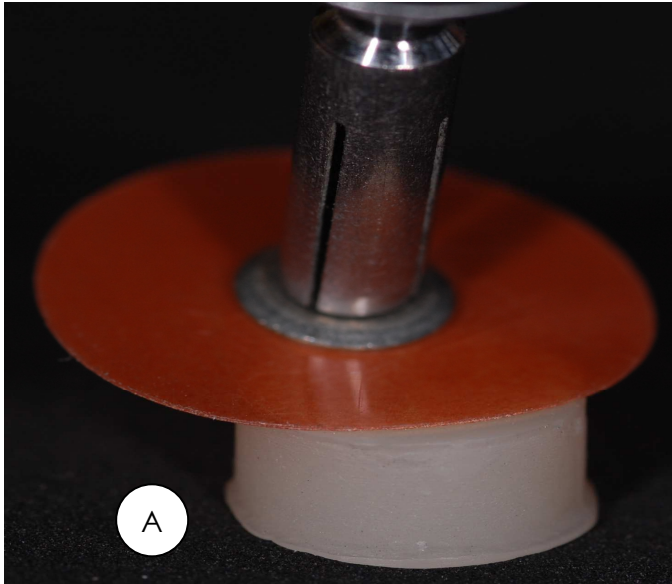
B - Polimento da amostra da resina sistema de acabamento e polimento multipassos Sof-lex 3M/ESPE

– Granulação fina (Laranja)

C - Polimento da amostra da resina sistema de acabamento e polimento multipassos Sof-lex 3M/ESPE

– Granulação super fina (amarelo)

D - Polimento da amostra da resina com sistema PoGo (Dentsply Caulk Milford, USA)



APENDICE 5 – Ilustrações da metodologia empregada no capítulo 2 e 3

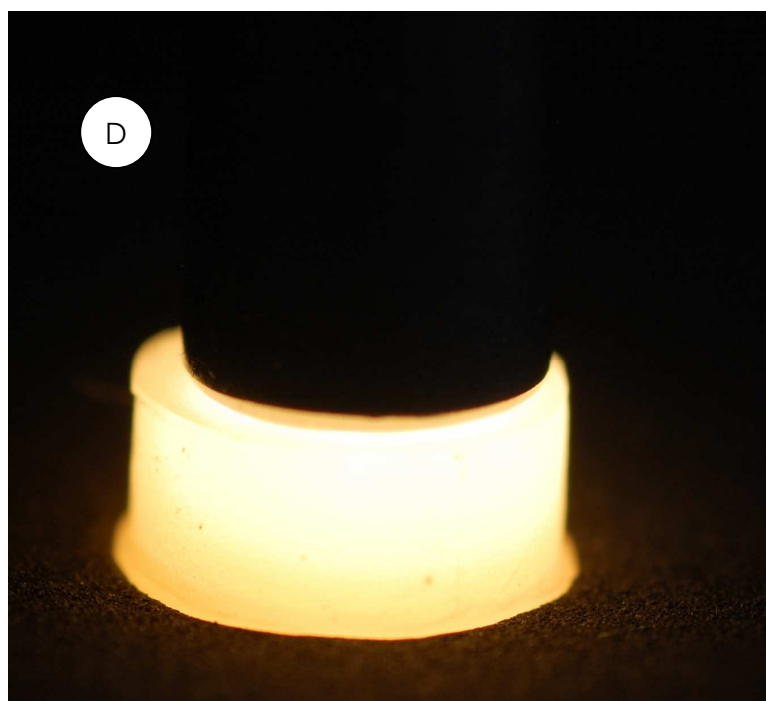
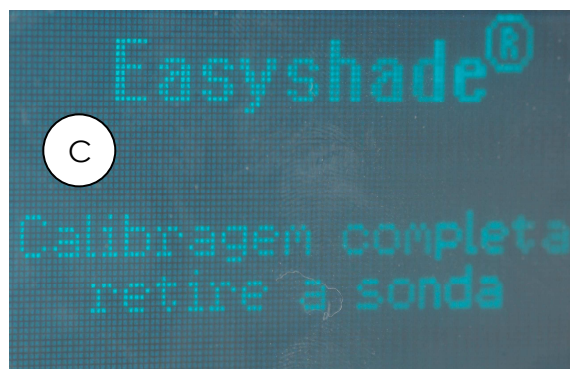
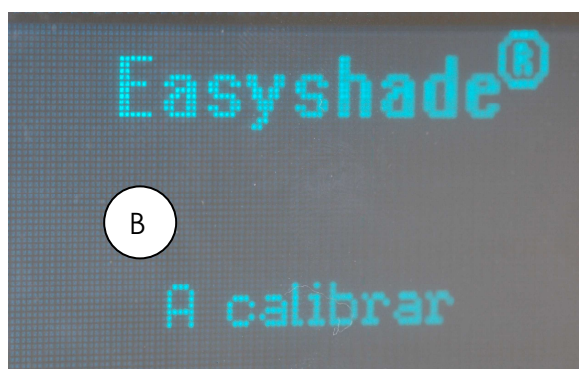
Determinação da cor inicial e final

A - Espectrofotômetro VITA Easyshade (Vident, Brea, CA, USA)

B – Informação na tela do espectrofotômetro antes de iniciar a leitura – (calibrar)

C –Calibragem completa do Espectrofotômetro VITA Easyshade, podendo iniciar a leitura da cor

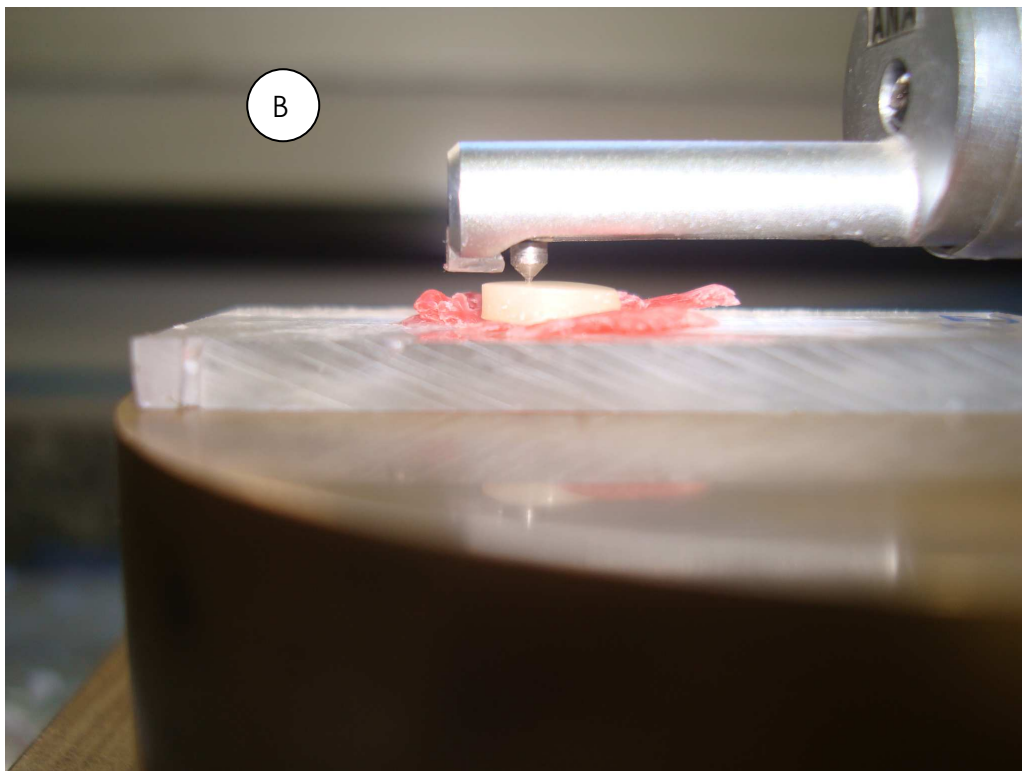
D - Espécime iluminado durante a realização da leitura da cor



APENDICE 6 - Ilustrações da metodologia empregada no capítulo 1, 2 e 3

A - Rugosímetro Surf-Corder (Kosaka Lab. SE 1700), para determinação da rugosidade superficial.

B – Visão aproximada da ponteira de diamante percorrendo o espécime

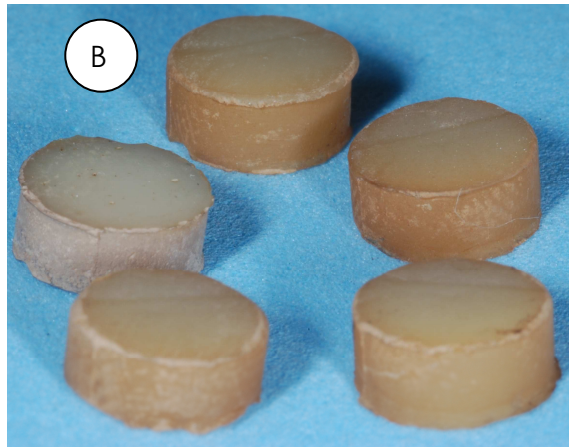
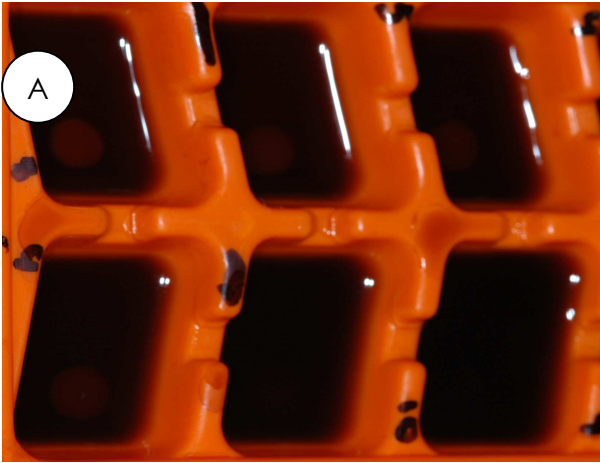


APENDICE 7 - Ilustrações da metodologia empregada no capítulo 2 e 3

Manchamento dos espécimes

A - Espécimes imersos em solução de café (Nescafé, Nestlé, Switzerland) por sete dias.

B – Espécimes após sete dias de imersão em solução de café



APENDICE 8 - Fluxograma representativo correspondente às etapas da pesquisa empregada na metodologia do capítulo 3

A - Resina compostas testadas no estudo Filtek supreme 3M, Tetric evo ceram Ivoclar Vivadent, Ceram X Duo denstply e Amelogen Ultradent;

B – inserção da resina na matriz metálica;

C – Espécime confeccionado nas dimensões 7mmX2mm e armazenamento do espécime por 24 horas em água deionizada a 37°C;

D -espectrofotômetro easy shade para avaliação da cor inicial;

E–verificação da cor inicial;

F - Aferição dos valores de rugosidade inicial com o rugosímetro;

G - Polimento dos espécimes com discos soflect 3M e pogo Dentsply;

H – Aferição dos valores de rugosidade final com o rugosímetro;

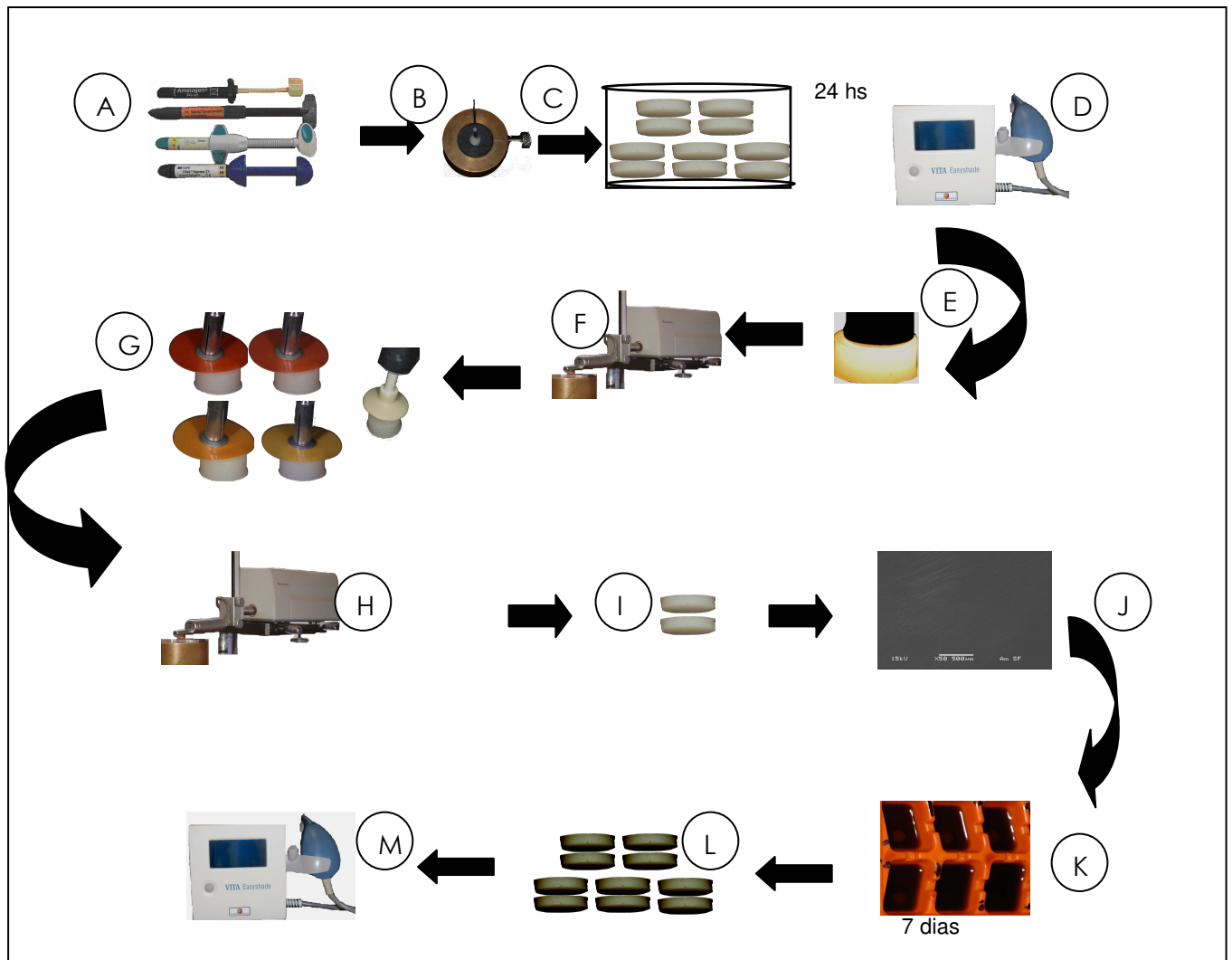
I – Dois espécimes selecionados aleatoriamente;

J – Observação em Microscopia Eletrônica de Varredura, com aumento de 50X e 1000X;

K – espécimes imersos em solução de café;

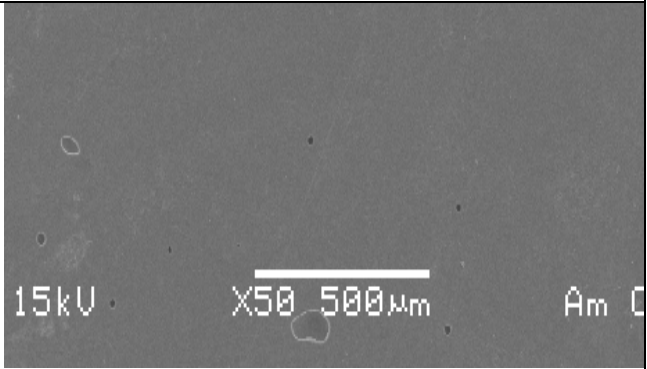
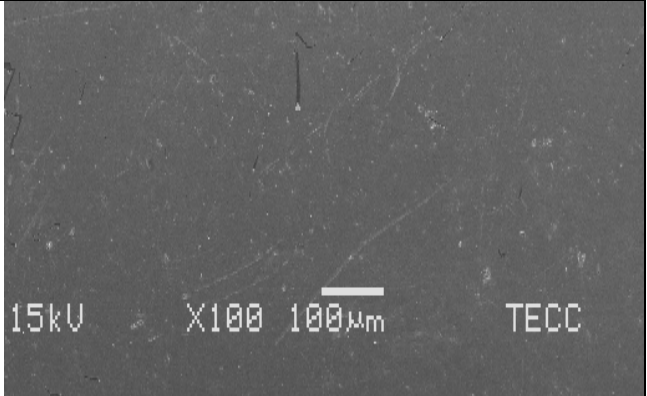
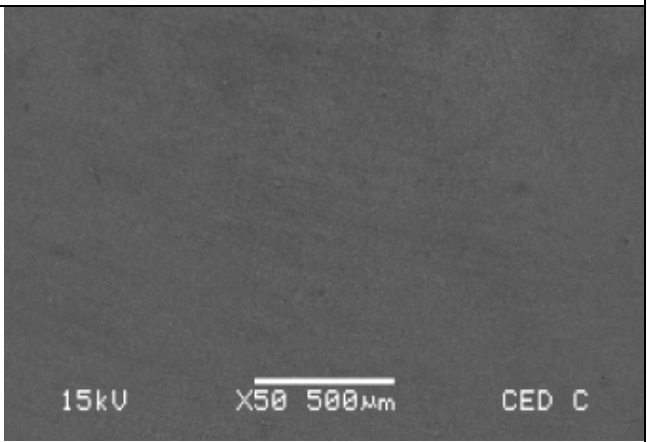
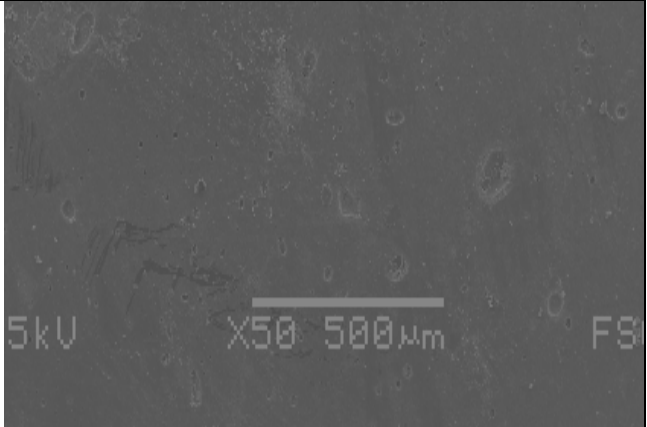
L – espécimes após sete dias de imersão em solução de café;

M- verificação da cor final



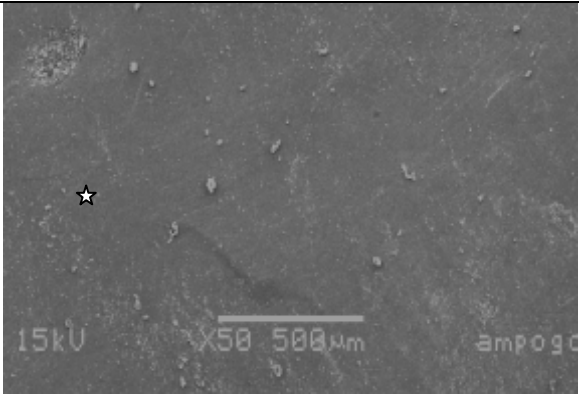
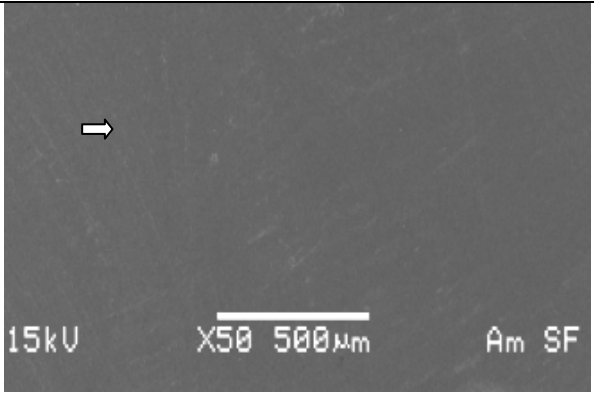
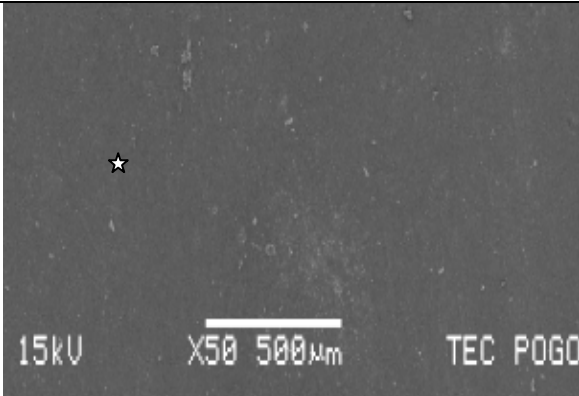
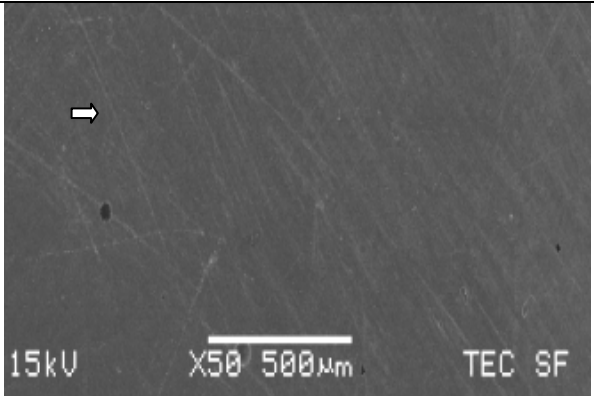
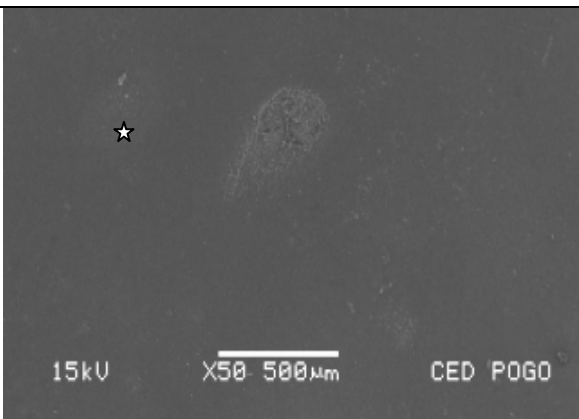
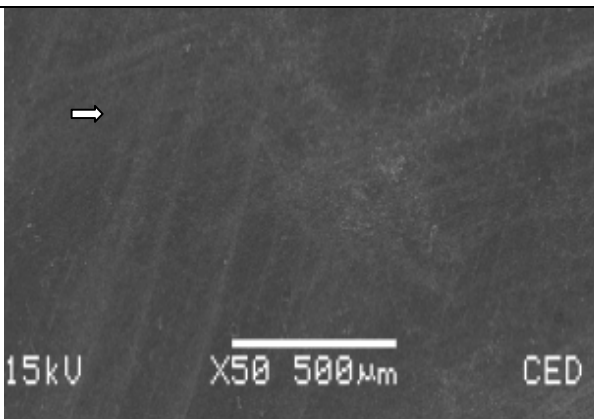
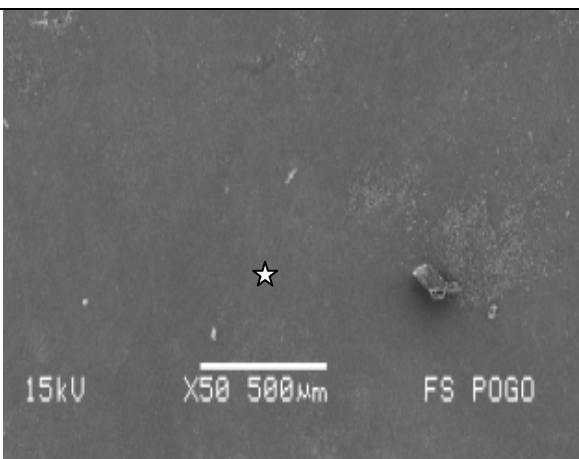
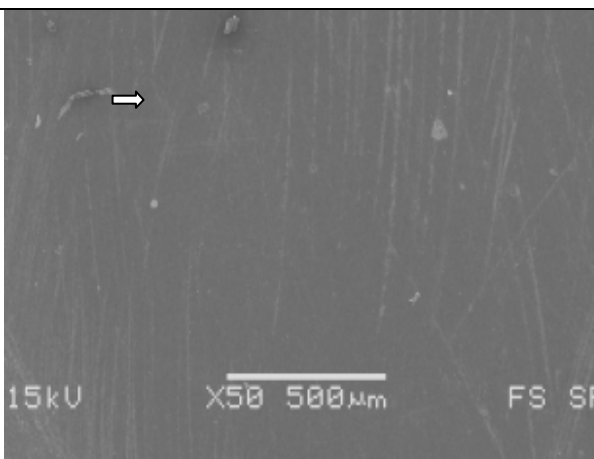
APÊNDICE 9 - Micrografia representativas, obtidas por meio de Microscopia Eletrônica de Varredura,

Fotomicrografia das resinas compostas Filtek supreme (FS); Tetric Evo Ceram (TEC); Ceram X Duo (CED) e Amelogem (AM) dispostas em linha encontram-se os tratamentos – controle (tira de poliéster); Note a lisura superficial produzida pelo contato com a matriz de poliéster. (MEV original 50X)

	Tira de poliéster
AM	
TEC	
CXD	
FS	

APÊNDICE 10 - Micrografia representativas, obtidas por meio de Microscopia Eletrônica de Varredura,

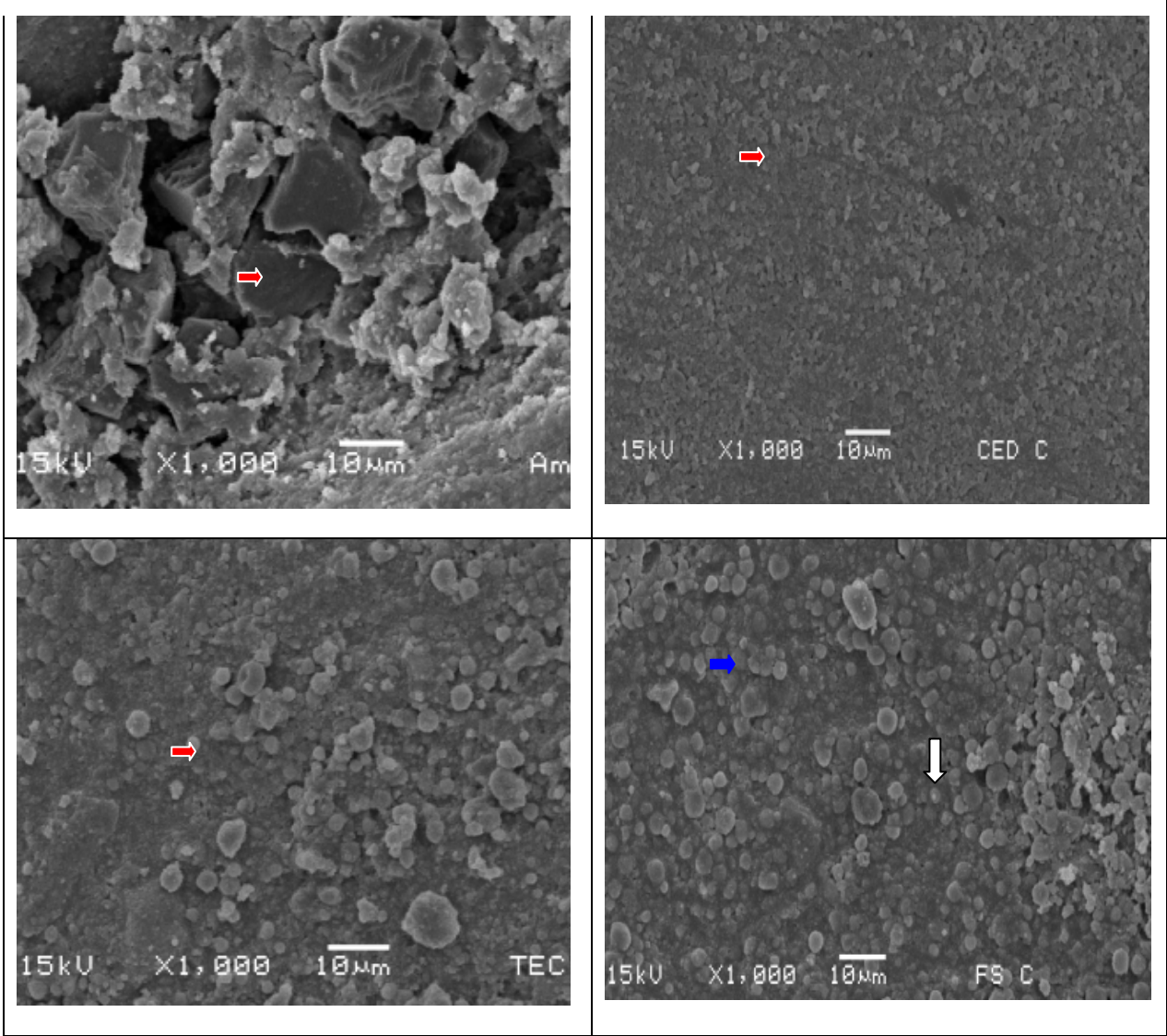
Fotomicrografia das resinas compostas Filtek supreme (FS); Tetric Evo Ceram (TEC); Ceram X Duo (CED) e Amelogen (AM) dispostas em linha. Nas colunas encontram-se os tratamentos – após polimento com discos soflex; após polimento com sistema PoGo (MEV original 50X) a superfície irregular produzida pelo sistema PoGo (estrelas); apesar das ranhuras produzidas (setas brancas) pelo sistema Sof-Lex (coluna 3) observa-se uma superfície mais uniforme nas amostras (aumento 50x).

	PoGo	Sof-Lex
AM		
TEC		
CXD		
FS		

APÊNDICE 11- Micrografia representativo, obtido por meio de Microscopia Eletrônica de Varredura.

Geometria das partículas das resinas, (MEV original 1000X). Observar o tamanho, a forma e distribuição das partículas de carga nos materiais. Amelogen – partículas de formas predominantemente de bordos irregulares e acentuados. TEC, CED e FS com partículas esféricas e características de compactação permitindo distribuição uniforme das partículas (setas vermelhas). FS – última coluna – observar a presença de nanoclusters (seta azul) e nanopartículas (seta branca).

GEOMETRIA DAS PARTÍCULAS



ANEXO 1 – Resolução CCPG/002/06 a qual dispõe a respeito do formato das teses de mestrado e doutorado aprovados pela UNICAMP (parte1)

INFORMAÇÃO CCPG/002/06

Tendo em vista a necessidade de revisão da regulamentação das normas sobre o formato e a impressão das dissertações de mestrado e teses de doutorados e com base no entendimento exarado no Parecer PG nº 1985/96, que trata da possibilidade do formato alternativo ao já estabelecido, a CCPG resolve:

Artigo 1º - O formato padrão das dissertações e teses de mestrado e doutorado da UNICAMP deverão obrigatoriamente conter:

- I. Capa com formato único ou em formato alternativo que deverá conter informações relativas ao nível (mestrado ou doutorado) e à Unidade de defesa, fazendo referência à Universidade Estadual de Campinas, sendo o projeto gráfico das capas definido pela PRPG.
- II. Primeira folha interna dando visibilidade à Universidade, a Unidade de defesa, ao nome do autor, ao título do trabalho, ao número de volumes (quando houver mais de um), ao nível (mestrado ou doutorado), a área de concentração, ao nome do orientador e co-orientador, ao local (cidade) e ao ano de depósito. No seu verso deve constar a ficha catalográfica.
- III. Folha de aprovação, dando visibilidade à Comissão Julgadora com as respectivas assinaturas.
- IV. Resumo em português e em inglês (ambos com no máximo 500 palavras).
- V. Sumário.
- VI. Corpo da dissertação ou tese dividido em tópicos estruturados de modo característico à área de conhecimento.
- VII. Referências, formatadas segundo normas de referenciamento definidas pela CPG da Unidade ou por critério do orientado.
- VIII. Todas as páginas deverão, obrigatoriamente, ser numeradas, inclusive páginas iniciais, divisões de capítulos, encartes, anexos, etc... As páginas iniciais poderão ser numeradas utilizando-se algarismos romanos em sua forma minúscula.
- IX. Todas as páginas com numeração “ímpar” serão impressas com “frente” e todas as páginas com numeração “par” serão impressas como “verso”.

§ 1º - A critério do autor e do orientador poderão ser incluídos: dedicatória; agradecimentos; epígrafe; lista de: ilustrações, tabelas, abreviaturas e siglas, símbolos glossário; apêndice; anexos.

§ 2º - A dissertação ou tese deverá ser apresentada na língua portuguesa, com exceção da possibilidade permitida no artigo 2º desta Informação.

§ 3º - As dissertações e teses cujo conteúdo versar sobre pesquisa envolvendo seres humanos, animais ou biossegurança, deverão apresentar anexos os respectivos documentos de aprovação.

Artigo 2º - A critério do orientador e com aprovação da CPG da Unidade, os capítulos e os apêndices poderão conter cópias de artigos de autoria ou de co-autoria do candidato já publicados ou submetidos para publicação em revistas científicas ou anais de congresso sujeito a arbitragem, escritos no idioma exigido pelo veículo de divulgação.

§ único – O orientador e o candidato deverão verificar junto às editoras a possibilidade de inclusão dos artigos na dissertação ou tese, em atendimento à legislação que rege o direito autoral, obtendo, se necessária, a competente autorização, deverão assinar declaração de que estão infringindo o direito autoral transferindo à editora.

Artigo 3º - Dependendo da área do conhecimento, a critério do orientador e com aprovação da CPG da Unidade, a dissertação ou tese poderá ser apresentada em formato alternativo, desde que observados os incisos I, II, III, IV, V e VII do artigo 1º.

Artigo 4º - Para impressão, na gráfica da Unicamp, dos exemplares definitivos de dissertação e teses defendidas, deverão ser adotados os seguintes procedimentos:

§ 1º - A solicitação para impressão dos exemplares de dissertação e teses poderá ser encaminhada à gráfica da Unicamp pelas Unidades, que se responsabilizarão pelo pagamento correspondente.

§ 2º - Um original da dissertação ou tese, em versão definitiva, impresso em folha tamanho carta, em uma só face, deve ser encaminhado à gráfica da Unicamp acompanhado do formulário “Requisição de Serviços Gráficos”, onde conste o número de exemplares solicitados.

§ 3º - A gráfica da Unicamp imprimirá os exemplares solicitados com capa padrão. Os exemplares solicitados serão retirados pelas Unidades em no máximo, cinco dias úteis para impressão preto e branco e 10 dias úteis para coloridas.

§ 4º - No formulário “Requisição de Serviços Gráfico” deverão estar indicadas as páginas cuja reprodução deva ser feita no padrão “cores” ou “foto”, ficando entendido que as demais páginas devam ser reproduzidas no padrão preto/branco comum.

§ 5º - As dissertações e teses serão reproduzidas no padrão frente e verso, exceção feita às páginas iniciais e divisões de capítulos; dissertações e teses com até 100 páginas serão reproduzidas no padrão apenas frente, exceção feita à página que contém a ficha catalográfica.

§ 6º - As páginas fornecidas para inserção deverão ser impressas em sua forma definitiva, ou seja, apenas frente ou frente/verso.

§ 7º - O Custo, em reais, de cada exemplar produzido pela gráfica será definido pela Administração Superior da Universidade.

Artigo 5º - É obrigatória a entrega de dois exemplares para homologação.

Artigo 6º - Esta Informação entrará em vigor na data de sua publicação, ficando revogadas as disposições em contrário, principalmente as Informações CCPG 001 e 002/98 e CCPG/001/00.

Campinas, 13 de setembro de 2006

Profa. Dra. Teresa Dib Zambon Atvars

Presidente

Comissão Central de Pós-Graduação

ANEXO 2 –Declaração do direito autoral transferido a editora quando a tese for defendida em formato alternativo



UNIVERSIDADE ESTADUAL DE CAMPINAS



Declaração

As cópias de artigo de minha autoria ou de minha co-autoria, já publicados ou submetidos para publicação em revista científicas ou anais de congresso sujeitos a arbitragem, que constam da minha Dissertação/Tese de Mestrado/Doutorado, intitulada “**Alterações nas características da superfície de compósitos restauradores submetidas a diferentes técnicas de acabamento e polimento**”, não infringem os dispositivos da Lei n.º9.610/98, nem o direito autoral de qualquer editora.

Campinas, 22 de abril de 2010

Vera Lucia Schmitt

RG n.º 2.126723 6

Autor(a)

Profa. Dra. Regina Maria Puppini Rontani

RG n.º10.723.931

Orientador