UNIVERSIDADE ESTADUAL PAULISTA
FACULDADE DE ODONTOLOGIA DE ARARAQUARA

Camila Cruz Lorenzetti

Influência da estabilidade de cor, rugosidade superficial e resistência de união em diferentes materiais restauradores CAD/CAM

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Orientador: Prof Dr José Roberto Cury Saad

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"Talvez não tenhamos conseguido fazer o melhor, mas lutamos para que o melhor fosse feito. Não somos o que deveríamos ser, não somos o que iremos ser.. mas Graças a Deus, não somos o que éramos".

Martin Luther King

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RESUMO

Com o avanço dos materiais cerâmicos e dos sistemas adesivos, aliados a alta exigência estética dos pacientes, restaurações cerâmicas têm sido cada vez mais utilizadas clinicamente. Restaurações cerâmicas realizadas pelo CAD/CAM aliam alta tecnologia com praticidade, especialmente os blocos que não requerem fase de cristalização em forno, obtendo resultados satisfatórios em curto tempo. Este estudo teve como objetivo avaliar a estabilidade de cor, rugosidade superficial e resistência de união de diferentes materiais restauradores do sistema CAD/CAM e uma resina composta. Foram utilizados os blocos cerâmicos na cor A2: Celtra Duo- ZL (Dentsply/Sirona), Enamic – HC (Vita), Lava Ultimate – NC (3M) e Resina Composta Filtek Z350XT- CR (3M). Para os estudos 1 e 2, foram realizados 2 tipos de procedimentos de polimento: borrachas Ceramisté – Standard, Ultra e Ultra II (Shofu); borrachas Ceramisté + pasta porcelize (Cosmedent) + discos de feltro Flexibuff (Cosmedent) e posteriormente realizadas imersões em saliva artificial(S1), café(2) e coca-cola(3). Espécimes para cada grupo (n=10), foram imersos em cada solução armazenada em estufa a 37ºC por 60 dias. As leituras de cor (ΔE) foram realizadas nos tempos inicial, 30 dias, 60 dias e repolimento. As leituras de rugosidade superficial (Ra) foram realizadas inicialmente antes das imersões, após 30 dias e após 60 dias de imersão. Estudo 3 avaliou diferentes técnicas de adesão (acido fluorídrico+silano+adesivo; acido fluorídrico+adesivo universal; jateamento sílica+silano+adesivo; jateamento sílica+adesivo universal). Os estudos 1 e 2 apresentaram resultados semelhantes nos quais a solução S2 apresentou os maiores valores de Ra e ΔE, não apresentando diferença significativa entre os sistemas de polimento. No estudo 3 mostrou que aplicação da sílica para os grupos ZL e HC diminuíram os valores de resistência de união, aumentando para os NC e CR. O uso do silano como passo separado não mostrou diferença do silano utilizado junto ao adesivo universal.

Palavras chave: Ceramica. Materiais dentários. Polimento. Cor. Reparação de Restauração Dentária.

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ABSTRACT

Considering the advancement of ceramic materials and adhesive systems, allied to the high esthetic requirement of patients, ceramic restorations have been increasingly used clinically. Ceramic restorations performed by CAD / CAM combine high technology with practicality, especially the blocks that do not require crystallization phase in the oven, obtaining satisfactory results in a short time. This study aimed to evaluate the color stability, surface roughness and bond strength of different CAD / CAM system restorative materials and a composite resin. The ceramic blocks in the A2 color were: Celtra Duo-ZL (Dentsply / Sirona), Enamic-HC (Vita), Lava Ultimate-NC (3M) and Filtek Z350XT-CR (3M) Composite Resin. For studies 1 and 2, two types of polishing procedures were performed: Ceramisté - Standard, Ultra and Ultra II rubbers (Shofu); Ceramisté rubbers + paste porcelain (Cosmedent) + Flexibuff felt discs (Cosmedent) and subsequently immersed in artificial saliva (S1), coffee (2) and coke (3). Specimens for each group (n = 10) were immersed in each solution stored in an oven at 37ºC for 60 days. Color readings (E) were performed at the initial, 30 days, 60 days and repolder times. Surface roughness readings (Ra) were performed before immersion, after 30 days and after 60 days of immersion. Study 3 evaluated different adhesion techniques (hydrofluoric acid + silane + adhesive, hydrofluoric acid + universal adhesive, silica sandblasting + silane + adhesive, silica sandblasting + universal adhesive). Studies 1 and 2 presented similar results in which the solution S2 presented the highest values of Ra and E, showing no significant difference between the polishing systems. In study 3 it was shown that silica application to the ZL and HC groups decreased the bond strength values, increasing for NC and CR. The use of the silane as a separate step showed no difference in the silane used with the universal adhesive.

Keywords: Ceramic. Dental Materials. Polishing. Color. Repair Dental Restoration.

SUMÁRIO

1 INTRODUÇÃO

O sistema CAD/CAM (computer-aided design/computer-aided manufacturing), uma tecnologia na qual o desenho e a manufatura são auxiliados pelo computador, tem crescido e ganhado cada vez mais espaço dentro da odontologia. Há uma variedade de materiais restauradores para o sistema CAD/CAM sendo cada vez mais comercializados e desenvolvidos, entre eles diferentes tipos de cerâmicas, resinas compostas e resinas acrílicas[1]. Alguns dos materiais disponíveis podem passar por procedimentos após a frezagem apenas com equipamentos disponíveis no consultório[2], enquanto outros necessitam passar por processos mais complexos como ter que ir ao forno para a queima do material ou para o glaze[3,4]. O sistema Cerec® foi comercializado pela primeira vez em 1985, e desde lá tem passado por uma evolução significativa em relação à eficiência do sistema, capacidade e tipos de materiais disponíveis[5].

Instrumentos e técnicas de acabamento e polimento possibilitam uma lisura superficial, melhorando a resistência flexural da restauração e consequentemente a longevidade da mesma[2,6], diminuindo a aderência bacteriana na superfície[7,8,9]. Procedimentos de acabamento e polimento também melhoram a aparência estética das restaurações cerâmicas confeccionadas pela fresagem no sistema CAD/CAM. Alguns estudos mostraram que o uso de borrachas de polimento e pastas diamantadas de polimento resulta em superfícies polidas com lisura[10,11]. Além dos processos mecânicos de polimento, outro fator a considerar é a aplicação do glaze. Em 2010, Yilmaz and Ozkan[12] compararam a rugosidade superficial de algumas cerâmicas após o tratamento com glaze, pastas, borrachas, discos abrasivos e concluíram que a lisura de superfície obtida a partir do glaze e uso de borrachas é clinicamente aceitável.

O uso de restaurações cerâmicas aliado à pequena espessura tem sido cada vez mais empregado clinicamente. Com o avanço dos sistemas adesivos e de materiais restauradores, aliados a grande exigência dos pacientes de um resultado estético satisfatório e com poucos desgastes dentários, tem crescido o uso das restaurações minimamente invasivas, apresentando-se assim em finas espessuras. A união química tanto dos cimentos resinosos com a superfície dentária quanto dos cimentos com a superfície vítrea da cerâmica, possibilita uma grande resistência de união da restauração cerâmica, com longevidade e baixo índice de fratura[13].

Pelo alto consumo de alimentos e bebidas pigmentantes, há a preocupação da impregnação de corantes em restaurações, podendo haver alterações ópticas do material restaurador. Visto que com o procedimento de polimento é possível obter maior lisura superficial, pode-se assim minimizar a impregnação de pigmentos. A mudança de cor de alguns materiais CAD/CAM pode levar a limitações clínicas, principalmente em regiões anteriores, e o aumento da rugosidade ao longo do tempo resulta no aumento de impregnação de pigmento e de acúmulo de placa bacteriana, o que pode também limitar o uso clínico.

Embora materiais restauradores CAD/CAM tenham boas propriedades físicas e mecânicas, sob diferentes condições clínicas, eles podem falhar. Uma abordagem conservadora sugere o reparo de restaurações, em alguns casos de falhas, ao invés de substituí-las para preservar a estrutura dental, para ser um procedimento mais rápido e menos oneroso[14]. Vários estudos têm relatado diferentes protocolos para este procedimento[15,16], incluindo métodos químicos, mecânicos ou físico-mecânicos para melhorar a área de ligação. Entre eles, estão o ácido fluorídrico, abrasão de partículas no ar com óxido de alumínio, revestimento de sílica químico-química, aplicação de silano, uso de adesivo dental ou a combinação de alguns deles[16]. A utilização de técnicas com resina composta como material de reparo é uma alternativa estética e funcional para a realização de reparo intraoral[15].

Com a praticidade das restaurações obtidas pelo sistema CAD/CAM e a possibilidade da instalação das mesmas em poucas horas clínica, pode ocorrer cada vez menos o uso de procedimentos laboratoriais para o polimento final. Devido a pouca documentação de estudos que avaliem sistemas de polimento e materiais de CAD/CAM atualmente utilizados, e protocolos para reparo restaurador, este estudo avaliou a estabilidade de cor, rugosidade superficial de alguns destes materiais com técnicas de polimento após imersão em bebidas pigmentantes e técnicas de adesão na resistência de união desses materiais com resina composta.

2 OBJETIVO GERAL

Avaliar a influência da estabilidade de cor, rugosidade superficial e resistência de união em diferentes materiais restauradores CAD/CAM.

2.1 Objetivos Específicos

Publicação 1: Effect of immersion solutions and polishing techniques on color stability of different CAD/CAM materials and a composite resin at different timepoints

Publicação 2: Effect on surface roughness of immersion solutions following two polishing techniques in different CAD/CAM materials and a composite resin

Publicação 3: Analysis of the bond strength of different repair techniques between indirect restorative materials and composite resin
Analysis between indirect restorative materials and composite resin on bond strength of different repair techniques

3 PUBLICAÇÕES

3.1 Publicação 1*

Effect of immersion solutions and polishing techniques on color stability of different CAD/CAM materials and a composite resin at different timepoints

* Artigo nas normas da revista "Clinical Oral Investigations"

Effect of immersion solutions and polishing techniques on color stability of different CAD/CAM materials and a composite resin at different timepoints

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ABSTRACT

Objective: To investigate the influence of immersion solutions and polishing techniques on color change of CAD/CAM materials and a composite resin at different time.

Material and Methods: Specimens (n=10 each group) obtained from a resin composite Filtek Z350xt - M1 (nanofilled composite) and CAD/CAM blocks Lava Ultimate – M2 (nanoceramic), Enamic – M3 (Feldspathic-composite Hybrid Ceramic), and Celtra Duo – M4 (Zirconia-reinforced lithium-silicate) were polished with different polishing protocol (P1: Ceramisté rubbers; P2: Ceramisté rubbers+Diamond paste+felt disc) and immersed for 60 days in solutions of artificial saliva (S1), Coffee (S2) and Coca-Cola®(S3). Color change (ΔE) was measured with a spectrophotometer according to CIE-Lab in 3 different times (before immersions- T0; after 30 days – T, and after 60 days of immersion - T2). Statistical analyses were performed using repeated measure analysis of variance (ANOVA) and Tukey test for each material ($\alpha = 0.05$).

Results: For all materials tested**,** significant difference was verified for the factor “time” ($p<0.001$), the interaction “time and solution” ($p<0.001$); and “solution” ($p<0.001$). However, no significant difference was identified for the interactions “time and polishing”, “time, solution and polishing”, and “solution and polishing”.

Conclusion: The highest ΔE were observed in coffee solution. Repolishing procedure decreased significantly ΔE for composite resin, Lava Ultimate and Enamic.

Keywords: CAD/CAM; ceramic; composite resin; color; polishing

Introduction

Computer-aided design and computer-aided manufacturing (CAD/CAM) is a technology that allows to create restorations with high quality in a short period of time. Due to the manufacturing process by high pressure and high temperature [1], the CAD/CAM materials can offer enhanced mechanical properties [2], better color stability, and less residual monomer [3].

Even though the CAD/CAM restorations show advantages to conventional ceramic made by laboratory technicians, it can show some defects. Polishing procedures can eliminate some these defects that can appear from machining process, also offering smooth surfaces, high gloss, color stability [4] and patient's comfort [5].

Some materials have been developed to balance the ceramic properties and composite properties, which can ally the benefits of both materials, as durability and color stability of ceramic with low abrasiveness and flexural properties of composite resins [6,1]. Examples of them, there are a resin nanoceramic and a hybrid ceramic containing polymeric matrix, that ally the advantages of composite materials [6].

Color stability is as an important property as the mechanical one, that can affect the longevity of a restoration and its quality [7]. Descoloration in the oral environment can be affected by staining solutions, exposure time in these solutions, surface roughness, and composition of the restorative material [8,9].

Several factors influence restorations in the oral cavity, such as humidity, alimentary diet, smoking habits [10]. Staining and acidic beverages are frequently consumed by the population around the world, and it can affect the surface of some materials. The success of a restorative treatment is not only dependent by mechanical properties, but also by quality esthetic [11].

Considering the development of new CAD/CAM materials that try to ally ceramic and composite advantages, there are few studies in literature showing their behavior in staining solutions, and if they show similarity with ceramic or composite resin on color stability.

Therefore, the present study investigated the effects of immersion solutions and polishing techniques on the color stability of different CAD/CAM materials and a composite resin at different time-points. The null hypothesis was that the immersion solutions, polishing techniques and different time would not affect the color stability of each material.

MATERIAL AND METHODS

The restorative materials and their composition are shown on Table 1. The materials used were blocks used were Composite Resin Filtek Z350 XT – M1 (A2 - Enamel), Lava Ultimate – M2 (color A2-HT), Enamic – M3 (color 2 M2-HT), Celtra Duo – M4 (color A2-LT).

Polishing / repolishing systems:

-Polishing system 1 (P1): Ceramisté 3-step polishing rubbers - Standard, Ultra and Ultra II (Shofu Dental GmbH, Ratingen, Germany)

-Polishing system 2 (P2): Ceramisté 3-step polishing rubbers - Standard, Ultra and Ultra II (Shofu Dental GmbH, Ratingen, Germany) + Porcelize paste (Cosmedent, Inc., Chicago, USA) + discs of felt with extra fine granulation Flexibuff (Cosmedent, Inc., Chicago, USA).

Immersion Solutions

S1: Artificial saliva (control group)

S2: Nespresso® Arpeggio Coffee

S3: Coca-Cola®

Experimental design

It is an experimental study presenting as dependent variable the color stability and as independent variables the restorative materials, polishing systems, immersion solutions and different times of evaluation.

The number of specimens used was 10 specimens per group, according to the studies on scientific literature about color stability evaluation.

Specimens preparation

The ceramic specimens were obtained from CAD/CAM blocks, which were cut using a water-cooled low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain rectangular shape specimens (6 x 6 x 1.5mm). The composite resin specimens Filtek Z350XT (3M) were made in a matrix made of Zetalabor (Zhermark) condensation silicone in dimensions 6 x 6 x 1.5mm. The resin was inserted by single increment in the silicon matrix, placing a polyester strip and a weight of 1kg on it for surface standardization.

Subsequently, the specimens were included in a circular matrix with a dimension of 10mm in diameter and 1.5mm in height filled with polyester resin, leaving the ceramic surface exposed. After 24 hours of inclusion in the matrix with polyester resin, the specimens were stantardized in a polishing machine (DP-10 Panambra, Struers, Ballerup, Denmark) under refrigeration with silicon carbide abrasive papers (600 and 800 grit). The final thickness (1.5mm) was measured using a digital caliper.

Polishing Procedures

The specimens were washed in water, dried with paper towel and followed by two polishing procedures:

- Ceramisté (Standard, Ultra and Ultra II): the polishing rubbers were used for 20 seconds in both directions of movement, at low speed.

- Ceramisté (Standard, Ultra and Ultra II) + Porcelize 1µm + Flexibuff felt discs: the polishing rubbers for 20 seconds in both directions of movement, at low speed. Then the 1m granulation paste was applied with the felt disk for more 20 seconds.

After the polishing procedures, the specimens were ultrasonically washed in distilled water for 2 minutes, and then stored in artificial saliva for 24 hours in an oven at 37 ° C ± 1 ° C (EBC1-Odontobras- Trade Eq, Medical-Dental LTDA, Ribeirão Preto, SP, Brazil) for rehydration prior to start of storage in pigment solutions.

Immersion procedures

All specimens were removed from the storage in artificial saliva, ultrasonically washed with distilled water and dried. Then, they were immersed in solutions of new artificial saliva, Nespresso® Arpeggio and Coca-Cola® stored in an incubator at 37°C ± 1°C for 60 days, renewing the solutions every 5 days to avoid contamination.

Measurement of optical parameters

Color readings were performed using a digital spectrophotometer CM-2600d spectrophotometer (Konica Minolta, Japan), making 3 measurements in each specimen, and considering the average of them. The CM-2600d spectrophotometer is a portable measuring device for evaluating color and brightness characteristics of small and large samples, especially on flat surfaces. The instrument was previously calibrated according to the manufacturer's recommendation, using the instrument’s calibrator. Then, the tip of the spectrophotometer was directly positioned on a white background, and perpendicular to the polished surface of the specimens. The light beam was fired and the color was performed according to the L *, a *, b * values of the system CIE-Lab. The reading moments were performed before the first immersion in the solutions (baseline - T0), after 30 days (T1), after 60 days of immersion (T2). A new reading of all the specimens was performed after procedures of re-polishing of the specimens performed after the immersion in the solutions (T3).

Statistical analysis

The normality of data distribution and sphericity were verified by Shapiro-Wilk ($p=0.054-0.945$) and Mauchly ($p\geq0.067$ for M1, M3 and M4; and $p<0.001$ for M2) tests, respectively. The Greenhouse-Geisser correction factor was used for M2, as the assumption of sphericity was not attended. Data was analyzed using the software PASW Statistics 25.0 (SPSS Inc, Chicago, IL, USA), with a confidence interval set at 95%. A repeated measure analysis of variance (ANOVA) and Tukey tests were used to evaluate the effect of immersion solution (S1, S2 and S3), polishing technique (P1 and P2) and assessment time (T1, T2 and T3) on the dependent factor (Color change - ΔE) for each material tested (M1, M2, M3 and M4).

RESULTS

The mean and standard deviation for color change (ΔE) of the specimens, according to material, immersion solution, polishing technique and assessment time, are shown in Table 2.

Material 1

Significant difference was verified for the factor "time" ($p<0.001$) and the interaction "time and solution" ($p<0.001$); however, no significant difference was identified for the interactions "time and polishing" ($p=0.703$) and "time, solution and polishing" ($p=0.512$). Significant difference was also observed for the factor "solution" ($p<0.001$), but not for "polishing" ($p=0.202$) or the interaction "solution and polishing" ($p=0.957$). Specimens immersed in solution 2 showed greater color change than the ones immersed in solutions 1 and 3 at all three assessment times, independent of the polishing technique. The ΔE values for solution 2 significantly decreased at T3 for both polishing techniques. The other solutions maintained similar color change at all assessment times, without any effect of the type of polishing. The mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time is shown in Figure 1.

Material 2

The repeated measures ANOVA identified significant difference for the factor "time" ($p<0.001$) and the interaction "time and solution" ($p<0.001$). No significant difference was verified for the interaction "time and polishing" ($p=0.106$) and "time, solution and polishing" ($p=0.870$). Significant difference was observed for the factors "solution" ($p<0.001$) and "polishing" ($p=0.048$); however, no difference was found for the interaction “solution and polishing” ($p=0.551$). Solutions 1 and 3 presented similar ΔE values among the different assessment time, despite the polishing technique. The greatest color change values were obtained for specimens immersed in solution 2 at the assessment times of T1 and T2. The values reduced at T3 for both polishing techniques and reached statistical significance when associated to polishing technique 2. The mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time is shown in Figure 2.

Material 3

Significant difference was found for the factor “time” ($p<0.001$) and the interaction “time and solution” ($p<0.001$). There was no significant difference for the interaction “time and polishing” ($p=0.331$) and “time, solution and polishing” ($p=0.067$). The ANOVA also verified significant difference for the factor “solution” ($p<0.001$) and for “polishing” ($p=0.022$); however, it did not identified difference for the interaction “solution and polishing” ($p=0.335$). Regardless of the polishing technique and assessment time, specimens immersed in solution 2 showed the greatest color change. The values for this group decreased at T3 and were statistically lower for specimens polished using technique 1. Specimens immersed in solutions 1 and 3 presented similar color change, independently of polishing technique and assessment time. The mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time is shown in Figure 3.

Material 4

Significant difference was verified for the factor “time” ($p<0.001$) and the interaction “time and solution” ($p<0.001$); however, repeated measures ANOVA did not identify difference for the interactions “time and polishing” ($p=0.880$) and “time, solution and polishing” ($p=0.230$). No significant difference was also observed for the factor “polishing” ($p=0.971$) and the interaction “solution and polishing” ($p=0.793$); however, there was a significant difference for the factor “solution” ($p<0.001$). Specimens immersed in solution 2 had the greatest color change at all assessment times, while the ones immersed in solutions 1 and 3 showed similar ΔE values among themselves. Polishing technique and assessment time did not have any effect on color change for all immersion solutions. The mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time is shown in Figure 4.

DISCUSSION

Considering that the color change was significantly affected by the immersion solution S2 in all restorative materials evaluated and by the time T3, the null hypothesis of this study was partially rejected.

Polishing procedure is an important step of a restorative treatment because can affect directly the surface roughness, impregnation of pigments and plaque, wear against the antagonist tooth. Some studies have shown smoother surface for polishing system than glazing in ceramic in oven [12]. Silva et al., [13] investigated several polishing systems, among the rubber points, diamond paste, felt discs, and all of them were effective in reducing the surface roughness of lithium disilicate ceramic. The color stability of this study was not affected by the polishing technique, which can be influenced by some factors as restorative material composition, polishing system composition, polishing pressure, polishing time. Heintze et al., [14] concluded that different applied press-on force showed different result for different restorative material, and the surface roughness and surface gloss were time-dependent.

Repolishing procedure showed be an effective procedure for M1, M2 and M3 when they were exposed for 60 day in coffee solution. A repolishing may remove the degraded resinous matrix of the specimens after immersed on solutions [7]. It can suggest the staining was not deep, what is easier to decrease the change after the repolishing procedure, removing the superficial staining. Garoushi et al. [15] showed a decrease in color alteration (ΔE) after submitting the specimens in solutions as coffee, Pepsi, tea and water to repolishing procedure. Some other studies also observed a significant decrease in ΔE after to repolish the specimens immersed on coffee [16, 17].

Although Celtra Duo has shown significant difference for S2, it was not possible to observe a decrease in T3, after the repolishing. It can suggest that the polishing techniques were not effective enough to this material.

CAD/CAM materials can show color stability due to the way it is manufactured. However, Arocha et al. [9], after comparing composites resins processed by and by indirect laboratory, immersed in staining solutions, the CAD/CAM composites showed the highest ΔE.

Among several reasons, color changes occur as result of plaque accumulation, chemical degradation, surface roughness, water sorption. Enamic consists of 33wt% hydrophilic particles (TEGDMA), which may result in great water sorption, and this way, to allow the infiltration of a hydrophilic colorant into the resin matrix [18]. In this present study, Enamic (M3) showed significant color change in S2 for P1, decreasing significantly ΔE after the repolishing.

Groups in M1 and M2 showed similar behavior, regardless of time, polishing system and immersion media. The same was showed by Acar et al.[19], which evaluated color change for composite resin, nanoceramic, hybrid ceramic and litium disilicate after thermocycling with coffee and, it showed similar behavior for composite resin and nanoceramic. The highest and significant ΔE values were observed in the coffee solution, even when compared with Coca-Cola®. Similar results were by Colombo et al.[20], that showed color change for coffee while did not show influence in coca-cola immersion for the CAD/CAM zirconia ceramic samples evaluated. Previous studies have shown that coffee is one of the most cause for significant color changes as in composite resins [21] as in ceramics [22]. Color change by coffee can be attributed to the penetration of the yellow colorant into the material microstructure. High uptake occurs in materials immersed in solutions with pH from 4 to 6. Considering the coffee within this ranging, this can have affected the sorption and consequently, the high color change for this solution [23, 24]. Another property than can affect is due to the coffee is a solution of low polarity, allowing its colorant to penetrate into the resin matrix [9, 24].

CONCLUSION

Coffee was the solution with showed the highest and significant ΔE for all restorative material evaluated. Overall, the polishing system with or without diamond paste and felt disc did not affect the color change. The repolishing procedure decreased significantly the color change for Composite Resin, Nanoceramic and Hybrid Ceramic. It can show the importance for periodic repolishing procedures on restorative treatments.

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Table 1- Materials and composition

Material; Manufacturer	Classification	Composition
Filtek Z350 XT; 3M ESPE Dental Products, St. Paul, MN	Nanofilled composite	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA, sílica filler, zirconia/sílica cluster filler
Lava Ultimate; 3M ESPE Dental Products, St. Paul, MN	Resin nanoceramic CAD/CAM block	Bis-GMA, UDMA, Bis-EMA, TEGDMA with 80%wt, 20-nm silica and 4- to 11-nm zirconia nanoparticles, and zirconia/silica nanoclusters
Enamic; Vita Zahnfabrik, Germany	Hybrid ceramic CAD/CAM block	UDMA, TEGDMA, 86wt% Feldspar ceramic enriched with aluminum oxide
Celtra Duo; Dentsply Sirona	Zirconia-reinforced lithium silicate ceramic block CAD/CAM	Lithium silicate with 10% ZrO_2
Ceramisté; Shofu Dental GmbH, Ratin- gen, Germany	Silicon Rubber	Silicon carbide impregnated polishers
Porcelize Paste; Cosmedent, Inc., Chicago, USA	Diamond paste	Diamond particles 1µm

Table 2- Mean ± standard deviation for color change (ΔE) for each material, according to solution/polishing and assessment time.

		T1		T2		T3	
		P1	P2	P1	P2	P1	P2
M1	S1	2.53 ± 2.10	2.82 ± 2.09	3.11 ± 1.70	4.04 ± 2.38	3.35 ± 1.94	4.63 ± 3.01
	S2	14.22 ± 4.19	16.12 ± 4.18	15.87 ± 3.92	16.71 ± 3.76	11.34 ± 2.07	11.06 ± 2.53
	S3	3.07 ± 1.77	3.34 ± 2.51	2.78 ± 2.08	3.91 ± 2.67	3.25 ± 1.71	3.30 ± 2.29
M2	S1	4.33 ± 1.33	5.67 ± 3.01	4.26 ± 2.67	5.21 ± 3.10	4.16 ± 2.14	5.43 ± 2.98
	S2	17.80 ± 5.30	18.51 ± 5.91	11.92 ± 3.21	15.48 ± 3.20	8.22 ± 3.01	9.85 ± 3.01
	S3	3.24 ± 1.87	4.00 ± 3.29	4.54 ± 1.85	4.15 ± 2.55	3.42 ± 1.66	4.23 ± 2.79
M3	S1	4.90 ± 2.71	2.57 ± 1.46	4.41 ± 2.47	1.90 ± 0.67	3.99 ± 2.46	2.20 ± 1.02
	S2	14.90 ± 5.40	12.46 ± 4.52	13.60 ± 3.62	10.94 ± 2.97	7.24 ± 2.53	8.48 ± 3.19
	S3	2.86 ± 1.86	2.86 ± 1.39	4.51 ± 2.43	4.84 ± 2.39	3.66 ± 1.94	2.53 ± 1.70
M4	S1	3.53 ± 1.33	2.95 ± 1.64	3.49 ± 1.85	2.35 ± 1.63	4.42 ± 2.25	4.75 ± 2.76
	S2	9.80 ± 3.74	9.97 ± 4.33	11.78 ± 3.18	13.77 ± 5.49	10.94 ± 3.57	10.39 ± 3.68
	S3	2.07 ± 1.71	2.46 ± 2.28	4.63 ± 2.60	4.43 ± 1.87	2.83 ± 1.68	2.62 ± 1.03

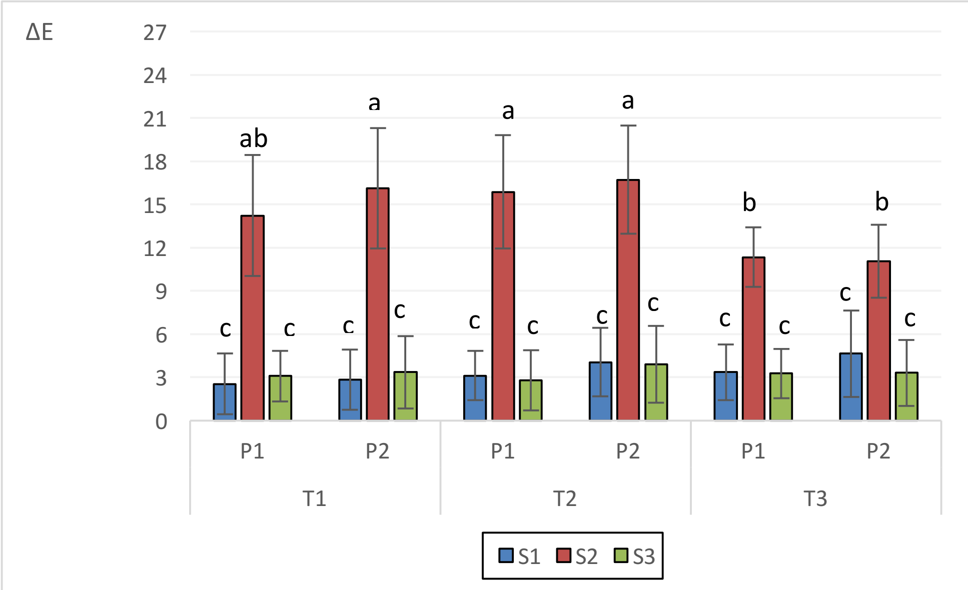

Figure 1 – Mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time for Material 1. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

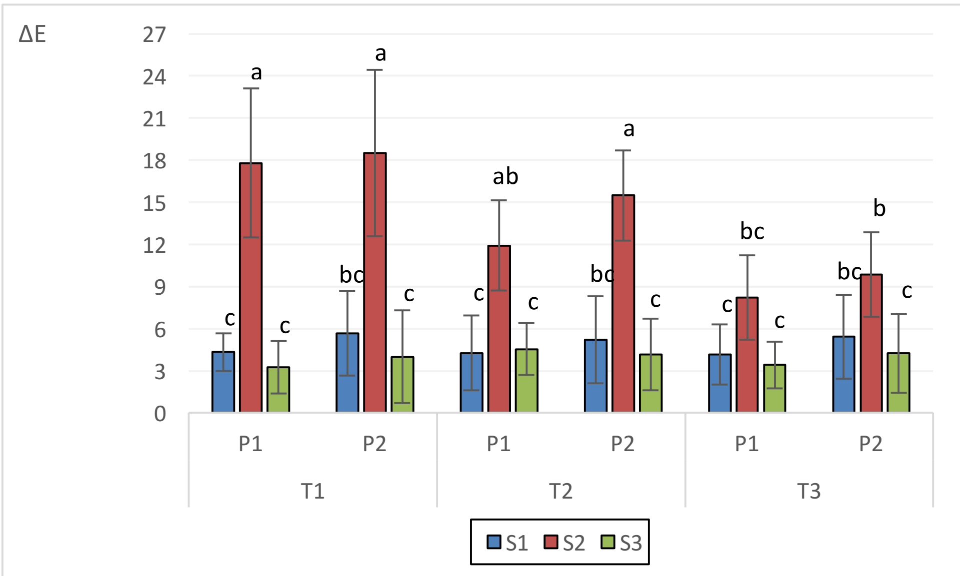

Figure 2 – Mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time for Material 2. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

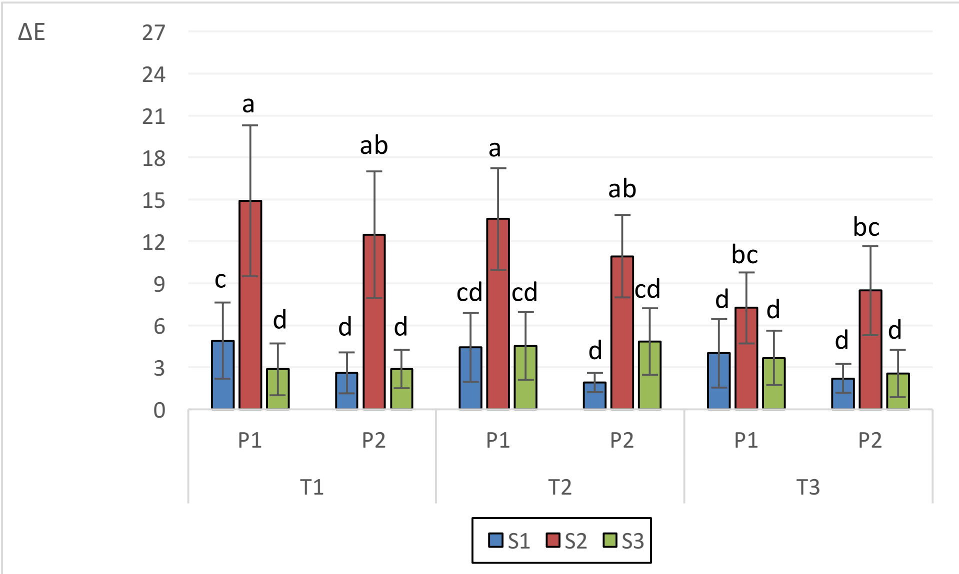

Figure 3 – Mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time for Material 3. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

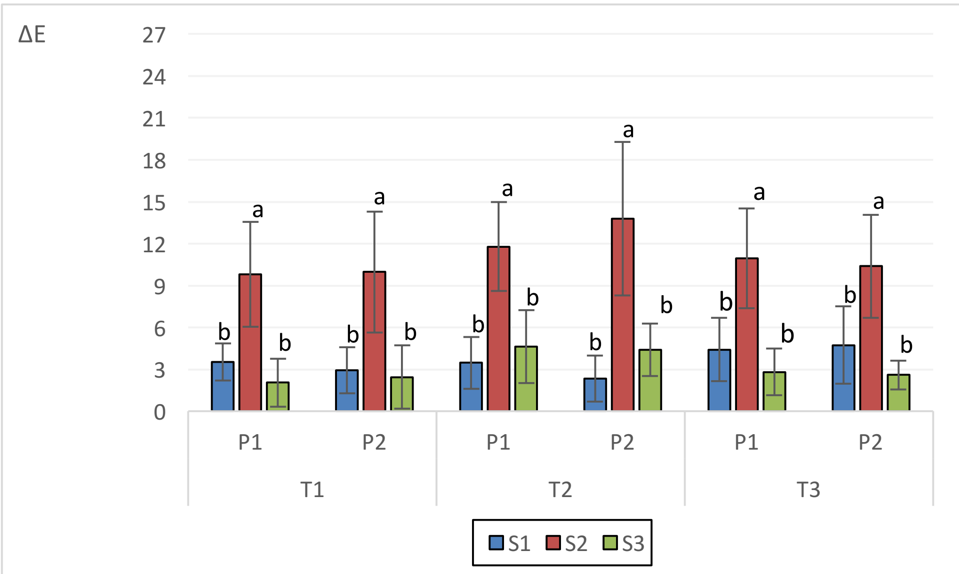

Figure 4 – Mean and standard deviation for color change (ΔE) according to solution/polishing and assessment time for Material 4. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

3.2 Publicação 2*

Effect on surface roughness of immersion solutions following two polishing techniques in different CAD/CAM materials and a composite resin

*Artigo nas normas da revista "Clinical Oral Investigations"

Effect on surface roughness of immersion solutions following two polishing techniques in different CAD/CAM materials and a composite resin

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ABSTRACT

Objective: To analysis the surface roughness of different CAD/CAM materials and a composite resin in immersion solutions and two polishing techniques in different timepoints.

Material and Methods: Specimens (n=10 each group) obtained from a resin composite Filtek Z350xt - M1 (nanofilled composite) and CAD/CAM blocks Lava Ultimate – M2 (nanoceramic), Enamic – M3 (Feldspathic-composite Hybrid Ceramic), and Celtra Duo – M4 (Zirconia-reinforced lithium-silicate) were polished with different polishing protocol (P1: Ceramisté rubbers; P2: Ceramisté rubbers+Diamond paste+felt disc) and immersed for 60 days in solutions of artificial saliva (S1), Coffee(S2) and Coca-Cola®(S3). Surface roughness was measured with a profilometer in 3 different times (before immersions- T0; after 30 days – T, and after 60 days of immersion - T2) and analyzed in SEM micrographs. Statistical analyses were performed using repeated measure analysis of variance (ANOVA) and Tukey test for each material ($\alpha = 0.05$).

Results: All materials showed significant difference for “time” ($p<0.05$); for the interactions “time and solution” ($p<0,05$), observing different results in other factors for each material. The highest values were in S2 in T2.

Conclusion: Polishing technique did not affect significantly all restorative materials tested, showing the highest surface roughness for coffee solution after 60 days of immersion.

Keywords: CAD/CAM; ceramic; composite resin; roughness; polishing

Introduction

Porcelains made by laboratory technicians require skills during condensation, in the firing process, and appropriate powder/liquid mixing ratio [1, 2]. Problems in theses process can result in formation of porosities, which may affect the texture, surface roughness and shade in ceramic structure [3].

A current technology, CAD/CAM, has led to fast procedures and a high quality of indirect restorations, besides avoiding problems in the material due to the process with high pressure and temperature to produce it in blocks [4]. Composite materials can cause lower wear to natural antagonist tooth than ceramic materials [4]. However, problems with color stability and wear of composites restorations can limit their use. Some CAD/CAM materials have been developed to balance advantages and disadvantages among ceramic and composite materials. These materials are CAD/CAM blocks of composite and of polymer infiltrated ceramic, and they show high flexural strength, low brittleness, and easy milling [5].

Restorations, regardless the composition, should have smooth external surface for esthetic quality as well as periodontal health. Rough surfaces facilitate plaque formation and retention, which can result in gingivitis, periodontitis, and also caries diseases [6]. Other consequences about the roughness are the presence of flaws that can compromise the flexural strength of the restorative material, the increase in the wear against the antagonist tooth surface, and the decrease in the longevity of both, the restored tooth and the antagonist [7].

Adjustments are necessary in laboratory procedures and even, sometimes, intraorally in dental clinics. This must be followed by polishing or re-glazing to avoid the decrease in longevity of the restoration and the wear against the antagonist. If an appropriate polishing procedure is not performed, there is also a possibility of happening micro-cracks, which may let to future catastrophic fractures [6].

Some materials do not need a two-step work process to reach the needed strength, which includes a design and machining as the first step and another step for additional heat treatment [8]. The materials that can be one-step CAD/CAM processed are manually polished, performing this in the same clinical session of the restorative treatment. There are several polishing systems and protocols recommended for polishing ceramic and composite restorations. Nevertheless, it is not clear if all systems have the same performance for every material and which polishing system and protocol should be used.

Polishing rubbers, diamond discs, polishing brushes, and the combination with diamond pastes are the most popular procedures used clinically [9].

Acidic and staining beverages are consumed daily, exposuring teeth and restorative materials to this environment. Acid environments can degrade surface of some restorative materials, affecting their surface roughness [10].

Therefore, the present study investigated the effects of immersion solutions and polishing techniques on the surface roughness of different CADM/CAM materials and a composite resin at different time-points. The null hypothesis is that the immersion solutions and the polishing techniques would not affect the roughness of each material in different time of evaluation.

MATERIAL AND METHODS

The restorative materials and their composition are shown on Table 1. The materials used were blocks used were Composite Resin Filtek Z350 XT – M1 (A2 - Enamel), Lava Ultimate – M2 (color A2-HT), Enamic – M3 (color 2 M2-HT), Celtra Duo – M4 (color A2-LT).

Polishing systems:

-Polishing system 1 (P1): Ceramisté 3-step polishing rubbers - Standard, Ultra and Ultra II (Shofu Dental GmbH, Ratingen, Germany)

-Polishing system 2 (P2): Ceramisté 3-step polishing rubbers - Standard, Ultra and Ultra II (Shofu Dental GmbH, Ratingen, Germany) + Porcelize paste (Cosmedent, Inc., Chicago, USA) + discs of felt with extra fine granulation Flexibuff (Cosmedent, Inc., Chicago, USA)

Immersion Solutions

S1: Artificial saliva (control group)

S2: Nespresso® Arpeggio Coffee

S3: Coca-Cola®

Experimental design

It is an experimental study presenting as dependent variable the surface roughness, and as independent variables the restorative materials, polishing systems, immersion solutions and different times of evaluation.

The number of specimens used was 10 specimens per group, according to the studies on scientific literature about surface roughness evaluation.

Specimens preparation

The ceramic specimens were obtained from CAD/CAM blocks, which were cut using a water-cooled low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain rectangular shape specimens (6 x 6 x 1.5mm). The composite resin specimens Filtek Z350XT (3M) were made in a matrix made of Zetalabor (Zhermark) condensation silicone in dimensions 6 x 6 x 1.5mm. The resin was inserted by single increment in the silicon matrix, placing a polyester strip and a weight of 1kg on it for surface standardization.

Subsequently, the specimens were included in a circular matrix with a dimension of 10mm in diameter and 1.5mm in height filled with polyester resin, leaving the ceramic surface exposed. After 24 hours of inclusion in the matrix with polyester resin, the specimens were stantardized in a polishing machine (DP-10 Panambra, Struers, Ballerup, Denmark) under refrigeration with with silicon carbide abrasive papers (600 and 800 grit). The final thickness (1.5mm) was measured using a digital caliper.

Polishing Procedures

The specimens were washed in water, dried with paper towel and followed by two polishing procedures:

- Ceramisté (Standard, Ultra and Ultra II): the polishing rubbers were used for 20 seconds in both directions of movement, at low speed.

- Ceramisté (Standard, Ultra and Ultra II) + Porcelize 1μm + Flexibuff felt discs: the polishing rubbers were used for 20 seconds in both directions of movement, at low speed. Then the 1m granulation paste was applied with the felt disk for more 20 seconds.

After the polishing procedures, the specimens were ultrasonically washed in distilled water for 2 minutes, and then stored in artificial saliva for 24 hours in an oven at 37 ° C ± 1 ° C (EBC1-Odontobras- Trade Eq, Medical-Dental LTDA, Ribeirão Preto, SP, Brazil) for rehydration prior to start of storage in pigment solutions.

Immersion procedures

All specimens were removed from the storage in artificial saliva, ultrasonically washed with distilled water and dried. Then, they were immersed in solutions of new artificial saliva, Nespresso® Arpeggio and Coca-Cola®, stored in an incubator at 37°C ± 1°C for 60 days, renewing the solutions every 5 days to avoid contamination.

Measurement of surface roughness

Three readings of surface roughness were performed using a digital profilometer (Mytutoyo Corporation, Tokyo, Japan; Model: SJ 400), and the means were determined as the Ra (μm) value. The reading accuracy of the profilometer was 0.01μm, reading length 2.4mm, active tip velocity of 0.5mm / s and radius of the active tip of 5μm.

The roughness readings were performed before the first immersion in the solutions (baseline - T0), after 30 days (T1) and after 60 days of immersion (T2).

Evaluation of surface topography

Samples were randomly selected to examine the surface topography by scanning electron microscopy (SEM) at the initial time (before starting the immersion procedures - T0) and after the immersion period (60 days - T2). Prior to the reading, the selected specimens were dried with 60 seconds air jets, performed the impression of surface with addition silicone and replicated with epoxy resin. Then these same specimens were placed in a desiccator with silica gel for 24 hours before visualization in SEM. The surface roughness evaluation was examined under magnification of 500x using a scanning electron microscope (JEOL 6060; Ltda, Tokyo, Japan), operating at 20kV.

Statistical analysis

The normality of data distribution and sphericity were verified by Shapiro-Wilk ($p=0.052-0.282$) and Mauchly ($p<0.001$) tests, respectively. The Greenhouse-Geisser correction factor was used, as the assumption of sphericity was not attended. Data was analyzed using the software PASW Statistics 25.0 (SPSS Inc, Chicago, IL, USA), with a confidence interval set at 95%. A repeated measure analysis of variance (ANOVA) and Tukey tests were used to evaluate the effect of immersion solution (S1, S2 and S3), polishing technique (P1 and P2) and assessment time (T0, T1 and T2) on the independent factor (roughness) for each material tested (M1, M2, M3 and M4).

RESULTS

The mean and standard deviation of roughness of the specimens, according to material, immersion solution, polishing technique and assessment time, are shown in Table 2. The SEM images of surface roughness of each material in immersion in the control solution (S1) and the solution which showed the highest values (S2) are shown from Figure 5 to Figure 8, in the times T0 and T2.

Material 1

Significant difference was verified for the factor “time” ($p<0.001$) and the interactions “time and solution” ($p=0.030$) and “time and polishing” ($p=0.030$); however, no significant difference was identified for the interaction “time, solution and polishing” ($p=0.128$). Significant difference was also observed for the factor “solution” ($p=0.002$), but not for “polishing” ($p=0.848$) or the interaction “solution and polishing” ($p=0.492$). Specimens immersed in solution 2 showed the highest roughness values at the assessment time of 60 days, independent of the polishing technique. The roughness values were similar for all the other conditions, without any effect of the type of treatment. The mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time is shown in Figure 1.

Material 2

The repeated measures ANOVA identified significant difference for the factor “time” ($p=0.003$) and the interactions “time and solution” ($p=0.014$) and “time, solution and polishing” ($p=0.013$). No significant difference was verified for the interaction “time and polishing” ($p=0.16$). Significant difference was observed for the factor “solution” ($p=0.001$); however, no difference was found for “polishing” ($p=0.209$) or the interaction “solution and polishing” ($p=0.962$). All solution presented similar roughness values among the different assessment time, despite the polishing technique. The highest roughness values were obtained for solution 2 associated to polishing technique 2 at the assessment times of 30 and 60 days. Contrariwise, solution 1 associated to polishing technique 2 at the assessment time of 30 days showed the lowest value which was statistically different than solution 2 associated to polishing technique 1 at the same assessment time. The mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time is shown in Figure 2.

Material 3

Significant difference was found for the factor “time” ($p<0.001$) and the interactions “time and solution” ($p<0.001$). There was no significant difference for the interaction “time and polishing” ($p=0.675$) and “time, solution and polishing” ($p=0.425$). The ANOVA also verified significant difference for the factor “solution” ($p<0.001$); however, it did not identified difference for “polishing” ($p=0.207$) or the interaction “solution and polishing” ($p=0.879$). Regardless of the polishing technique, specimens immersed in solution 2 showed the highest roughness values at the assessment time of 60 days. The values for these two groups were statistically different than all the other conditions, which had similar roughness values, without any effect of the type of treatment. The mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time is shown in Figure 3.

Material 4

Significant difference was verified for the factor “time” ($p<0.001$) and the interactions “time and solution” ($p=0.001$) and “time, solution and polishing” ($p=0.003$); however, repeated measures ANOVA did not identify difference for the interaction “time and polishing” ($p=0.969$). No significant difference was also observed for the factor “solution” ($p=0.050$) and “polishing” ($p=0.895$); however, there was a significant difference for the interaction “solution and polishing” ($p=0.021$). Specimens immersed in solution 1 had the highest increase in roughness values after 30 and 60 days when associated to polishing technique 1, showing statistical significance when compared to baseline. Contrarily, polishing technique decrease the roughness values for specimens immersed in solutions 2 and 3. There was a statistical significance between 30 and 60 days for solution 2 associated to polishing technique 1. Although no significant difference was found for solution 3, the values decreased after 60 days. The roughness values for specimens immersed in solution 3 associated to polishing technique 2 were lower than the ones immersed in solution 2 associated to the same polishing technique at the assessment time of 30 days. Immersion in solution 1 associated to polishing technique 1 showed significantly higher roughness at the assessment time of 60 days when compared to solutions 2 and 3 using the same polishing technique. The mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time is shown in Figure 4.

DISCUSSION

Overall, the polishing techniques and the time of immersion did not affect significantly the surface roughness of each restorative materials evaluated, but the immersion solution showed to affect the roughness in S2. Therefore, the null hypothesis of this study was partially accepted.

Although is known by some studies that resin-based materials are more susceptible to degradation and change in surface roughness [7, 11], M1 showed significant difference only for S2 in the last time-point, after 60 days. Resin-based materials show water diffusion through the polymer chains and as consequence, a hydrolytic deterioration of these chains [11]. This way, after aging in solutions, this material can show a structure deterioration, which can justify the only significant difference have been occurred after 60 days in coffee. Coffee was the solution with the highest surface roughness values, showing significant difference for composite resin and hybrid ceramic after 60 days.

Resin nanoceramic has small fillers in high filler content and some ZrO_2 fillers, which can collaborate to lower maximum surface roughness [12]. This can justify the results with no significant difference for this material (M2), regardless the immersion media, polishing system and time-points evaluated.

Although there was not difference between the polishing techniques evaluated, it is a need procedure. Duarte et al., [13] showed that unpolished specimens of Enamic caused severe enamel wear due to the exposition of feldspathic ceramic network, suggesting an appropriate polishing to be an essential procedure. It is still difficult to determinate the best polishing system to restorative materials, considering several variables that can affect the result, as the restorative material composition, polishing system, and pressure used during the polishing procedure. Some studies have shown more effective results for polishing ceramic materials than glazing [14, 15]. The use of diamond pastes for this procedure is a common practice in dental office. The present study did not show significant difference between to use or not the diamond paste. But, Camacho et al.,[16] claim that theses polishing pastes provide an efficient surface polishing.

Celtra Duo (M4) is a ceramic that provides two processing pathways: can be cemented after milling and polishing, which is a faster procedure, or after milling, glaze and firing, which is a slower procedure but offers an additional strength [17]. This present study showed a significant increase in surface roughness for this material after aging on artificial saliva. It suggests that, due to have not used the fire step, it can have affected its surface roughness, which could show better behavior after an additional step. Martzinger et al.,[12] showed a different morphology in SEM images between Celtra Duo with only polishing step and Vita Suprinity crystallized, which ones show similar composition. The images showed some defects on surface and grooves for Celtra Duo after prepolishing procedure, which can occur due to the removing of soluble crystals in its composition during milling or finishing procedures. During grinding process, ceramic materials have 50% of possibility for strength reduction, while resin-based materials are less susceptible to these effects [18]. These failures that may occur and affect the material structure show by these studies can show a hypothesis in the present study for M4, that showed an increase on surface roughness even in the control group (S1).

Further studies are need to investigate better polishing protocols that could be appropriate for each material, which can avoid more efficiently the affect mainly of substances as coffee.

CONCLUSIONS:

Overall, surface roughness was not affected by polishing technique and time-points for each restorative material evaluated. The coffee solution showed the highest roughness values, showing significant difference for composite resin and hybrid ceramic after 60 days of immersion. The use of diamond paste associated to polishing rubbers did not affect the roughness.

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Table 1- Materials and composition

Material; Manufacturer	Classification	Composition
Filtek Z350 XT; 3M ESPE Dental Products, St. Paul, MN	Nanofilled composite	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA, sílica filler, zirconia/sílica cluster filler
Lava Ultimate; 3M ESPE Dental Products, St. Paul, MN	Resin nanoceramic CAD/CAM block	Bis-GMA, UDMA, Bis-EMA, TEGDMA with 80%wt, 20-nm silica and 4- to 11-nm zirconia nanoparticles, and zirconia/silica nanoclusters
Enamic; Vita Zahnfabrik, Germany	Hybrid ceramic CAD/CAM block	UDMA, TEGDMA, 86wt% Feldspar ceramic enriched with aluminum oxide
Celtra Duo; Dentsply Sirona	Zirconia-reinforced lithium silicate ceramic block CAD/CAM	Lithium silicate with 10% ZrO_2
Ceramisté; Shofu Dental GmbH, Ratin- gen, Germany	Silicon Rubber	Silicon carbide impregnated polishers
Porcelize Paste; Cosmedent, Inc., Chicago, USA	Diamond paste	Diamond particles 1μm

Table 2- Mean ± standard deviation of roughness (Ra) for each material, according to solution/polishing and assessment time.

		T0		T1		T2	
		P1	P2	P1	P2	P1	P2
M1							
	S1	0.13 ± 0.02	0.14 ± 0.04	0.12 ± 0.03	0.15 ± 0.04	0.15 ± 0.02	0.16 ± 0.04
	S2	0.13 ± 0.05	0.14 ± 0.05	0.12 ± 0.03	0.15 ± 0.07	0.25 ± 0.13	0.17 ± 0.09
	S3	0.11 ± 0.02	0.13 ± 0.04	0.10 ± 0.03	0.10 ± 0.02	0.14 ± 0.04	0.13 ± 0.03
M2	S1	0.10 ± 0.02	0.14 ± 0.02	0.11 ± 0.05	0.09 ± 0.01	0.13 ± 0.02	0.13 ± 0.02
	S2	0.12 ± 0.02	0.11 ± 0.02	0.12 ± 0.04	0.13 ± 0.06	0.14 ± 0.03	0.15 ± 0.04
	S3	0.11 ± 0.02	0.12 ± 0.03	0.09 ± 0.02	0.10 ± 0.02	0.10 ± 0.02	0.11 ± 0.03
M3	S1	0.16 ± 0.01	0.16 ± 0.02	0.16 ± 0.02	0.16 ± 0.02	0.17 ± 0.02	0.16 ± 0.02
	S2	0.16 ± 0.02	0.16 ± 0.02	0.18 ± 0.04	0.17 ± 0.03	0.27 ± 0.07	0.24 ± 0.09
	S3	0.17 ± 0.03	0.15 ± 0.02	0.17 ± 0.04	0.16 ± 0.03	0.17 ± 0.02	0.17 ± 0.03
M4	S1	0.14 ± 0.05	0.17 ± 0.03	0.23 ± 0.07	0.21 ± 0.04	0.25 ± 0.07	0.18 ± 0.02
	S2	0.18 ± 0.05	0.22 ± 0.04	0.19 ± 0.13	0.24 ± 0.08	0.14 ± 0.04	0.19 ± 0.02
	S3	0.17 ± 0.04	0.14 ± 0.02	0.20 ± 0.05	0.17 ± 0.03	0.16 ± 0.02	0.17 ± 0.05

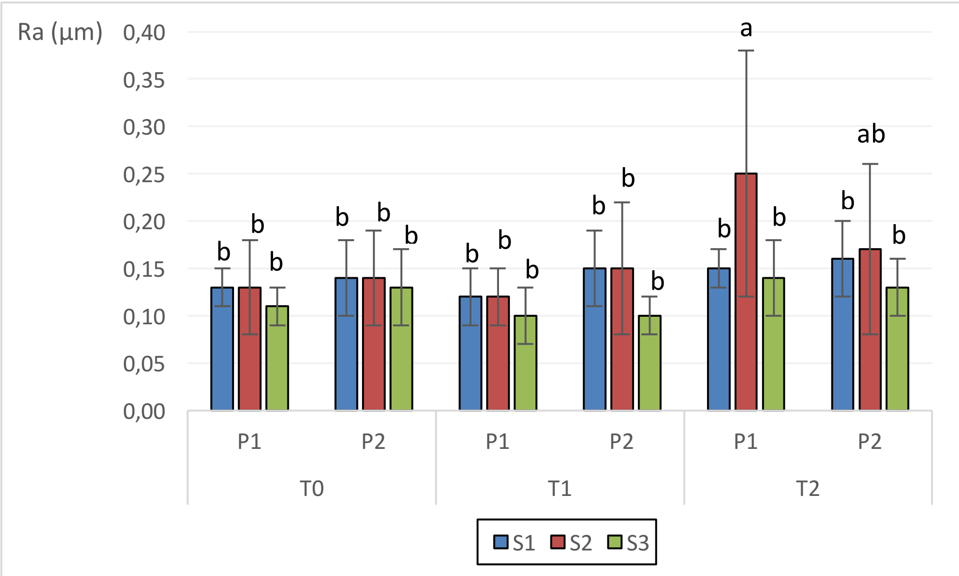

Figure 1 – Mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time for Material 1. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

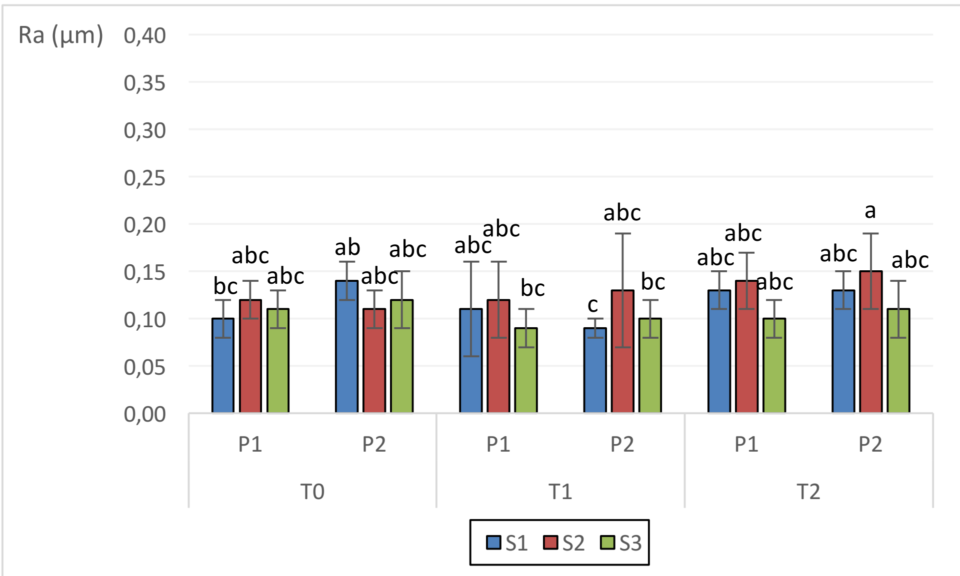

Figure 2 – Mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time for Material 2. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

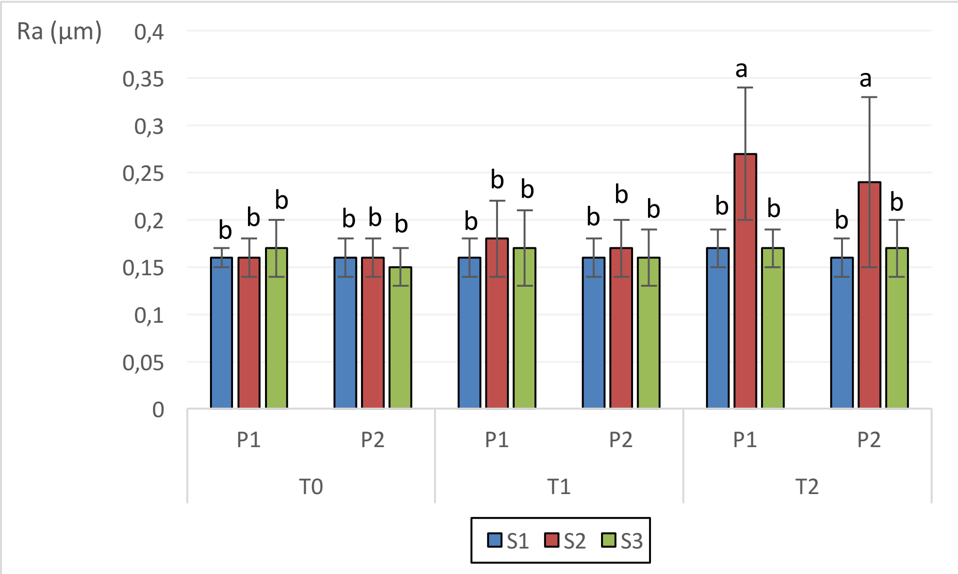

Figure 3 – Mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time for Material 3. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

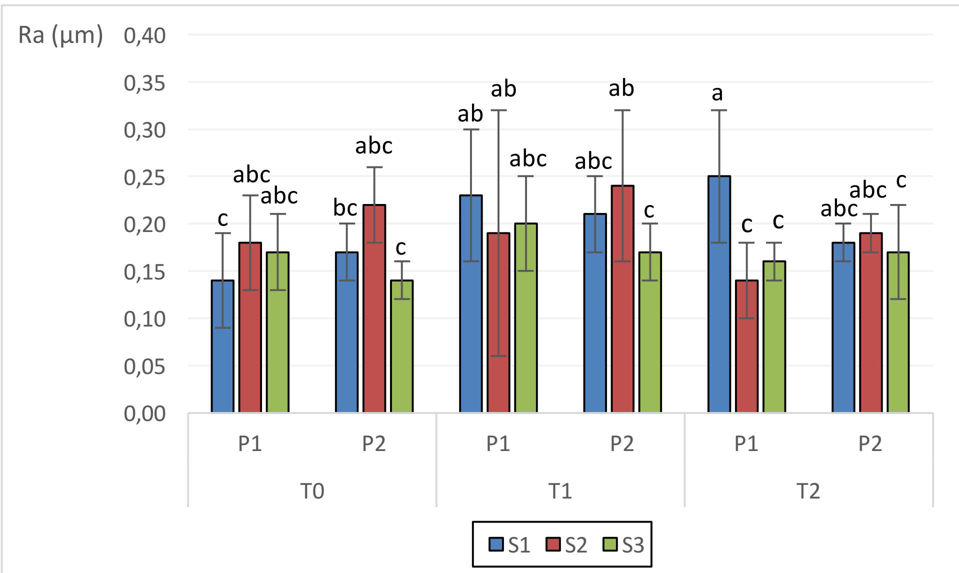

Figure 4 – Mean and standard deviation of roughness (Ra) according to solution/polishing and assessment time for Material 4. Identical lower-case letters indicate no significant difference among the groups (α=0.05).

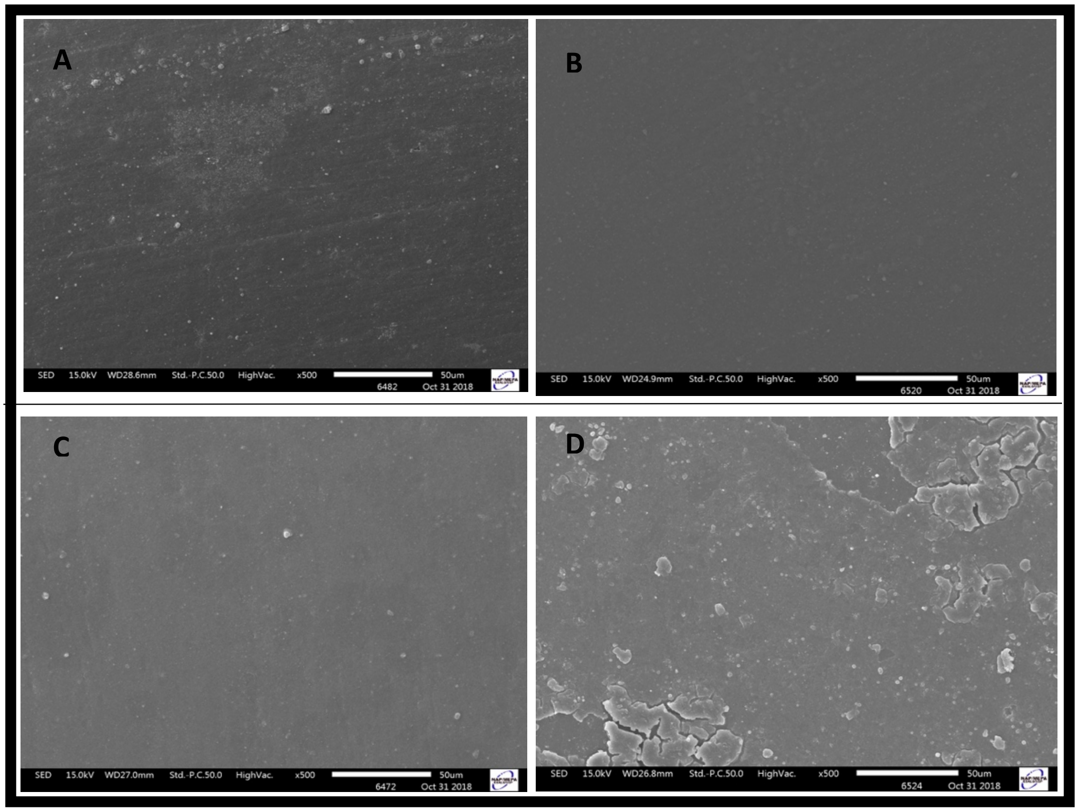

Figure 5 - Composite Resin – M1 (scanning electron pictures x 500). A) Solution 1 in T0; B) Solution 1 in T2; C) Solution 2 in T0; D) Solution 2 in T2.

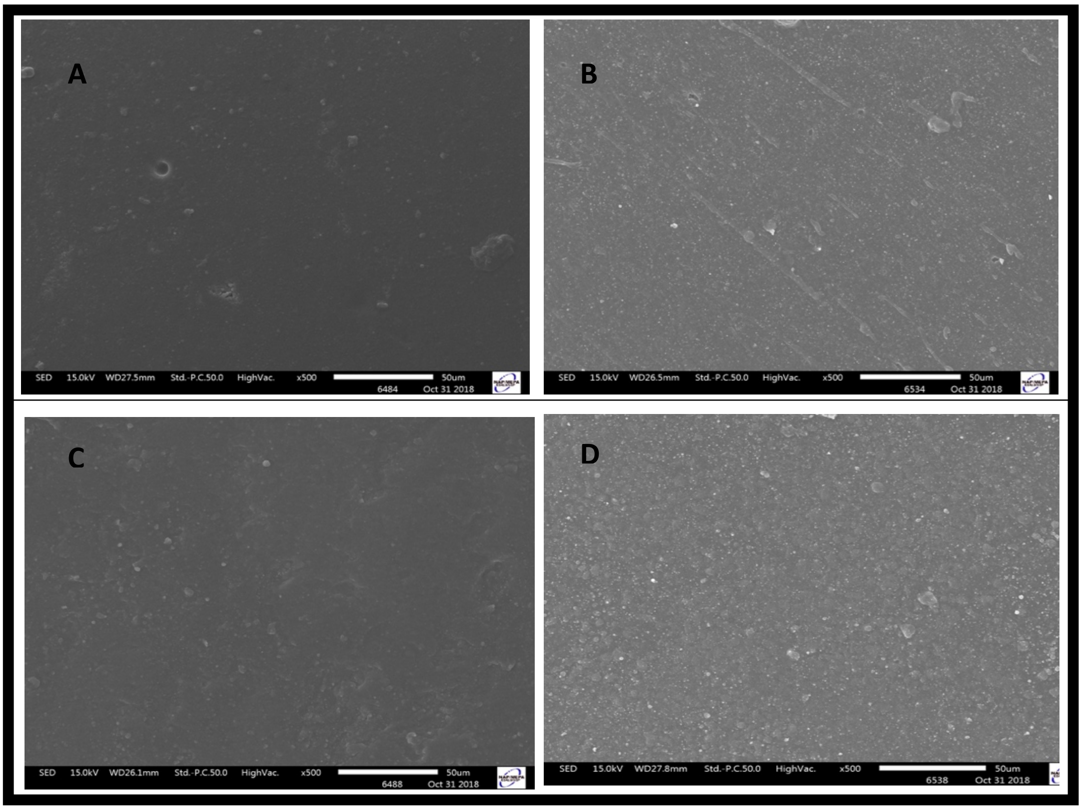

Figure 6 - Lava Ultimate – M2 (scanning electron pictures x 500). A) Solution 1 in T0; B) Solution 1 in T2; C) Solution 2 in T0; D) Solution 2 in T2.

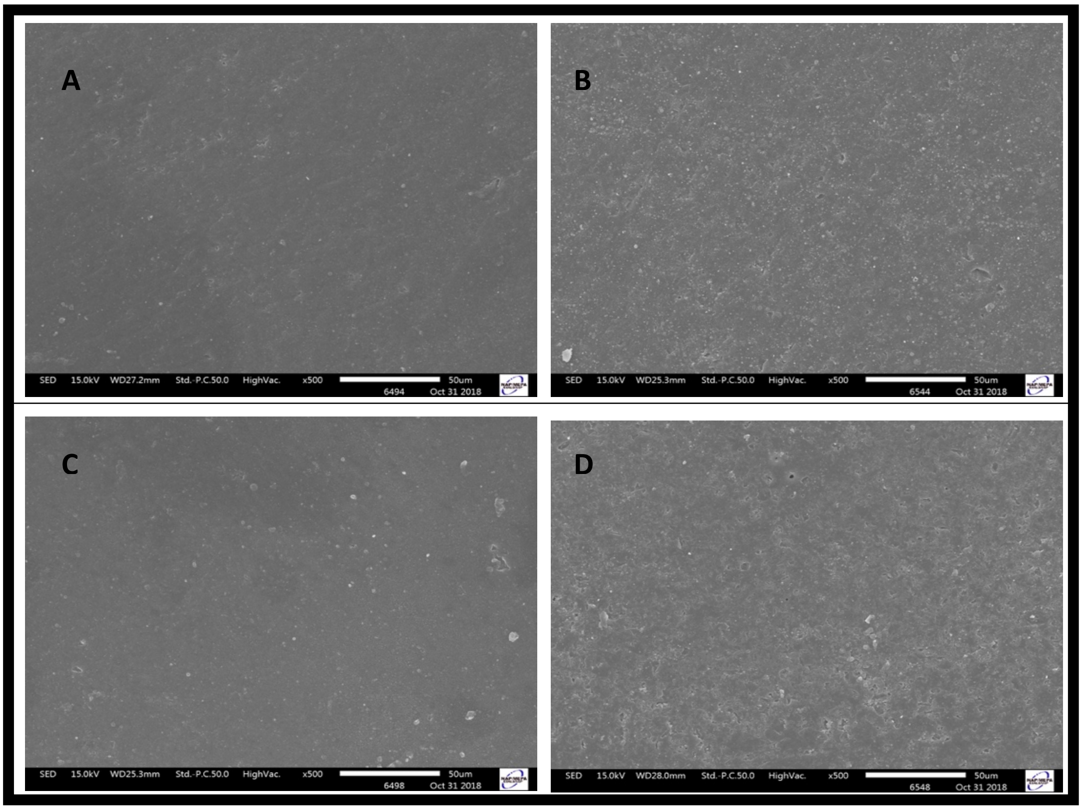

Figure 7 - Enamic – M3 (scanning electron pictures x 500). A) Solution 1 in T0; B) Solution 1 in T2; C) Solution 2 in T0; D) Solution 2 in T2.

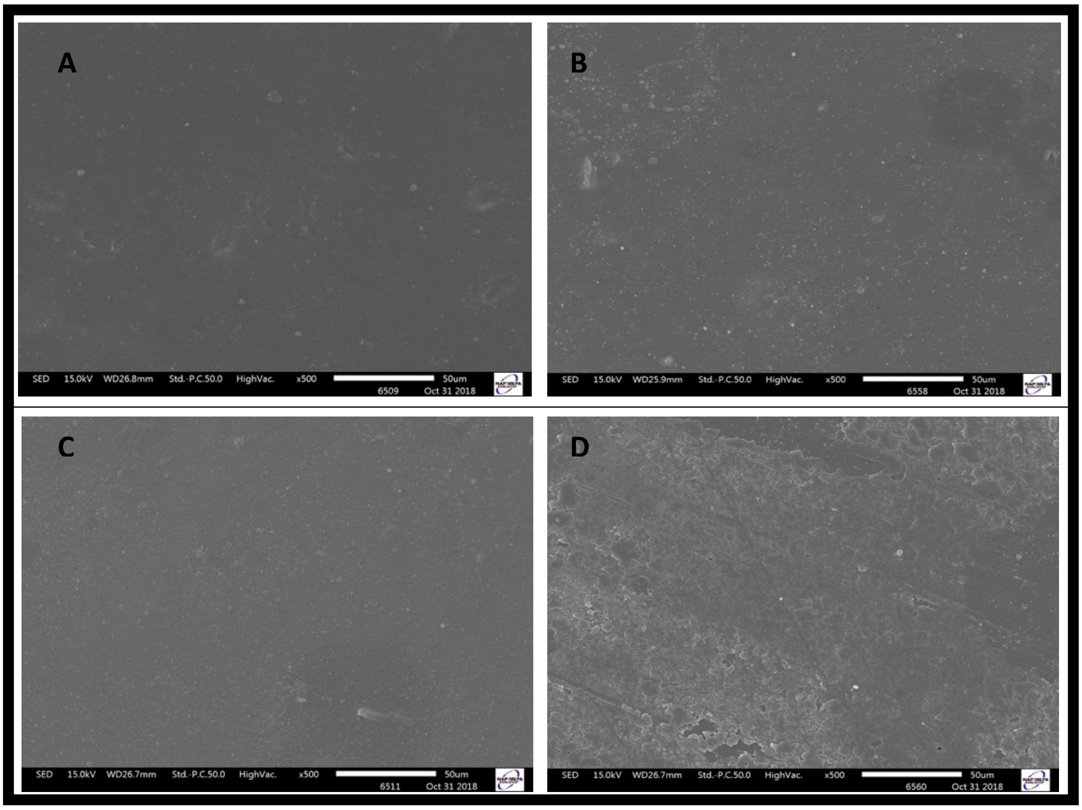

Figure 8 - Celtra Duo – M4 (scanning electron pictures x 500). A) Solution 1 in T0; B) Solution 1 in T2; C) Solution 2 in T0; D) Solution 2 in T2.

3.3 Publicação 3*

Analysis of the bond strength of different repair techniques between indirect restorative materials and composite resin

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Analysis of the bond strength of different repair techniques between indirect restorative materials and composite resin

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ABSTRACT

Background: Computer-aided-design/computer-aided-machining (CAD/CAM) technology has been used for manufacturing high-quality restorations in a short time. Materials with different compositions may be used and, upon failure, need to be repaired.

Objective: To evaluate the effect of bonding strategies on the bond strength between composite resin as a repair material and different CAD/CAM restorative materials.

Methods: Zirconia-reinforced lithium-silicate – ZL (Celtra Duo; Dentsply Sirona), Feldspathic-composite Hybrid Ceramic – HC (Enamic; Vita), Resin Nanoceramic – NC (Lava Ultimate Restorative; 3M ESPE) and Composite Resin - CR (nanofilled composite) were used to produce blocks that received one of the following treatments: hydrofluoric acid (HF) + ceramic primer (CP) + total-etch adhesive (TE); HF + self-etching adhesive (SE); Tribochemical Silica Coating (SC) + CP + TE; SC + SE. Microshear bond strength cylinders were built with composite resin on the treated surfaces, and were tested after 24 hours stored in water. Data was analyzed using two-way analysis of variance (Two-Way ANOVA) and Tukey test (5%).

Results: Restorative material ($p<.001$), bonding strategy ($p=.014$) and the interaction ($p<.001$) were significant. Overall, silica coating resulted in lower bond strength to ZL and HC and higher bond strength to NC and CR. Silane on adhesive bottle and silane on separated bottle did not show significant difference.

Conclusion: Zirconia-reinforced lithium-silicate and Feldspathic-composite hybrid ceramic do not benefit from surface silicatization, decreasing their perfomance. Surface silica impregnation improves bonding to NC and CR. The use of silane either on adhesive bottle or as separated step does not affect the bond strength.

Keywords: Ceramic; Bond Strength; CAD/CAM materials; Silica Coating, Composite Resin

INTRODUCTION

CAD/CAM technology enables a faster restorative treatment by the dentist, obtaining milled restorations with high precision in marginal fit, occlusal/interproximal contacts and anatomic,[1]mainly when the image of dental preparation is obtained with direct digitalization (from patient's mouth).[2]

Some CAD/CAM materials have been developed with different properties, among them, ceramic and resin blocks to create crowns, inlays, onlays, overlays, veneers. Blocks of composite resin and of ceramic containing polymeric matrix ally the advantages of composite materials. The low abrasiveness to antagonistic teeth, that preserves enamel structure,[3] the easier possibility to repair ,[4] and the low elastic modulus of composite resin restorations, which allow more absorption of functional stress.[5] it make this material a good restorative option.

Novel CAD/CAM ceramics have been introduced as Celtra Duo (Dentsply Sirona), that is zirconia-reinforced lithium-silicate. Celtra Duo is a lithium silicate ceramic with 10% of zirconia in the glass phase and it is fully crystallized, offering the option to cement after milling to be a faster procedure or use a furnace to increase its strength from 210MPa to 370MPa, which is considerably greater than the as-milled material.[6] As examples of materials containing polymeric matrix there are Enamic (Vita) and Lava Ultimate (3M ESPE). Enamic is a porous feldspathic ceramic matrix (86 wt% of glass phase according to the manufacturer) that is infiltrated with a methacrylate-based polymer by capillary action .[7] This material's fabrication process demands two steps: the first one is to produce a porous pre-sintered ceramic network and to condition it by a coupling agent; the second one is to infiltrate this network with a polymer.[7] Lava Ultimate is a polymeric matrix reinforced by ceramic nanofillers agglomerated into clusters, among them silica of 20 nm diameter, and zirconia of 4 to 11 nm diameter, that express approximately 80 wt% of nanoceramic and 20 wt% resin.[8] The polymerization process is under high temperature and high pressure, making it a highly compact internal structure.[9]

However, under different clinical conditions, they can fail. A conservative restorative approach suggests the repair of restorations, in some cases of failures, instead of replacing them to preserves dental structure, to be a faster procedure and less costly.[10] Several studies have been reported different protocols for this procedure,[11,12] including chemical, mechanical or physico-mechanical methods to enhance the bond area. Among them, there are hydrofluoric acid, airborne particle abrasion with aluminum oxide,[13] tribochemical silica coating, application of a silane, use of a dental adhesive, or the combination of some of them.[12] Using techniques with composite resin as the repair material is an esthetic and functional alternative to make intraoral repair.[11]

The current literature about bonding strategies to repair some CAD/CAM materials is limited. Considering the properties and composition of the previously mentioned materials, this study investigated if the materials with polymer components show more similarity in a repair procedure to a full ceramic material (Celtra Duo) or to a composite resin. Then, the aim of this study was to evaluate the effect of different repair protocols on different CAD/CAM materials to resin composite on the micro shear bond strength.

The null hypotheses are that the bonding strategy and the restorative material will not influence the bond strength after the repair.

MATERIALS AND METHODS

All the materials and their composition used in this present study are listed in Table 1. The blocks used were Celtra Duo (color A2-LT), Enamic (color 2 M2-HT), Lava Ultimate (color A2-HT), Composite Resin Filtek Z350 XT (color A2).

Experimental design

This experimental study has the bond strength as the dependent variable and restorative materials and adhesion systems as the independent ones.

Each group was composed of 10 specimens, according to the existing literature, with a total of 16 groups, which totalizes a N of 160 specimens.

Specimens preparation

The ceramic specimens were obtained from CAD/CAM blocks. They were cut using a water-cooled low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) to obtain rectangular shape specimens (13 x 6 x 1.5mm).

The composite resin specimens were prepared using a Filtek Z350XT resin (3M) by inserting the material into a matrix made of condensation silicone (Zetalabor, Zhermark) in the same dimension (13 x 6 x 1.5mm). The resin was inserted in a single increment into the silicone matrix; then, a polyester strip and a 1kg weight were placed on top of it for surface standardization. Each specimen was light cured for 20 seconds using a LED light (VALO, Ultradent Products Inc, USA).

Subsequently, the specimens were embedded in a 2.5cm diameter PVC cylinder filled with polyester resin (Maxi Rubber, Diadema, SP, Brazil), leaving the surface of the restorative material exposed. After 24 hours, the surface on both sides of the specimens were standardized using a polishing machine (DP-10 Panambra, Struers, Ballerup, Denmark) with 600 and 1000 grit silicon carbide papers for 20 seconds under refrigeration. After polishing and prior to adhesive procedure, all specimens were ultrasonically washed in distilled water for 5 minutes.

Adhesion systems and Surface treatments

The four groups of different restorative materials (ZL; HC; NC; CR) were subdivided into 4 subgroups: 1- 5% Hydrofluoric acid + Clear Fil ceramic primer + Scotchbond Multi Purpose Adhesive; 2- Scotch Bond Universal; 3- Cojet + Clear Fil ceramic primer + Scotchbond Multi Purpose Adhesive; 4- Cojet + Universal Scotch Bond.

1- Except for the Lava Ultimate group, which received no acid treatment due to manufacturer's recommendations (only ethanol cleaning and air drying), and the Composite Resin group treated with 37% phosphoric acid for 30s, the other two materials (Enamic and Celtra Duo) were treated with 5% hydrofluoric acid for 30s and 60s as recommended for Celtra Duo and Enamic, respectively and air dried with jets of air. After, the silane agent was applied for all groups (Clear Fil ceramic primer) with microbrush for 60s + jet of air for 10s + application of Scotchbond Multi Purpose adhesive for 20s + jets of air for 5s + light curing for 10s using VALO.

2- All groups received the same initial acid procedures described in the previous item + application of ScotchBond Universal adhesive with microbrush for 20 s + jet of air for 5s + light curing for 10s.

3- All groups were blasted with the Cojet system at a distance of 10mm for 15s and pressure of 2.8bar + jet of air for 10s + silane application (Clear Fil ceramic primer) with microbrush for 60s + jet of air for 10s + application of Scotchbond Multi Purpose adhesive for 20s + jets of air for 5s + light curing for 10s.

4- All groups received blasting with Cojet system following the same protocol previously described + jet of air for 10s + application of ScotchBond Universal adhesive using a microbrush for 20s + jet of air for 5s + light curing for 20s.

Delimitation of adhesive area and adhesive procedure

An acid resistant double-sided tape was cut to the same dimension than the specimens and linearly perforated with four holes using a rubber dam punch, in order to standardize the adhesive area.[14,15] One side of the tape was adhered to the specimen, delimiting the four areas where the adhesive systems were applied and, the other side was used to position the cylinders for insertion of composite resin. The cylinders were obtained by cutting pasta noodles with internal diameter of 1.15mm and 1.5mm of height. The cylinders were filled with composite resin (Filtek Z350 XT – 3M/ESPE, color A2E), light cured for 20 seconds using VALO (Ultradent Products, USA). Specimens were immersed in deionized water for 24h prior to the micro-shear bond strength test. After 24 hours, the softened noodles cylinders were easily removed with the adhesive tape. Specimens were visually inspection for possible defects and a marked near to each cylinder with a hydrographic pen to facilitate their identification during the mechanical test.

Micro-shear bond strength

The specimens were adapted to a jig for micro-shear bond strength test coupled to the mechanical test machine (Instron 4301). The shear loading was carefully aligned as close as possible to the bonding interface and the stress was applied through this interface using a 0.2 mm diameter orthodontic wire. The crosshead speed of the mechanical test machine was set at 0.5mm/min and specimens were tested until failure. The micro-shear bond strength was calculated by dividing the maximum force recorded (N) by the bond area (mm^2) and expressed in MPa.

Analysis of adhesive interface

Specimens were examined after fracture under 40x magnification with a stereomicroscope to determine the fracture pattern. The pattern was classified as: adhesive - between ceramic/composite resin and restorative material (A), cohesive in ceramic/composite resin (CC), cohesive in the repair material (CR), or mixed (M).

Statistical analysis

Data for each material was analyzed by a Two-Way ANOVA and Tukey tests to determine the best repair technique for each one. The level of significance was set at 5%.

RESULTS

Significant difference was verified among the restorative materials ($p<.001$), the repair protocols ($p=.014$) and the interaction of both ($p<.001$).

The mean and standard deviation for bond strength (MPa) of all bonding protocols for each restorative material, are shown in Figure 1. For all materials, the use of silane as a separate step did not show significant difference to silane incorporated into adhesive bottle. The use of silica blasting decreased significantly the bond strength means for the materials ZL and HC, and increased for the NC and CR.

The most common failure present in the study was the adhesive for all groups, as shown in Figure 2. NC and CR showed 100% of adhesive failure, decreasing this percentage after the application of silica blasting. The opposite occurred for ZL and HC, that although predominated adhesive failures, it was in lower percentage that for NC and CR, besides to increase the percentage after silica blasting treatment.

DISCUSSION

The null hypotheses are rejected, considering there were significant difference in bond strength values by micro-shear test (μSBS) of the different materials and the different bonding strategies to repair with resin composite.

Analysis in vitro for bonding materials, considering small adhesive areas to be more truthful, can be done either microshear or microtensile. Although there is study that suggests the microtensile test for evaluating the adhesion between ceramic and composite resin due to advantages as the stress concentration on adhesive interface,[16] this test requires special care with fragile specimens and there is some risk of causing micro-cracks at the interface during the preparation of the specimens.[17] Microshear, a test widely used for bonding evaluation, is a simpler process than of microtensile test and shows relatively small standard deviations in precise results.[18,19] For reliable results on microshear, it is important that the test distributes the forces on the adhesive interface, which is possible using the wire loop due to its more uniform distribution of shear bond stress than of the chisel.[20]

Some bonding techniques have been proposed to repair restorative materials. The use of hydrofluoric acid is a treatment suggested to repair composite restorations due to the surface roughness promoted by its action of dissolving filler particles.[21] However, the hydrofluoric acid has shown the lowest mean bond strength result in the disintegration of the nanoclusters particles contained on the resin nanoceramic material and composite resin, which may not increase roughness needed to improve bonding strengths,[22] even can weaken the material and accelerates the process of hydrolytic degradation.[21] The use of hydrofluoric acid should be avoided for repair procedures in resin composite restorations,[22] which shows the reason why this acid treatment has not been used for the groups NC and CR.

The efficacy of micromechanical retention using diamond burs and sandblasting in composite materials repair has already been reported by previous studies.[22,23] This present study showed significant improvement in bond strength means for NC and CR when it was treated with tribochemical silica coating, independent of the adhesive system used. Some studies have found similar results.[12,22,24] Rodrigues et al., 2009,[22] that evaluated different surface treatments on bond strength of repaired composite resin, and showed, independent of the primer used, the greatest values for sandblasting treatments with aluminum oxide and silica coating, with no significant increase between these both treatments.

Silica coating affects in an additional retention mechanism,[25] and increase the adhesion area by the deposition of particles of silica and by the energy produced during the impact on the material surface.[26] Even though the improvement of this treatment was observed for NC and CR materials, the opposite was shown for ZL and HC, which ones showed higher bond strength for hydrofluoric acid than for silica coating, as follow Barutcigil et al., 2019.[27] This can suggest due to the glass phase on the ZL and HC materials are more affected by the dissolution the glass matrix for the hydrofluoric acid treatment, providing microporosity on the ceramic surface, which enhance micromechanical retention with the repair material, composite resin.[28] Furthermore, sandblasting treatment may damage ceramic surfaces and affect in wide volume loss. Thus, silica coating of ceramic with feldspar glass composition should be avoided.[29]

Silanization procedure improves chemical bonding with the methacrylate,[30] showing the chemical capacity to bond with filler particles of composite materials.[31] Also, a silane improves the wettability of composite surface for adhesive systems.[21] The application of a dental adhesive for repairing procedure is required to improve the bond strength, suggesting that a micromechanical retention alone is not enough to result in appropriate bond strengths.[12] Althought Yoshihara et al., 2017 [32] showed lower effectiveness of the silane incorporated in universal adhesive system than the silane used as a separate step, some studies showed that the use of silane as a separate step after sandblasting (aluminum oxide or silica coating) didn't increase the bond strength means.[12,33] Moreover, the application of silane and adhesive as separate steps can result in multiphase layer, increasing the risk for flaws in each step.[34]

This present study showed the highest bond strength values for NC and CR when they were treated with silica coating followed by universal adhesive, which is according to Arpa et al., 2018.[12] Although the groups ZL and HC showed a decreasing of the means when applied silica coating, all materials evaluated were no affected by silane as separate step or as the same step to the adhesive (universal adhesive). It can suggest that the use of universal adhesive, that contain silane and MDP, other functional monomers, is a simple bonding procedure and effective.

In general, the microshear tests showed the highest bond strength values of repair bonding with composite resin to ZL and HC.

The failures mode analysis in this study showed predominant adhesive failures for all groups, considering 100% for NC and CR in groups before silica coating. Adhesives failures suggest lower bond strength and mixed failures are correlated with the improving of bond strength.[35] Because of the manufacturing process of CAD/CAM blocks, which improves mechanical and physical properties, the failure of type adhesive is expected to be more frequent than cohesive failures. The decrease in adhesive failures after silica coating for the NC and RC materials can be correlated with the increase of bond strength. The increase of adhesive failures for the ZL and HC materials can be correlated to the decrease the bond strength after the use of silica coating.

CONCLUSIONS

Based on the findings of this study is possible to conclude:

1) Tribochemical silica coating (Cojet) improves bonding with composite resin as repair material to nanoceramic (Lava Ultimate) and composite resin.

2) Tribochemical silica coating has negative effect on zirconia-reinforced lithium-silicate (Celtra Duo) and Feldspathic-composite hybrid ceramic (Enamic),

3) Separated silane and adhesive steps (silane + total-etch adhesive) results in similar bond strength to single bottle silane/adhesive systems (self-etching universal adhesive).

4) Silica coating followed by universal adhesive is recommended to increase bond strength of nanoceramic and composite resin on repair procedures.

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Table 1 - Chemical composition of materials used

Material ; Manufacturer	Type of material	Composition
Celtra Duo; Dentsply Sirona	Zirconia-reinforced lithium silicate ceramic block CAD/CAM	Lithium silicate with 10% ZrO_2
Enamic; Vita Zahnfabrik, Germany	Hybrid ceramic CAD/CAM block	UDMA, TEGDMA, 86wt% Feldspar ceramic enriched with aluminum oxide
Lava Ultimate; 3M ESPE Dental Products, St. Paul, MN	Resin nanoceramic CAD/CAM block	Bis-GMA, UDMA, Bis-EMA, TEGDMA with 80%wt, 20-nm silica and 4- to 11-nm zirconia nanoparticles, and zirconia/silica nanoclusters
Filtek Z350 XT; 3M ESPE Dental Products, St. Paul, MN	Nanofilled composite	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA, sílica filler, zirconia/sílica cluster filler
Condac porcelain; FGM, Joinville, SC, Brazil	5% Hydrofluoric acid	5% Fluoridric Acid, water, thickener, surfactant and colorant
Cojet; 3M ESPE Dental Products, St. Paul, MN	Tribochemical silica coating	SiO_2-coated Al_2O_3 (30μm)
Ultrap-Etch; Ultradent Products Inc, South Jordan, UT	35% phosphoric acid	35% phosphoric acid in aqueous solution form with CMC viscosity-enhancing agent
Clearfil Ceramic Primer Plus; Kuraray Noritake Dental Inc., Okayama, Japan	Ceramic primer	Ethanol, 3-MPS, 10-MDP
Scotchbond Universal; 3M ESPE Dental Products, St. Paul, MN	Self-etching universal adhesive	MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, and silane
Adper Scotchbond Multi Purpose – only the adhesive; 3M ESPE Dental Products, St. Paul, MN	Total-Etch adhesive	BisGMa, HEMA, Triphenylantimony

Abbreviations: UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol-dimethacrylate; Bis-GMA: bisphenol A glycidyl methacrylate; Bis-EMA: bisphenol A polyethylene glycol diether dimethacrylate; PEGDMA: polyethylene glycol dimethacrylate; 3-MPS: 3-methacryloxypropyl trimethoxysilane; 10-MDP: 10-methacryloxydecyl dihydrogen phosphate.

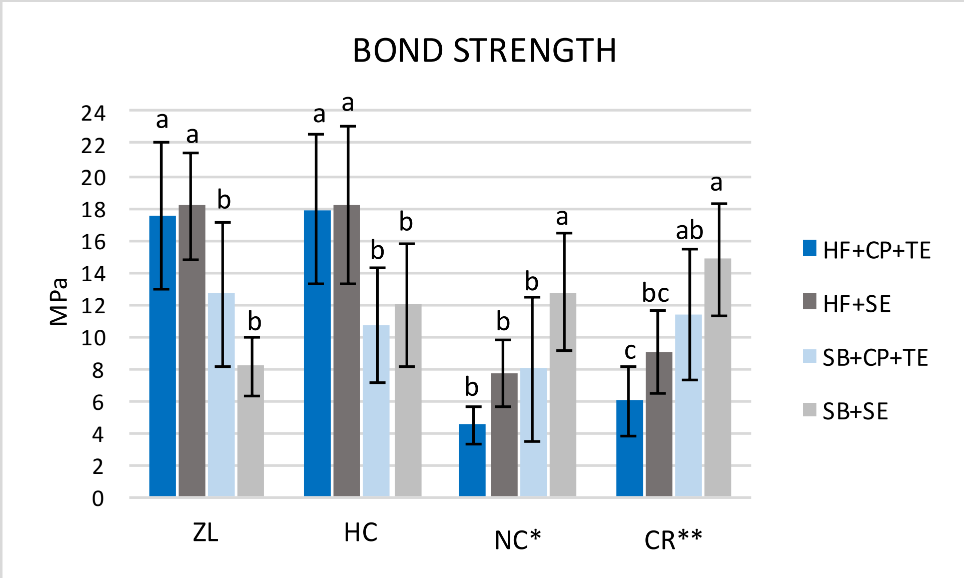

Figure 1 – Bond strength values (MPa) among all materials with different adhesive techniques. Similar lower-case letters indicate no significant differences among the techniques for each material tested.
*Indicates the group received ethanol only for surface cleaning instead of hydrofluoric acid
**Indicates the group received phosphoric acid only for surface cleaning instead of hydrofluoric acid

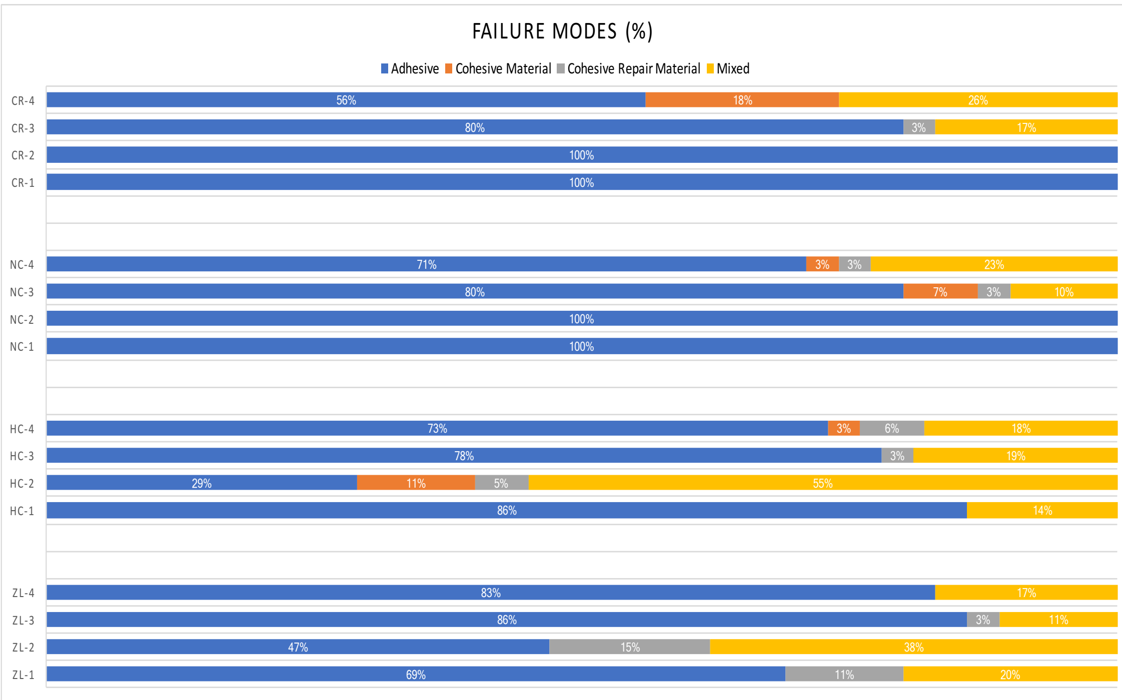

Figure 2 – Failure modes (%) for each material and techniques tested

4 CONSIDERAÇÕES FINAIS

Os materiais CAD / CAM podem apresentar maior estabilidade de cor e rugosidade devido à maneira como são fabricados com alta pressão e temperatura, porém há estudos que mostra estabilidade de cor maior para resinas processadas em laboratório, quando comparada às fresadas em CAD/CAM[17].

Existem estudos na literatura que afirmam que 24horas de compósitos imersos in vitro em soluções pigmentantes, correspondem aproximadamente a 1 mês in vivo[17,18]. Considerando que tanto para o Estudo 1 (cor) quanto para o Estudo 2 (rugosidade) foi avaliado um período de 60 dias in vitro, equivaleria aproximadamente a 5 anos em um envelhecimento clínico.

Procedimentos de polimento é uma etapa importante e imprescindível após um tratamento restaurador, para que assim possa ser evitado uma rugosidade da superfície, a impregnação de pigmentos e placas bacterianas, e desgaste contra o dente antagonista. Alguns estudos mostraram obtenção de superfícies mais lisas quando utilizado um sistema de polimento apropriado mais eficaz do que glaze ao forno[19].

No estudo 1 e 2, a solução de imersão "café", foi a que apresentou maiores valores significativos de alteração de cor e de rugosidade superficial.

No estudo 1, o procedimento de repolimento mostrou ser um procedimento eficaz na redução de alteração de cor superficiais aos materiais resina composta, Lava ultimate e Enamic. Outros estudos também demonstraram a eficácia do procedimento de repolimento na redução da alteração de cor[20,21].

Em relação ao estudo 3, no qual avaliou protocolos adesivos para reparo de materiais CAD/CAM com resina composta, mostrou os maiores valores de resistência de união aos grupos dos materiais Celtra Duo e Enamic. Para esses materiais, foi possível concluir que o melhor procedimento adesivo para reparo seria com ácido fluorídrico 5%, não diferindo da aplicação do silano em frasco separado ou junto ao sistema adesivo (adesivos universais). Para os materiais Lava e Resina composta, fica indispensável o uso de jateamento, que segundo alguns estudos, pode ser de sílica ou óxido de aluminio devido apresentarem resultados de resistência de união semelhantes[22].

Em geral, avaliando estes 3 estudos, entre os materiais de CAD/CAM e a resina composta avaliados, foi observado que Lava Ultimate se aproxima mais de propriedades à resina composta, e o Enamic se comporta mais próximo ao material Celtra Duo, considerando os testes avaliados nestes estudos.

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