UNIVERSIDADE ESTADUAL PAULISTA
“Júlio de Mesquita Filho”
Faculdade de Odontologia de Araraquara

Università Degli Studi di Padova

Francesco Saverio Ludovichetti

CAD/CAM monolithic materials: Wear resistance and abrasiveness, and the effect of grinding and polishing on their roughness and fracture resistance.

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Orientadora: Profa Dra. Renata Garcia Fonseca - UNESP
Orientador: Prof. Gaetano Granozzi - UNIPD

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ὁ δὲ ἀνεξέταστος βίος
οὐ βιωτὸς ἀνθρώπῳ

Σωκράτης

Resumo

Ludovichetti FS. Materiais monolíticos CAD/CAM: Resistência ao desgaste e abrasividade, e efeito do desgaste e polimento nas suas rugosidade e resistência à fratura [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2019.

Resumo

Esta tese consiste em dois estudos, ambos investigando materiais monolíticos fresados por tecnologia CAD-CAM.

Primeiro estudo. Objetivo. A resistência ao desgaste e abrasividade do Lava Ultimate, Vita Enamic, Vita Suprinity, IPS e.max CAD e Lava Plus, bem como algumas propriedades que podem estar relacionadas, foram analisadas. **Métodos.** Espécimes desses materiais tiveram sua rugosidade, dureza e coeficiente de atrito avaliados, respectivamente, em microscópio confocal, microdurômetro e tribômetro. O teste de desgaste conhecido como “2-body”, no qual os materiais atuaram como abrasivos e, juntamente com o esmalte bovino, também como antagonistas, também foi realizado. A taxa de desgaste foi determinada com perfilômetro de superfície e as superfícies desgastadas foram observadas por microscopia eletrônica de varredura (MEV). **Resultados.** Vita Enamic e Lava Ultimate mostraram a maior rugosidade, enquanto IPS e.max CAD e Vita Suprinity, a menor. O resultado da dureza foi Lava Plus> (Vita Suprinity = IPS e.max CAD)> Vita Enamic> Lava Ultimate. O Lava Ultimate exibiu maior coeficiente de atrito do que o IPS e.max CAD e o Lava Plus. O Lava Plus e o IPS e.max CAD mostraram um potencial significativamente maior para desgastar o Lava Ultimate. Estes dois materiais, juntamente com o Vita Suprinity, foram os que mais desgastaram o esmalte e o Vita Enamic. Vita Suprinity e IPS e.max CAD promoveram o maior desgaste do Lava Plus, e o inverso também ocorreu. Vita

Enamic e Lava Ultimate causaram o menor desgaste do esmalte e de todos os outros materiais avaliados. **Conclusão.** A resina composta nanoparticulada e a cerâmica infiltrada com polímero foram mais amigáveis ao antagonista (seja esmalte ou material) do que as vitrocerâmicas e zircônia. Cuidados devem ser tomados ao selecionar o material que irá entrar em contato principalmente com a vitrocerâmica. A dureza também deve ser considerada ao selecionar um material.
Palavras-chave: Projeto Auxiliado por Computador. Cerâmica. Resinas Compostas. Desgaste de Restauração Dentária. Propriedades Físicas. Dureza. Fricção.

Segundo estudo. Objetivo. Avaliar o efeito do desgaste e do polimento na rugosidade e resistência à fratura do Lava Ultimate, Vita Enamic, Vita Suprinity e IPS e.max CAD, submetidos ao envelhecimento mecânico. **Métodos.** Os discos destes materiais foram analisados quanto à rugosidade: 1) após o polimento com lixas de carbeto de silício (Lava Ultimate e Vita Enamic) ou aplicação do glaze (IPS e.max CAD e Vita Suprinity) (controle); 2) após o desgaste com ponta diamantada de 30 μm; 3) e após desgaste e polimento com o kit de polimento Ceramiste Polishers. Para a resistência à fratura, um modelo simplificado de três camadas consistindo de disco restaurador, disco de resina epóxi e um anel de aço, cimentados entre si, foi usado. Os discos tri-camada receberam as mesmos tratamentos de superfície descritos para a análise de rugosidade. Metade dos espécimes foi submetida ao envelhecimento mecânico por 1×10^6 ciclos. Todos os espécimes foram ensaiados até sua fratura. O módulo de Weibull foi calculado. **Resultados.** Entre os grupos controle, não foi encontrada diferença significativa entre o IPS e.max CAD e o Vita Suprinity e entre o Lava Ultimate e o Vita Enamic, os quais foram mais rugosos que os materiais vitrocerâmicos. Após o desgaste, esse comportamento foi mantido, com exceção do Vita Enamic, cuja rugosidade foi semelhante à do IPS e.max CAD. Após o polimento, o Vita Enamic mostrou a maior rugosidade, enquanto os outros materiais não foram estatisticamente diferentes. O IPS e.max CAD e o Vita Suprinity apresentaram a menor rugosidade nos grupos controle. Para Lava Ultimate e Vita Enamic, o polimento promoveu a menor rugosidade. O desgaste, seguido ou não por polimento, e o envelhecimento mecânico não afetaram adversamente a resistência à fratura ou a confiabilidade dos materiais. **Conclusões.** O polimento não recuperou a maior lisura inicial dos materiais vitrocerâmicos. A resistência à fratura não foi afetada pelo desgaste, seguido ou não por polimento, mesmo após o envelhecimento mecânico.

Palavras-chave: Projeto Auxiliado por Computador. Cerâmica. Resinas Compostas. Ajuste Oclusal. Polimento Dentário. Propriedades Físicas. Resistência dos Materiais.

Abstract

Ludovichetti FS. CAD/CAM monolithic materials: Wear resistance and abrasiveness, and the effect of grinding and polishing on their roughness and fracture resistance [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2019.

Abstract

This thesis consists of two studies, both investigating the computer-aided design and computer-aided manufacturing (CAD-CAM) monolithic materials.

First study. **Aim.** The wear resistance and abrasiveness of Lava Ultimate, Vita Enamic, Vita Suprinity, IPS e.max CAD, and Lava Plus, as well as some properties that might be related to, were analyzed. **Methods.** Specimens from these materials had their roughness, hardness, and coefficient of friction evaluated, respectively in confocal microscope, microdurometer, and tribometer. The 2-body wear test, wherein the materials acted as abraders and, together with bovine enamel, also as antagonists, was also carried out. The wear rate was determined with surface profilometer and the worn surfaces were observed by scanning electron microscopy (SEM). **Results.** Vita Enamic and Lava Ultimate showed the highest roughness, whereas IPS e.max CAD and Vita Suprinity, the lowest. The hardness result was Lava Plus > (Vita Suprinity=IPS e.max CAD) > Vita Enamic >Lava Ultimate. Lava Ultimate exhibited a higher coefficient of friction than IPS e.max CAD and Lava Plus. Lava Plus and IPS e.max CAD showed significantly higher potential to wear Lava Ultimate. These two materials, together with Vita Suprinity, provided the highest wear of enamel and Vita Enamic. Vita Suprinity and IPS e.max CAD exhibited the highest wear against Lava Plus, and the inverse also occurred. Vita Enamic and Lava Ultimate were among the materials that caused the lowest wear of enamel and all other evaluated materials. **Conclusion.** The nanofilled composite resin and polymer-infiltrated ceramic were more antagonist-friendly (whether enamel or CAD-CAM material) than glass-ceramics and zirconia. Care should be taken when selecting the material that will contact mainly with glass-ceramics. Hardness should also be considered when selecting a material.

Keywords: CAD-CAM. Ceramics. Composite Resins. Dental Restoration Wear. Physical Properties. Hardness. Friction.

Second study. **Aim**. To evaluate the effect of grinding and polishing on the roughness and fracture resistance of Lava Ultimate, Vita Enamic, Vita Suprinity, and IPS e.max CAD, submitted to mechanical aging. **Methods.** Disks from these materials were analyzed for roughness: 1) after polishing with silicon carbide papers (Lava Ultimate and Vita Enamic) or glazing (IPS e.max CAD and Vita Suprinity) (control); 2) after grinding with 30-µm grit diamond rotary instruments; 3) and after grinding and polishing with the polishing kit Ceramiste Polishers. For fracture resistance, a simplified tri-layer model consisting of restorative disk, epoxy resin disk, and a steel ring was used. The bonded tri-layer disks received the same conditions described for the roughness analysis. Half of the specimens underwent mechanical aging for 1×10^6 cycles. All specimens were loaded until failure. The Weibull modulus was calculated. **Results.** Among the control groups, no significant difference was found between the IPS e.max CAD and Vita Suprinity and between the Lava Ultimate and Vita Enamic, which were rougher than the glass-ceramic materials. After grinding, this behavior was maintained, except for the Vita Enamic, whose roughness was similar to that of the IPS e.max CAD. After polishing, the Vita Enamic showed the highest roughness, whereas the other materials were not statistically different. IPS e.max CAD and Vita Suprinity showed the lowest roughness in the control groups. For Lava Ultimate and

Vita Enamic, polishing provided the lowest roughness. Grinding followed or not by polishing, and mechanical aging, did not adversely affect fracture resistance or the reliability of the materials. **Conclusions.** Polishing did not recover the initial roughness of the glass-ceramic materials. Fracture resistance was not affected by grinding, followed or not by polishing, even after mechanical aging.

Keywords: CAD-CAM. Ceramics. Composite Resins. Occlusal Adjustment. Dental Polishing. Physical Properties. Material Resistance.

Introduction

1 Introduction

Thanks to the continuing scientific advancement, CAD-CAM technology has recently made tremendous progresses and it is able to easily satisfy many of the prosthetic requirements in Dentistry[1]. The great interest in dental prosthesis aesthetics, has led to an increased research of materials that mimic natural teeth behavior, both from the functional point of view and from the aesthetic one[2 3]. Nowadays the use of materials for CAD-CAM technology has shown different results with many options available to realize dental restorations[4]. Thanks to its good mechanical characteristics and aesthetic clinical results, the most used material for indirect CAD-CAM restorations is lithium disilicate (IPS e.max CAD, Ivoclar Vivadent)[5 6]. Recently, new materials with different composition and indications, including the Vita Suprinity (Vita Zahnfabrik), Vita Enamic (Vita Zahnfabrik), Lava Ultimate (3M ESPE), are being studied as viable substitutes to realize anterior and posterior crowns, implant supported crowns, anterior veneers and 2 or 3 elements bridges[7]. Vita Enamic contains feldspathic ceramic matrix (86 wt%) infiltrated with a low-viscosity copolymer (urethane dimethacrylate and triethylene glycol dimethacrylate)[8]. Vita Suprinity is composed by a lithium silicate glassy matrix and zirconia fillers[9]. Lava Ultimate is a resin nanoceramic material, consisting of 80 wt% zirconia/silica nanoparticles embedded in a highly cross-linked polymer matrix (BisGMA, UDMA, BisEMA, TEGDMA)[10]. Since their composition is different, it can be assumed that the mechanical and physical properties might change depending on the material, and literature still has gaps regarding both the surface characteristics and the mechanical behavior of these new materials.

In restorative dentistry, an ideal material should present a wear potential similar to that of the human enamel[11]. As reported by literature[12 13], some of the recently introduced dental ceramic systems seem to present this relevant property. Different Authors in many studies, showed that lithium disilicate (e.max CAD Ivoclar Vivadent), Lava Plus (3M ESPE), Enamic (VITA) and the zirconia reinforced glass ceramic do not show statistically significant difference in wear potential when compared with human enamel [12] [13] [26]. Even if wear potential is considered as "key role" material property in indirect restorative dentistry, studying it may not be sufficient, due to the multifactorial nature of this process which might be influenced by several other surface characteristics such as roughness, coefficient of friction and hardness.

Materials roughness is a known data provided by the manufacturer, but clinically, due to various adjustments and aging, it may drastically change. Surface clinical interventions lead to different roughness values, thus material roughness and its changes have to be investigated as they may predict the opposing teeth wear[14] [15] [16]. Ghazal et al.[17] (2009) and Janyavula et al.[14] (2013) studied how surface roughness may influence wear of human enamel and composite resin and of polished, glazed, polished and re-glazed zirconia respectively. They found that, when surface roughness increases, human enamel wear increased dramatically too. Authors explain that this could be caused by the frictional resistance: when surface roughness increases, coefficient of friction increases, and this results in a greater wear. They both concluded that surface roughness plays a fundamental role in the wear of opposing materials.

As previously seen, material roughness and coefficient of friction seems to be related and since friction forces occur in almost every oral cavity movement and they may negatively influence dental materials properties, Authors suggested that a better understanding of the relation between friction coefficient, roughness and wear may help in indirect restorative materials behavior investigation[18 19]. Ghazal et al.[17] (2009) showed that high surface roughness increases the friction coefficient, which results in greater wear. In the literature review of Oh et al.[20] (2002) was suggested that roughness, high loads, and high sliding speeds increases the coefficient of friction and this lead to a greater wear potential.

Finally, Literature is still divided about material hardness influence on wear potential. Some authors exclude it as a wear influencing property [20 21,] some others suggest that as wear is a multifactorial process, hardness could influence it [22 23]. As explained in the literature review of Oh et al.[20] (2002), wear potential of some materials, such as metals, is highly influenced by hardness: gold based casting alloys are relatively soft in comparison with harder metals, and they are more antagonist friendly. But when it comes to ceramics, material hardness would not able to affect wear potential by itself, but it can be seen as a co-factor (Seghi et al.[23] 1991, D'Arcangelo et al.[13] 2015). This may be explained due to the different wear mechanism of metal and ceramics. In metal wear mechanism is by plastic deformation while in dental ceramic materials it occurs by material micro-fractures.

The different composition of these materials could result in a different behavior in the wear of antagonist elements. Moreover, clinically, when they are submitted to adjustment processes, this difference may be greater as the surface characteristics change depending on the surface treatment adopted[24 25]. The ideal order for glass ceramics would be to adjust the restoration when it is still not cemented, then glaze it and cement it in mouth [26]. For Enamic (VITA) and Lava Ultimate (3M ESPE), the ideal procedure would be to cement the restoration right after it has been milled[8 10]. However, clinically this situation is not always reachable due to premature contacts that may appear after cementation. This lead to the need of occlusal adjustments and though to surface roughness increase, which could change the wear potential of the material[27]. In the attempt to decrease adjustment-caused roughness, a commonly used clinical protocol is to polish adjusted restorations with one of the numerous polishing systems available. However, literature has not still achieved a consensus regarding how material resistance could be affected by adjustments and polishing protocols and a better understanding of how different finishing material protocols may change their properties may lead to greater predictability in prosthesis duration and in its behavior towards antagonist teeth. This led to the second part of this thesis, where a situation as similar as possible to what we can find "in vivo" was analyzed. Manufacturer recommendations suggest that, after cementing prosthetic elements, no occlusal adjustments should be performed, but from the clinical point of view, this order is not always respectable. Often, it happens that, after cementation, there is a need for adjustments. For this reason, we wanted to reproduce realistic clinical situations that lead the dentist to deviate from the manufacturer recommendations. For this reason, we investigated if mechanical resistance of the studied materials could have undergone variations due to the adjustments procedures and tested this situation with non-aged and aged materials to assess if, in addition to adjustments, aging can alter the mechanical resistance. The ultimate goal of this thesis is to provide clinicians a guide in choosing the most suitable material for prosthetic rehabilitation, according to the different situations that can be found "in vivo", since today there are countless possibilities in terms of prosthetic materials, and decision making can be complicated and not always correct. Therefore, in this thesis the influence of Hardness, Coefficient of Friction and Roughness material properties on wear potential and how different finishing surface clinical protocols influence the material mechanical strength on the short and on the long time were investigated.

Manuscript 1

2 Manuscript 1

Wear resistance and abrasiveness of CAD-CAM monolithic materials.

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ABSTRACT

Statement of problem. Computer-aided design and computer-aided manufacturing (CAD-CAM) restorations are in contact with the antagonist tooth, either a natural tooth or a restoration. Therefore, clinicians should be aware of the wear resistance of CAD-CAM materials and the wear behavior of the antagonist.

Purpose. The purpose of this in vitro study was to evaluate the wear resistance and abrasiveness of CAD-CAM materials.

Material and methods. In a 2-body wear test, the materials IPS e.max CAD (Ivoclar Vivadent AG), Vita Suprinity (Vita Zahnfabrik), Lava Ultimate (3M ESPE), Vita Enamic (Vita Zahnfabrik), and Lava Plus (3M ESPE) acted as abraders and, together with bovine enamel, also as antagonists. Each antagonist wheel ran against each abrader wheel for 200 000 cycles, with a spring force of 15 N, and at a rotational speed of 1 Hz in distilled water. The wear rate was determined with a surface profilometer. The surfaces were observed with scanning electron microscopy, and their hardness, coefficient of friction, and roughness were evaluated.

Results. Results. Lava Plus and IPS e.max CAD exhibited the highest potential for wear of Lava Ultimate. These 2 materials, together with Vita Suprinity, provided the highest wear of enamel and Vita Enamic. Vita Suprinity and IPS e.max CAD had higher wear than Lava Plus, and the inverse was also true. Vita Enamic and Lava Ultimate were among the materials that caused the lowest wear of enamel and all other evaluated materials. Scanning electron microscopy images revealed that except for Lava Ultimate, all other materials damaged enamel, in which Vita Suprinity and IPS e.max CAD were more aggressive when sliding against the materials. Lava Plus had the greatest hardness, followed by Vita

Suprinity and IPS e.max CAD, Vita Enamic, and then Lava Ultimate. The coefficient of friction varied from 0.42 to 0.53. The Vita Enamic and Lava Ultimate showed the highest surface roughness.

Conclusions. The nanofilled composite resin and polymer-infiltrated ceramic were more antagonist-friendly (whether enamel or CAD-CAM material) than glass-ceramics and zirconia. Care should be taken when selecting the material that will contact mainly glass-ceramics. Hardness should also be considered when selecting a material.

CLINICAL IMPLICATIONS

Nanofilled composite resin and polymer-infiltrated ceramics are antagonist-friendly materials, whereas glass-ceramics promote high wear rates on the antagonist enamel and materials. Hardness should be considered when selecting CAD-CAM monolithic materials. The roughness and coefficient of friction of some materials may change during the wear process.

INTRODUCTION

An improvement in the esthetics of lithium disilicate and yttria-stabilized tetragonal zirconia ceramics has led to monolithic restorations, eliminating the problem of fracture and chipping of veneering porcelain.[1] In addition, the advent of computer-aided design and computer-aided manufacturing (CAD-CAM) technology has enabled restorations to be provided in a single session.[2] More recently, other esthetic CAD-CAM monolithic materials with similar indications as lithium disilicate and yttria-stabilized tetragonal zirconia ceramic have been introduced. These include zirconia-reinforced lithium silicate ceramic, polymer-infiltrated ceramic, and nanofilled composite resin.

In the absence of a veneering porcelain, these materials are in contact with the antagonist, which can be a natural tooth or a restoration. Therefore, the wear resistance and abrasiveness of these materials is important. The wear properties of zirconia,[3-8] lithium disilicate,[3,4,6,9,10,11] zirconia-reinforced lithium silicate,[10] polymer-infiltrated ceramic, and nanofilled composite resin[6,10,12] when opposed to enamel have been reported. However, the multifactorial nature of wear[6,13,14] makes it important to understand the wear potential of the materials based on the composition and microstructural aspects[15-17] that will determine their properties.[18-24] The role that the roughness,[6-9,25] coefficient of friction,[4,14] elastic modulus,[10] and hardness[6,14,15] play in determining wear potential has been investigated.

However, these studies used enamel as an antagonist,[4,5,7-13,15,26] and studies exploring the different possibilities of combinations among these materials are lacking. Therefore, the purpose of the present study was to evaluate the wear provided by 5 CAD-CAM monolithic materials on bovine enamel and on each other. To better understand the material behavior, the surface roughness, hardness, and coefficient of friction of the materials were also investigated. The null hypothesis was that no difference would be found among the materials regarding their potential to wear the enamel or each other.

MATERIAL AND METHODS

The materials used in the present study and their composition are listed in Table 1. Disk-shaped specimens were prepared from each material. The CAD-CAM blocks were shaped into cylinders by using a mechanical turner and cut into disks with a precision saw (Isomet 1000; Buehler) and then polished with silicon carbide abrasive papers (400- 600- 1200-grit; 3M) in a polisher (Metaserv 2000; Buehler) under water irrigation. Vita Suprinity and IPS e.max CAD disks were crystallized (Programat P310; Ivoclar Vivadent AG). Lava Plus disks were cut 20% thicker and then sintered (inFire HTC speed; Dentsply Sirona). Crystallization and sintering followed the manufacturers' recommendations.

For the microhardness test (n=5), 5 indentations were made in each specimen using a Vickers diamond indenter under 20 N load and a 20-second dwell time. Hardness values (GPa) were calculated according to the following equation: $H = P/2d^2$, where P is the load in newtons and d is the average of the diagonal values.

The coefficient of friction (n=5) was measured using a ball-on-flat tribometer (UMT-II; CETR Corp) according to AS G133–05.[27] Each specimen was secured on a holder and a load of 5 N was applied with a 2-mm-diameter stainless steel sphere. A tangential, cyclic 9.7-mm back and forward motion was applied to the specimen at 5 Hz for 600 seconds without irrigation. A new stainless-steel ball was used for each specimen. Testing was conducted at room temperature and humidity.

Surface roughness (µm) (n=8) was analyzed using a confocal microscope (Lext OLS 4100; Olympus). Three equidistant parallel measurements were made on each specimen. The average reading was designated as the Rq (root mean square roughness) value of each specimen. A single calibrated operator (F.S.L.) recorded all measurements.

The 2-body wear test was performed using the ACTA wear machine profiles.[28,29] One wheel (21 mm diameter and 7.5 mm wide) containing a 2-mm inner hole (abrader wheel) from each material was prepared (Fig. 1). After gluing 4 CAD-CAM blocks of each material together, a round diamond tip with an inner diameter of 21 mm was mounted on a table drill and used to obtain the abrader wheels under constant water irrigation. An inner hole of 2 mm was obtained following the same procedure. The round tip used for the Lava Plus had an inner diameter of 26 mm to compensate for zirconia shrinkage. Next, the IPS e.max CAD and Vita Suprinity were crystallized and the Lava Plus sintered. Each wheel was then mounted in the ACTA wear machine and polished against sequential abrasive wheels to obtain an even and curved cylindrical outer surface shape. A final polish of the outer surfaces was obtained using P1000 silicon carbide abrasive paper mounted on a wheel. Two antagonist metal wheels (48 mm diameter and 10 mm thick) with 10 rectangular compartments (14 mm long, 10 mm wide, and maximum 3 mm thick) were used. Rectangular specimens (3 from each material, 4 from bovine enamel, and 1 from Z250 composite resin were bonded with Panavia F2.0 to the antagonist wheel compartments of the ACTA wear machine (Fig. 2). Each specimen wheel was polished following the same protocol used for the abrader wheel. Finally, each abrader wheel rotated against each antagonist wheel.

Each antagonist wheel ran for 200 000 cycles with a spring force of 15 N at a rotational speed of 1 Hz in distilled water at room temperature. Next, 10 tracings were made at fixed positions on the worn surface of the specimens using a surface profilometer (PRK profilometer no. 20702; Perthen GmbH) to determine the loss of material in µm. The average wear rate was calculated from these profiles.[28,29] The worn surfaces were observed by scanning electron microscopy at ×100, ×500, ×1000, ×2000, ×5000, and ×10 000 (LS15; Zeiss). SEM specimens were made indirectly from a polyvinyl siloxane impression poured in epoxy resin (Araldite; Ciba-Geigy) and gold sputtered for electron conductivity.

The hardness and roughness data were square root transformed before the statistical analysis. Since the assumptions of the analysis of variance were satisfied (Shapiro-Wilk and Levene tests, $P>.05$), the hardness, coefficient of friction, and roughness data were submitted to 1-way ANOVA followed by the Tukey post hoc test ($\alpha=.05$) to determine significant differences among the materials. The wear data were analyzed by 2-way

ANOVA (abrader and antagonist) followed by the Tukey post hoc test. Statistical analysis was performed using statistical software (IBM SPSS Statistics v22.0; IBM Corp).

RESULTS

Table 2 shows the Vickers hardness mean values, standard deviations, and the statistical results (F=1898.12, *P*<.001). The hardness result was Lava Plus>(Vita Suprinity= PS e.max CAD)>Vita Enamic>Lava Ultimate. Table 3 presents the coefficient of friction mean values, standard deviations, and statistical results (F=6.126, *P*<.01). Lava Ultimate exhibited a higher coefficient of friction than IPS e.max CAD and Lava Plus. Roughness mean values (Rq), standard deviations, and statistical results (F=167.88, *P*<.001) are shown in Table 4. Vita Enamic and Lava Ultimate showed the highest roughness values, while the IPS e.max CAD and Vita Suprinity exhibited the lowest mean value. The 2-way ANOVA indicated that the abrader (F=31.37, *P*<.001) and antagonist (F=114.66, *P*<.001) factors, and their interaction (F=29.62, *P*<.001) were significant. Table 5 presents the antagonist wear provided by the abrader materials. The IPS e.max CAD, Lava Plus, and Vita Suprinity materials provided higher wear of enamel when compared with the Lava Ultimate and Vita Enamic. Lava Plus and the IPS e.max CAD showed significantly higher potential to wear the Lava Ultimate. The wear of the Vita Enamic against different materials exhibited the same behavior as the enamel. Vita Suprinity and IPS e.max CAD exhibited the highest wear against the Lava Plus, and the lowest, against Vita Enamic and Lava Ultimate. IPS e.max CAD and Vita Suprinity exhibited the highest potential to wear the Lava Plus.

The SEM images of the enamel surface worn by the materials (Fig. 3) revealed deep parallel grooves with broken fragments of Vita Suprinity (Fig. 3C) and a deeply pitted surface when sliding against the IPS e.max CAD (Fig. 3B). Sliding grooves were created by the Lava Plus (Fig. 3E). Slight cracks were caused by Vita Enamic (Fig. 3D), whereas an essentially smooth surface was detected when the enamel was rubbed against the Lava Ultimate (Fig. 3A). SEM images of the surface of the materials against themselves are presented in Figure 4. Lava Ultimate did not modify the surface of the materials, but some of its fragments can be seen on the surface of the Vita Suprinity (Fig. 4A) and IPS e.max CAD (Fig. 4B). Vita Enamic created superficial grooves on the Lava Ultimate (Fig. 4C). Vita Suprinity produced some pits on the Lava Ultimate (Fig. 4D) and Vita Enamic (Fig. 4E), while the surfaces of the other materials were flat with some fragments of Vita Suprinity on the IPS e.max CAD (Fig. 4F). Pits created by the IPS e.max CAD can be found on the Vita Enamic (Fig. 4G) and Vita Suprinity (Fig. 4H), while smooth flat surfaces were observed for the IPS e.max CAD itself and the Lava Plus. Finally, flat surfaces of Lava Ultimate, Vita Enamic, and Vita Suprinity were created when sliding against the Lava Plus. Some SEM images were lost because of technical problems, and, therefore, some of them were repeated at a different magnification.

DISCUSSION

In the current study, the 2-body wear produced by CAD-CAM monolithic materials on bovine enamel and on themselves[28,29] was evaluated. The null hypothesis that no difference would be found among the materials regarding their potential to wear the enamel and themselves was rejected. The enamel wear provided by all the tested materials after simulation of 200 000 cycles was lower than the clinical 2- and 3-body wear determined in vivo by Lambrechts et al[26] after 1 year. Nevertheless, ranking the restorative materials concerning their potential to wear enamel is important, especially in patients with high occlusal force and/or bruxism.

In general, Lava Plus and IPS e.max CAD wore the antagonists more than did Vita Enamic and Lava Ultimate, and no significant difference was found in wear potential, either between IPS e.max CAD and Suprinity (except for Lava Ultimate antagonist) or between Vita Enamic and Lava Ultimate. Other studies also reported that the IPS e.max CAD showed significantly higher enamel wear than the Vita Enamic[10] and Lava Ultimate.[6,10] In these studies, as in the present, the IPS e.max CAD showed significantly higher hardness than the Vita Enamic and Lava Ultimate. In addition to the IPS e.max CAD, Lava Plus also exhibited significantly higher hardness than Vita Enamic and Lava Ultimate. Differently from other studies[14-16] that did not find a strong relationship between hardness and wear potential, Mormann et al[6] reported that the lower the hardness, the lower the enamel wear, which is in accordance with the present study. We also observed that the lower the hardness, the lower the antagonist materials wear. Considering the hardness of enamel reported by Chun et al[18] (274.8 VH) and that found in the present study for the Lava Ultimate, Vita Enamic, IPS e.max CAD, Vita Suprinity, and Lava Plus, we observed that the materials with higher hardness than that of enamel presented higher enamel wear than those with lower hardness. In contrast with our result for the Lava Plus, Mormann et al[6] found that the inCoris (Dentsply Sirona), despite its high hardness value, provided the lowest enamel wear rate among the evaluated materials. Possibly, this is related to differences in grain size, composition, or manufacturing process. According to Seghi et al,[15] an understanding of the materials' microstructure might be useful in predicting their wear potential.

Besides hardness, other properties may be useful in determining the wear potential of the materials. Wang et al[4] reported that when the mismatch of the elastic modulus and the strength between the enamel and restorative materials is large, the enamel suffers high stress concentration and, consequently, stress abrasion. They stated[4] that the high strength and toughness of zirconia enabled it to resist surface damage under stress, keeping its fineness and coefficient of friction over time. In contrast, enamel suffers fatigue wear with microcrack formation and propagation in the subsurface.[4,5] Therefore, high hardness combined with high flexural strength (1200 MPa) and elastic modulus (210 GPa) may explain the considerable wear potential of the Lava Plus. In addition, as the specimens were exposed to water during the entire experiment, the low temperature degradation of zirconia with an increase in surface roughness[19-21] might have contributed to the high abrasion of the antagonists. In contrast, the elastic modulus of the IPS e.max CAD (67.2 GPa[10] and 95 GPa according to the manufacturer) was similar to that of enamel (60 to 100 GPa[22]).

Despite the differences in the composition and microstructure[17] of the lithium disilicate (IPS e.max CAD) and the zirconia-reinforced lithium silicate (Vita Suprinity) materials, they were not statistically different concerning the properties evaluated. In addition, Belli et al[17] reported that the Young modulus of these materials was similar. These similar properties explain the similarity in wear behavior found for IPS e.max CAD and Vita Suprinity.

Vita Enamic and Lava Ultimate showed the 2 lowest hardness values among all materials, while in the study by Mormann et al[6] the hardness of the Vita Enamic was not significantly higher than that of the Lava Ultimate. In addition, the elastic modulus of 21.5 GPa and 16.0 GPa found for the Vita Enamic and Lava Ultimate[10] is close to that of human dentin (20 GPa)[23] as has been reported by Awada et al.[24] The low stiffness and hardness explain the lower potential of the Vita Enamic and Lava Ultimate to wear enamel and the other materials. Lawson et al[10] observed that IPS e.max CAD and the zirconia-reinforced lithium silicate material, Celtra Duo, with a similar microstructure of that of Vita

Suprinity,[17] were generally stronger, stiffer, and harder than Vita Enamic and Lava Ultimate. The similarity in wear potential observed in the present study was also reported by Mormann et al[6] stating that the hybrid ceramic behaved similarly to composite resins concerning enamel wear.

A relationship was not found between roughness, coefficient of friction, and wear behavior. Lava Plus and IPS e.max CAD exhibited significantly higher wear potential but lower roughness than Vita Enamic and Lava Ultimate and a lower coefficient of friction than the Lava Ultimate. Mormann et al[6] reported that the IPS e.max CAD showed the lowest roughness value among all the evaluated materials (including Lava Ultimate, Vita Enamic, and inCoris) and significantly higher wear potential than the Lava Ultimate and inCoris. In contrast with some authors[7,25] that concluded that surface roughness influences and may predict enamel wear, the lack of a relationship found in the present study between surface roughness and wear potential is consistent with other studies.[8,9] During cyclic sliding, some materials may undergo changes in surface topography, which might modify their abrasiveness.[13,15] IPS e.max CAD contains approximately 70% lithium disilicate crystals in a glassy matrix. Wang et al[4] reported that during wear against enamel, the lower strength and softer glass matrix of the lithium disilicate glass ceramic wears more easily than the stronger and harder crystals, increasing the surface roughness and the coefficient of friction of the material. In addition, Culhaoglu et al[11] assumed that after the lithium disilicate loses the glaze, an increased rate of particle fracture occurs. The presence of material debris between the rubbing surfaces might increase the friction and accordingly the wear rate, both from the glass ceramic and enamel. These facts, along with the high hardness, explain the high enamel and antagonist material wear associated with IPS e.max CAD. The SEM images reported by Wang et al[4] of the worn enamel surface against lithium disilicate reveal an abrasive wear, with rough furrows with enamel granules chipped off, extruded lithium disilicate crystalline grains and fragments. Therefore, some materials may have their surface roughness and coefficient of friction changed during the wear process, explaining the poor relationship between these 2 properties and wear potential. Metzler et al[13] evaluating the wear of enamel provided by feldspathic porcelains, commented that the surface of the material is important in the beginning, but after the surface has been changed, the nature of the material determines the wear rates.

The SEM images of the enamel surface worn by the Vita Suprinity and IPS e.max CAD indicates an abrasive wear mechanism, as observed by some authors for IPS e.max CAD.[4,10,11] The sliding grooves created by the Lava Plus differ slightly from the study by Stawarczyk et al[5] in which cracks were observed on the enamel surface worn by polished monolithic zirconia. The Vita Enamic produced small cracks and Lava Ultimate an essentially smooth surface, which is in agreement with the images reported by Morman et al.[6] The SEM images of the materials show that the Lava Ultimate and Lava Plus did not damage the surface of the materials, despite the high wear rate produced by this last material. In contrast, the Vita Suprinity and IPS e.max CAD caused more damage to the materials. Some pits were observed on the surface of the Lava Ultimate and Vita Enamic when against Vita Suprinity. Pits were also present on the surface of the Vita Enamic and Vita Suprinity as a result of the sliding against the IPS e.max CAD with supposed fragments of Vita Suprinity on the surface of this last material, suggesting an abrasive wear mechanism.

A limitation of this study was that the enamel was not used as abrader (not possible with the methodology used); therefore, qualitative and quantitative information about its potential to wear itself and the evaluated antagonist materials is lacking.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The nanofilled composite resin and polymer-infiltrated ceramic were the most antagonist-friendly materials when sliding against enamel and any other material.

2. Lithium disilicate, zirconia-reinforced lithium silicate, and zirconia caused high wear rates on the enamel and materials, with the difference that zirconia did not damage the surface of the materials, except for the enamel.

3. Hardness should be considered in the selection of materials, especially in patients with bruxism.

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FIGURES

Figure 1. Abrader wheel from Vita Enamic before polishing.

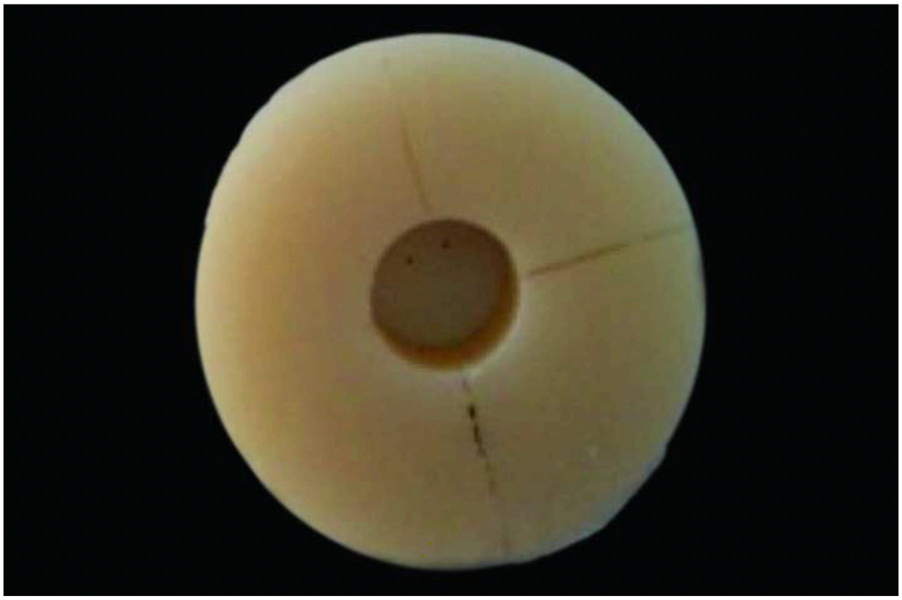


Figure 2. Antagonist wheels before polishing.

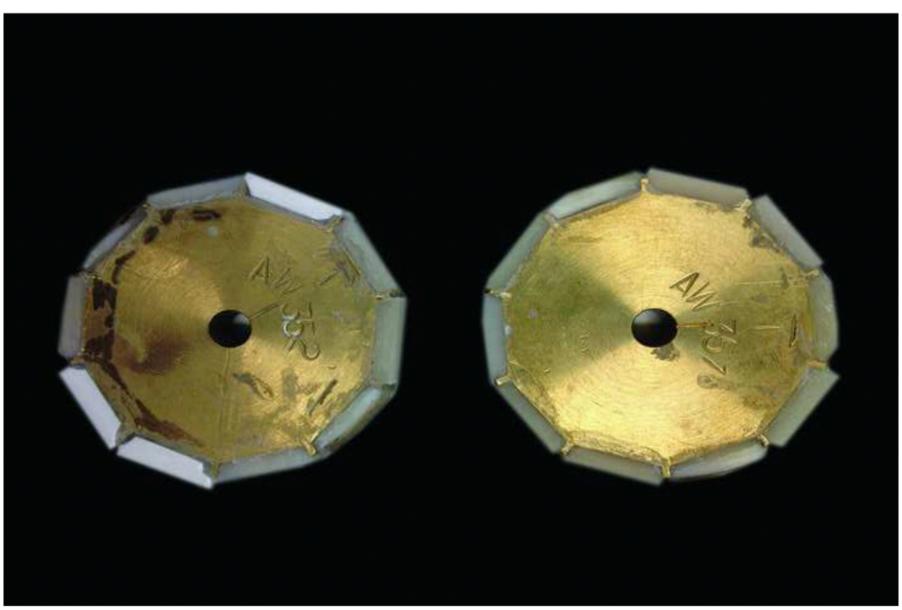


Figure 3. Scanning electron microscope images (original magnification ×2000) of enamel surfaces. A, Against Lava Ultimate. B, Against Vita Enamic. C, Against Vita Suprinity. D, Against IPS e.max CAD. E, Against Lava Plus.

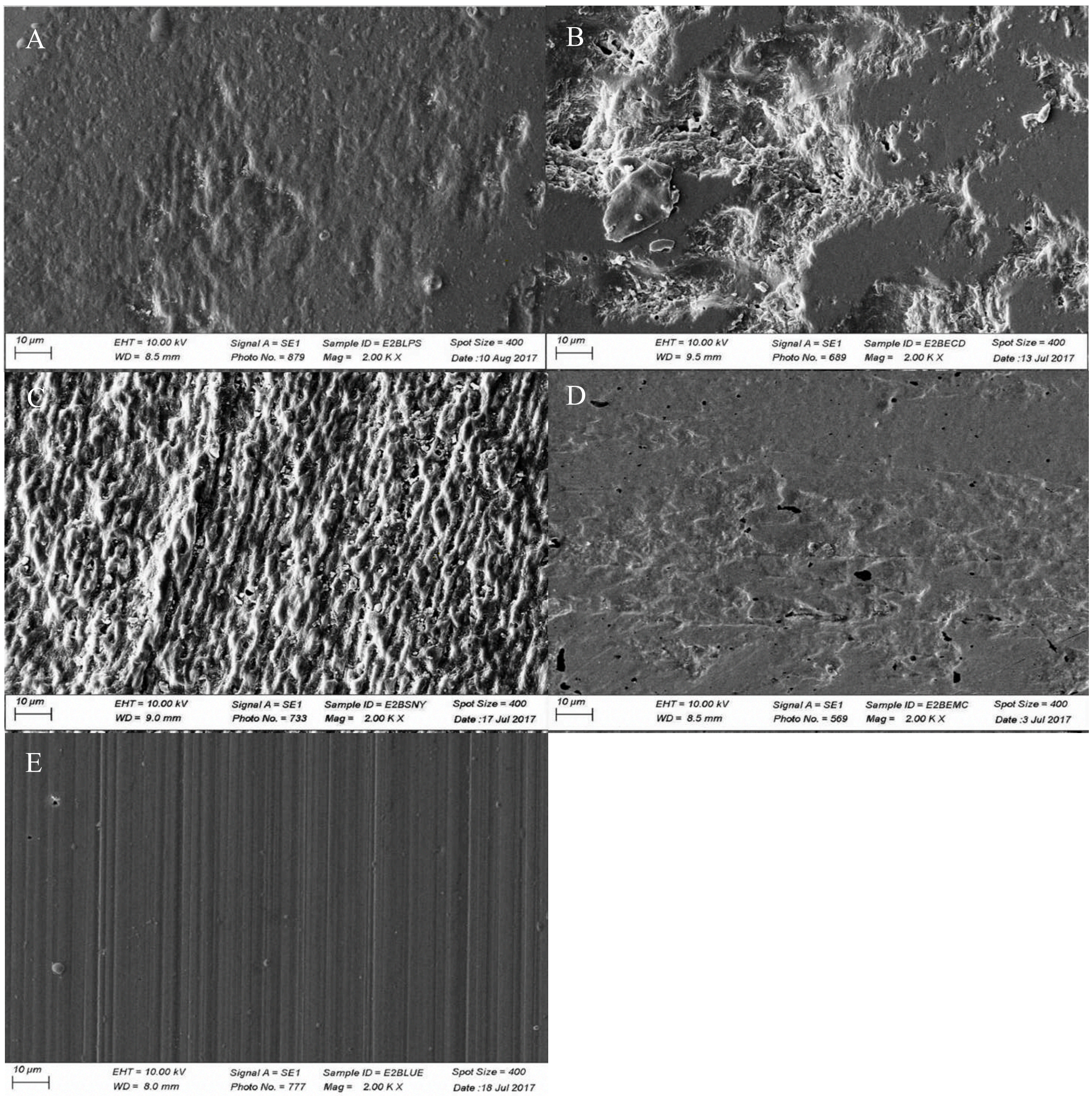
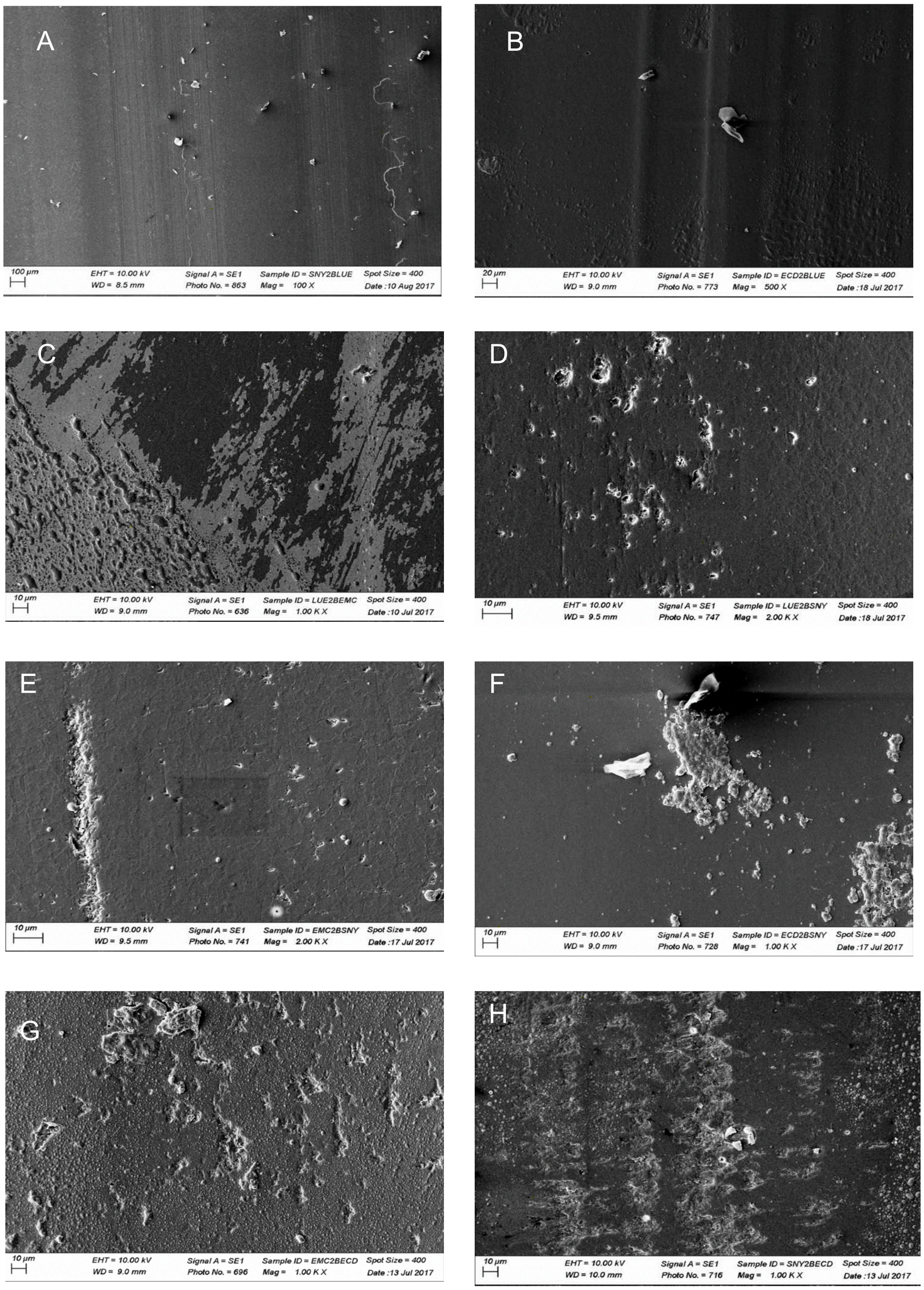


Figure 4. Scanning electron microscope images. A, Vita Suprinity surface against Lava Ultimate (original magnification ×100). B, IPS e.max CAD surface against Lava Ultimate (original magnification ×500). C, Lava Ultimate surface against Vita Enamic (original magnification ×1000). D, Lava Ultimate surface against Vita Suprinity (original magnification ×2000). E, Vita Enamic against Vita Suprinity (original magnification ×2000). F, IPS e.max CAD against Vita Suprinity (original magnification ×2000). G, Vita Enamic against IPS e.max CAD (original magnification ×1000). H, Vita Suprinity against IPS e.max CAD (original magnification ×1000).


A
100 µm
EHT = 10.00 kV
WD = 8.5 mm
Signal A = SE1
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Sample ID = SNY2BLUE
Mag = 100 X
Spot Size = 400
Date :10 Aug 2017
B
20 µm
EHT = 10.00 kV
WD = 9.0 mm
Signal A = SE1
Photo No. = 773
Sample ID = ECD2BLUE
Mag = 500 X
Spot Size = 400
Date :18 Jul 2017
C
10 µm
EHT = 10.00 kV
WD = 9.0 mm
Signal A = SE1
Photo No. = 636
Sample ID = LUE2BEMC
Mag = 1.00 K X
Spot Size = 400
Date :10 Jul 2017
D
10 µm
EHT = 10.00 kV
WD = 9.5 mm
Signal A = SE1
Photo No. = 747
Sample ID = LUE2BSNY
Mag = 2.00 K X
Spot Size = 400
Date :18 Jul 2017
E
10 µm
EHT = 10.00 kV
WD = 9.5 mm
Signal A = SE1
Photo No. = 741
Sample ID = EMC2BSNY
Mag = 2.00 K X
Spot Size = 400
Date :17 Jul 2017
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EHT = 10.00 kV
WD = 9.0 mm
Signal A = SE1
Photo No. = 728
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EHT = 10.00 kV
WD = 9.0 mm
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Sample ID = SNY2BECD
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Spot Size = 400
Date :13 Jul 2017

Table 1. Materials used in this study.

Material	Classification	Composition	Manufacturer
Lava Ultimate	Resin nanoceramic	80% nanoceramic and 20% resin matrix	3M ESPE
Vita Enamic	Polymer-infiltrated ceramic network	86% feldspathic ceramic and 14% polymer	Vita Zahnfabrik
Vita Suprinity	Zirconia-reinforced lithium silicate ceramic	Lithium silicate with ~10% ZrO_2	Vita Zahnfabrik
IPS e.max CAD	Lithium disilicate ceramic	57-80% SiO_2, 11-19% Li_2O, 0-13% K_2O, 0-11% P_2O_5, 0-8% ZrO_2, 0-8% ZnO, 0-5% Al_2O_3, 0-5% MgO	Ivoclar Vivadent AG
Lava Plus	Tetragonal polycrystalline zirconia partially stabilized with 3mol-% yttria	99% ZrO_2	3M ESPE

Table 2. Mean Vickers hardness (VH), SD and statistical results.

	Mean	Statistical results
Lava Ultimate	96.0 ± 6.5	A
Vita Enamic	200.4 ± 5.5	B
Vita Suprinity	632.0 ± 16.8	C
IPS e.max CAD	617.5 ± 44.3	C
Lava Plus	1343.0 ± 46.7	D

SD, standard deviation. Different uppercase letters indicate significant differences ($P<.05$).

Table 3. Mean coefficient of friction, ±SD, and statistical results

Material	Mean	Statistical results
Lava Ultimate	0.53 ± 0.05	A
Vita Enamic	0.50 ± 0.04	ABC
Vita Suprinity	0.51 ± 0.05	AB
ISP e.max CAD	0.45 ± 0.04	BC
Lava Plus	0.42 ± 0.02	C

SD, standard deviation. Different uppercase letters indicate significant differences ($P<.05$).

Table 4. Mean roughness Rq (μm) of polished surfaces, ±SD, and statistical results

	Mean	Statistical results
Lava Ultimate	0.37 ± 0.09	A
Vita Enamic	0.40 ± 0.06	A
Vita Suprinity	0.05 ± 0.01	C
ISP e.max CAD	0.05 ± 0.01	C
Lava Plus	0.29 ± 0.04	B

SD, standard deviation. Different uppercase letters indicate significant differences ($P<.05$).

Table 5. Mean antagonist wear (μm), SD and statistical results.

	Antagonist					
Abrader	enamel	Lava Ultimate	Vita Enamic	Vita Suprinity	IPS e.max CAD	Lava Plus
Lava Ultimate	1.8 ± 1.0 Ba	1.2 ± 1.1 Bab	0.2 ± 1.0 Bbc	0.7 ± 0.4 Cab	-0.1 ± 0.5 Cc	-0.2 ± 0.3 Bc
Vita Enamic	0.9 ± 1.9 Bab	2.2 ± 2.4 Ba	0.5 ± 0.6 Bab	0.6 ± 1.2 Cab	0.5 ± 1.4 Cab	-0.1 ± 0.5 Bb
Vita Suprinity	3.8 ± 4.0 Aa	1.8 ± 1.6 Ba	4.3 ± 5.3 Aa	2.4 ± 3.3 Ba	2.9 ± 3.5 Ba	0.1 ± 0.6 ABa
IPS e.max CAD	6.0 ± 7.3 Aa	4.0 ± 2.6 Aa	4.0 ± 4.6 Aa	3.2 ± 3.9 Ba	2.3 ± 2.8 Ba	0.4 ± 0.6 Aa
Lava Plus	5.4 ± 1.9 Ac	4.2 ± 2.6 Ac	6.5 ± 3.4 Abc	14.5 ± 5.5 Aa	13.8 ± 5.6 Aab	0.0 ± 0.3 Bd

Different uppercase letters indicate significant differences in columns (*P*<.05)
Different lowercase letters indicate significant differences in rows (*P*<.05)

Manuscript 2

3 Manuscript 2

The effect of grinding and polishing on the roughness and fracture resistance of cemented CAD/CAM monolithic materials submitted to mechanical aging.

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RESEARCH AND EDUCATION

Effect of grinding and polishing on the roughness and fracture resistance of cemented CAD-CAM monolithic materials submitted to mechanical aging

ABSTRACT

Statement of problem. The effect of clinical adjustments on the strength of cemented computer-aided design and computer-aided manufacturing (CAD-CAM) monolithic materials under aging challenge is unclear.

Purpose. The purpose of this in vitro study was to assess the surface roughness and fracture resistance (with or without mechanical aging) of cemented CAD-CAM monolithic materials submitted to grinding and polishing procedures.

Material and methods. Disks of Lava Ultimate, Vita Enamic, crystallized Vita Suprinity, and IPS e.max CAD were analyzed for roughness after polishing with silicon carbide papers (Lava Ultimate and Vita Enamic) or glazing (IPS e.max CAD and Vita Suprinity) (control), after grinding with 30-µm grit diamond rotary instruments, and after grinding and polishing with a polishing kit. For fracture resistance, a simplified trilayer model consisting of a restorative disk, an epoxy resin disk, and a steel ring was used. The bonded trilayer disks received the same conditions described for the roughness analysis. Half of the specimens underwent mechanical aging for 1×10^6 cycles. All specimens were loaded until failure. The Weibull modulus was calculated.

Results. The IPS e.max CAD and Vita Suprinity showed higher roughness after grinding but lower than the control groups. For the Lava Ultimate and Vita Enamic, polishing

provided lower roughness than at baseline. Grinding, followed or not by polishing, and mechanical aging did not adversely affect the fracture resistance or the reliability of the materials.

Conclusions. Polishing did not recover the initial surface roughness of the glass-ceramic materials. Fracture resistance was not affected by grinding, followed or not by polishing, even after mechanical aging.

CLINICAL IMPLICATIONS

Grinding glass-ceramic restorations should be avoided, since the smoothness given by the glaze can be compromised, even after polishing.

INTRODUCTION

Research to improve the esthetic properties of monolithic materials proceeds in parallel with the development of their physical and mechanical properties in an attempt to replicate those of the natural teeth.[1-3] Monolithic materials indicated for indirect restorations are available for computer-aided design and computer-aided manufacturing (CAD-CAM) technology, including yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramic, lithium disilicate, zirconia-reinforced lithium silicate ceramic, polymer-infiltrated ceramic, and nanofilled composite resin.[4-7]

Ideally, indirect restorations should not require any adjustment at the delivery appointment. However, this is not always possible, since the removal of premature contacts or adjustment of the proximal contact areas may be needed.[8-11] Clinical adjustments may have undesirable consequences such as rougher surfaces,[9,12-15] which may facilitate biofilm formation,[16,17] increase antagonist wear,[18-20] or affect the restoration color.[21-26] In addition, surface roughness induces stress concentrations, compromising porcelain strength.[27-31] Clinical adjustments with diamond rotary instruments may also be detrimental to the strength of lithium disilicate glass-ceramics[32,33] and Y-TZP ceramics.[34-36] In addition to the adjustment configuration (diamond rotary instrument grit size, handpiece speed, wet or dry conditions), the damage tolerance of indirect restorative materials submitted to grinding depends also on their mechanical properties,[32,37] which are determined by the composition and microstructure.[38,39] Since polymer-containing materials exhibit lower elastic modulus than lithium disilicate,[40-42] they absorb stress better by elastic deformation, minimizing the flaws or defects that grow in brittle materials.[32,43,44]

When ceramic restorations have been adjusted before cementation, they can be glazed. However, once cemented, polishing is recommended to reduce the roughness of the ground surfaces and minimize the adverse effects on the strength of the ceramic or metal-ceramic fixed dental prostheses,[8] glass-ceramics,[32,43,45-47] and Y-TZP ceramics.[35] To the best of the authors' knowledge, no studies have investigated the effect of clinical adjustments and polishing in cemented specimens submitted to aging. The purpose of this in vitro was to assess the surface roughness and fracture resistance (with or without mechanical aging) of cemented CAD-CAM monolithic materials submitted to grinding and polishing procedures. The null hypothesis was that the surface treatments would not affect the surface roughness and fracture resistance (even with aging) of the evaluated materials.

MATERIAL AND METHODS

The evaluated materials are listed in Table 1. The CAD-CAM blocks were transformed into cylinders (Ø 10 mm), which were sliced in sixty-eight 1.8-mm disks and thirty 1.5-mm disks with a precision saw (Isomet 1000; Buehler). The disks were polished (400-600-

1200-grit silicon carbide papers) (3M) under irrigation in a polisher (Metaserv 2000; Buehler). Vita Suprinity and IPS e.max CAD disks were glazed (VITA AKZENT Plus GLAZE SPRAY; VITA Zahnfabrik) and crystallized (Programat P310; Ivoclar Vivadent AG).

For the roughness (Fig. 1), eight 1.8-mm disks from each material were analyzed after polishing (Lava Ultimate and Vita Enamic) or glazing (IPS e.max CAD and Vita Suprinity) (control groups). Using a custom matrix with a circular central hole (10-mm diameter and 1.5- mm thickness), the same disks were ground to 0.3 mm with 30-µm grit diamond rotary instruments (#3101FF; KG Sorensen) in a high-speed handpiece (KaVo Dental Corp) under constant irrigation, and a second measurement was recorded. Then, the ground disks were polished with a polishing kit (Ceramiste Polishers; SHOFU Dental GmbH) in a slow-speed motor (BELTEC MICROMOTOR LB100) for 30 seconds in one direction and 30 seconds in the opposite direction under constant irrigation. A 2-µm to 4-µm diamond paste (Diamond Excel; FGM Produtos Odontológicos) was applied with a felt disk in the same way as described for polishing, and a third measurement was made. Grinding and polishing were performed by a single operator in a custom-made device (USICAP). The surface roughness (Rq) was measured using a 3D Laser Confocal Microscope (LEXT OLS 4100; Olympus) at ×5 magnification. Three equidistant parallel measurements were made on each specimen, and the mean value was calculated (µm). The baseline, ground, and ground and polished surfaces were examined using a scanning electron microscope (SEM) (Leica Cambridge Stereoscan 440) at ×3000 magnification.

Sixty 1.8-mm disks and thirty 1.5-mm disks from each material were prepared for the fracture resistance test. A simplified trilayer model[48] was used consisting of a restorative disk, G10 epoxy resin disk (Ø 10 mm and 2.0 mm thickness) to simulate the dentin, and a steel ring (6.5 mm inner diameter, 10 mm outer diameter, and 1.5 mm thickness) to replicate the pulp chamber. The restorative disk was cemented to the epoxy resin disk, which in turn was cemented to the steel ring. For this, the Lava Ultimate, Vita Enamic, and the steel ring were airborne-particle abraded with Al_2O_3, the IPS e.max CAD and Vita Suprinity were etched with 9.5% hydrofluoric acid (PorcelEtch; Cosmedent), and the epoxy resin disks were etched with 40% phosphoric acid (K-Etchant Gel; Kuraray). The restorative disks were treated with mixed Clearfil SE Bond; Primer and Clearfil Porcelain Bond Activator (Kuraray), while the epoxy resin disks and the steel rings were treated and cemented to each other with Clearfil SE Bond; Primer. The restorative disks were cemented to the epoxy resin disks with the resin cement Panavia F 2.0 (Kuraray). A 10-N load was applied on the specimen, and the 3 opposing sides were each light polymerized for 60 seconds.

The trilayer disks were allocated according to Figure 2. The surface procedures for the control, ground, and ground and polished conditions have been previously described. Half of the specimens was stored in distilled water at 37 °C, and the other half was aged in a universal cycling machine (Biocycle; Biopdi) for 1×10^6 cycles at a frequency of 2 Hz with a 100-N load in distilled water at 37 °C. All the specimens were loaded in a mechanical testing machine (EMIC DL2000; EMIC Equipment and Systems Testing Ltd) at a crosshead speed of 1 mm/min with a hemispherical steel indenter (Ø=4.9 mm) centered on the top surface. The load (N) at failure of each specimen was recorded as the fracture resistance.

Strength reliability was assessed by the formula: $P=1 - \exp[(-\sigma/\sigma0)^m]$, where P is the probability of failure, σ is the biaxial flexural strength, σ0 is the characteristic strength at the fracture probability of 63.21%, and m is the Weibull modulus. For the roughness analysis, the data were log-transformed to meet the assumptions of parametric analysis (Shapiro-Wilk and Levene, $P>.05$) and submitted to a mixed repeated-measures ANOVA followed by the Tukey test. The fracture resistance data were analyzed by 3-way ANOVA and the Tukey test ($\alpha=.05$). Statistical software (IBM SPSS Statistics, v22.0; IBM Corp) was used for the analysis.

RESULTS

Statistics on the roughness data (Table 2) indicated significance for the main effects and interaction. Table 3 shows the roughness results. Of the control groups, no significant difference was found between the IPS e.max CAD and Vita Suprinity or between the Lava Ultimate and Vita Enamic, which were rougher than the glass-ceramic materials. These results are corroborated by the SEM images (Fig. 3). After grinding, this behavior was maintained, except for the Vita Enamic, whose roughness was similar to that of the IPS e.max CAD. After polishing, the Vita Enamic showed the highest roughness, whereas the other materials were not statistically different. The IPS e.max CAD and Vita Suprinity exhibited lower baseline roughness, which was also observed in the SEM images, that show that polishing the IPS e.max CAD and Vita Suprinity (Fig. 3C and 3F) did not recover their initial smoothness (Fig. 3A and 3D). In contrast, for the Vita Enamic and Lava Ultimate, polishing provided the lowest roughness.

Statistical analysis of the fracture resistance data (Table 4) showed that the 3 factors and their interactions were significant. Table 5 shows the fracture resistance results. The surface treatment did not influence the fracture resistance of the materials, except when the IPS e.max CAD was submitted to cyclic fatigue. Aging did not reduce the fracture resistance of the materials. The IPS e.max CAD/control, Vita Suprinity/control, Vita

Suprinity/ground, and Vita Suprinity/polished aged groups showed a significant increase in fracture resistance compared with their respective non-aged groups. The results of the Weibull modulus (Table 6) show that reliability was not influenced by the material, surface treatment, or aging.

DISCUSSION

One of the purposes of the present study was to investigate whether occlusal adjustment and polishing protocol modified the initial roughness of CAD-CAM monolithic materials. In view of the obtained results, the first null hypothesis was rejected. In this study, differences in roughness among the materials were observed, which was expected considering that distinct materials were evaluated. Of the control groups, the similarity between the IPS e.max CAD and Vita Suprinity was probably because the same glaze was applied on their surfaces. However, even after the grinding and polishing procedures, this similarity was maintained (except for the comparison between the polished groups analyzed by the SEM), despite the differences in composition and microstructure between these materials.[40] Strasser et al[12] also reported statistically similar roughness after both glass-ceramics had been ground with 4-µm or 80-µm diamond rotary instruments. Differently, Vichi et al[45] reported that Vita Suprinity presented significantly lower roughness than IPS e.max CAD after glazing and polishing. This difference can be attributed to the different systems used for each material. Regarding the Lava Ultimate and Vita Enamic, the similarity between the control groups was in good agreement with the SEM images and with the study by Mörmann et al.[9] In contrast, the similarity found between the ground groups was not consistent with the study by Strasser et al,[12] in which Vita Enamic was rougher than the Lava Ultimate after being ground with a coarse-grit diamond rotary instrument (80-µm).

In the present study, even though grinding was carried out with extrafine diamond rotary instruments, it increased the roughness of the materials, except for the Vita Enamic. The rougher topography caused by grinding is evidenced in the SEM images, mainly for the IPS e.max CAD and Vita Suprinity. This finding was also reported in other studies after the materials had been ground with different grit sizes.[12-15] For both glass-ceramics, polishing after grinding was not able to recover the smoothness given by the glaze that was below the threshold for bacterial adhesion (0.20 µm).[16] This behavior was also reported in previous studies[11,13,46] that investigated lithium disilicate ceramic and is probably related to the high hardness of both materials.[9,41] However, when the polishing was finished with a diamond paste, the roughness of the lithium disilicate was similar to[13] or significantly lower[47] than that of the glazed material, evidencing the importance of this step when clinical adjustments are performed on these materials. For the Lava Ultimate and Vita Enamic, the higher smoothness achieved by polishing after grinding, as previously reported by Fasbinder et al,[10] shows that, even though these materials do not require clinical adjustments, they should be polished.

The second null hypothesis was rejected, since the IPS e.max CAD/aged was adversely affected by the surface treatments. Albakry et al[43] also reported that grinding did not influence the strength of a non-aged pressable lithium disilicate, even with the use of a110-µm diamond disk. In contrast, Curran et al[32] reported that grinding with an18-µm diamond disk was still quite detrimental to the IPS e.max CAD, with chip crack formation and a strength loss estimated at 42%. Song et al[44] also observed intergranular and transgranular fractures after diamond rotary instrument penetration into the lithium disilicate ceramic. In the present study, in which the specimens had been cemented before they were ground, grinding affected the fracture resistance of the IPS e.max CAD only in the aged groups. This was consistent with the findings of Mohammadibassir et al,[47] who commented that with cyclic loading and moisture, cracks resulted from the grinding propagate and decreased the strength. However, this significant reduction in the IPS e.max CAD/aged groups was due to the high mean value of the IPS e.max CAD/control/aged group, which draws attention when it is compared with the IPS e.max CAD/control/non-aged group and for which an explanation was not found in the literature. Despite this reduction, the IPS e.max CAD/ground/aged and IPS e.max CAD/polished/aged groups showed no significant difference compared with their respective non-aged groups and with the IPS e.max CAD/control/non-aged group, ensuring such procedures when they are required.

Although Vita Suprinity was not affected by grinding, it showed an increase in fracture resistance after cyclic fatigue, regardless of the surface treatment condition. This was unexpected, since aging was expected to reduce the fracture resistance, especially of both the glass-ceramics submitted to grinding. Strasser et al[12] reported that water-cooled grinding with 80-μm diamond rotary instruments caused severe microchipping in both the Vita Suprinity and IPS e.max CAD. However, the crack formation was slight for the former and moderate for the latter. This can be explained by the significantly higher fracture toughness found for Vita Suprinity compared with IPS e.max CAD,[38] indicating that Vita Suprinity presents higher resistance against crack propagation than IPS e.max CAD. Also, Vita Suprinity has significantly higher flexural strength than IPS e.max CAD.[38,39] Elsaka and Elnaghy[38] and Sen et al[39] attributed this behavior to the presence of the zirconia fillers used to reinforce the glassy matrix of Vita Suprinity. Ramos et al[42] detected zirconium oxide and cerium throughout the entire surface of this material. These findings help explain why the fracture resistance of Vita Suprinity was not affected by the surface treatments, even after aging.

Regarding the maintenance of the fracture resistance of Vita Enamic and Lava Ultimate, Curran et al[32] found that, even after grinding and mechanical aging, these materials, due to a combination of lower hardness and lower elastic modulus, have a high resistance to crack initiation and growth. They stated that loss in their strength was not observed even when ground with a 75-µm grit size. This is in accordance with 2 other studies that reported little damage from grinding, with only shallow or absent cracks.[12,37]

In the current study, the Weibull modulus was not influenced by the material, surface treatment, or aging. The Weibull modulus represents the scattering of the fracture resistance data, being that a high modulus means that the defects of the material are evenly distributed, having a low risk for presence of critical flaws. The present results differ from those of a previous study[43] reporting that grinding a heat-pressed lithium disilicate ceramic with a 110-µm diamond disk reduced its reliability compared with that of the untreated material. According to Albakry et al,[43] grinding introduces defects and flaws distributed over a wide area, resulting in a wider range of strength values. Probably, the disparity of the results can be attributed to the methodological differences between the studies, considering that, in the present study, the grit size of the diamond instrument used was much smaller and that the specimens were cemented. Further studies involving coarser diamond rotary instruments and additional aging methods are required for a more comprehensive picture of the behavior of materials when submitted to clinical adjustments. The evaluation of flat specimens can be considered a limitation of the present study, since it does not closely reproduce what may happen in the oral cavity.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Grinding increased the roughness of the materials, except for the Vita Enamic. The smoothness of the glazed glass-ceramics was not restored by the polishing kit, while the Lava Ultimate and Vita Enamic showed a smoother surface compared with the baseline.

2. Grinding followed or not by polishing did not impair the fracture resistance of the materials.

3. Aging did not reduce the fracture resistance of the materials, not even in the ground groups.

4. The reliability of the materials was not influenced by the material, surface treatment, or aging.

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FIGURES

Figure 1. Roughness analysis scheme.

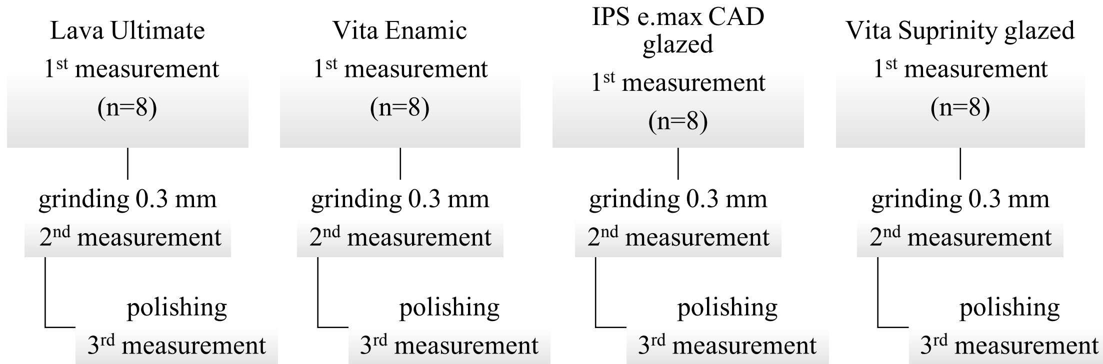


Figure 2. Fracture resistance: Groups analyzed for each material.

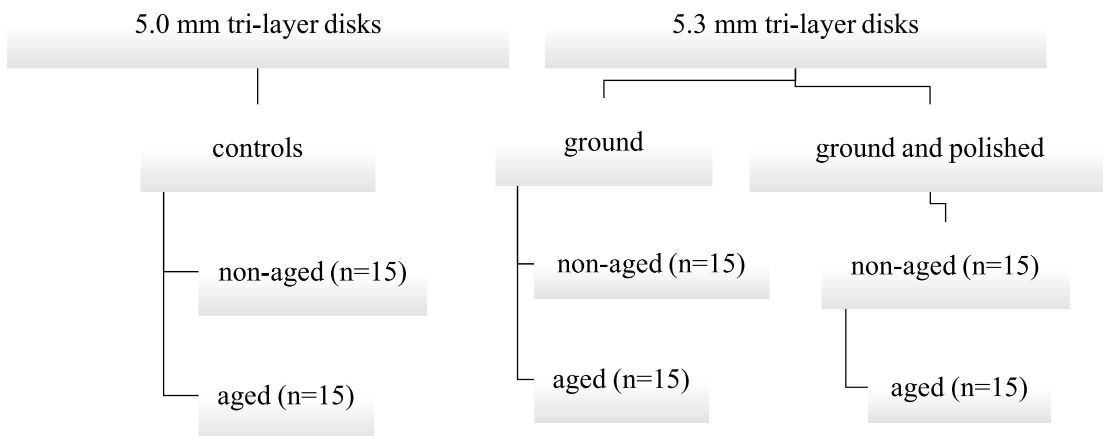
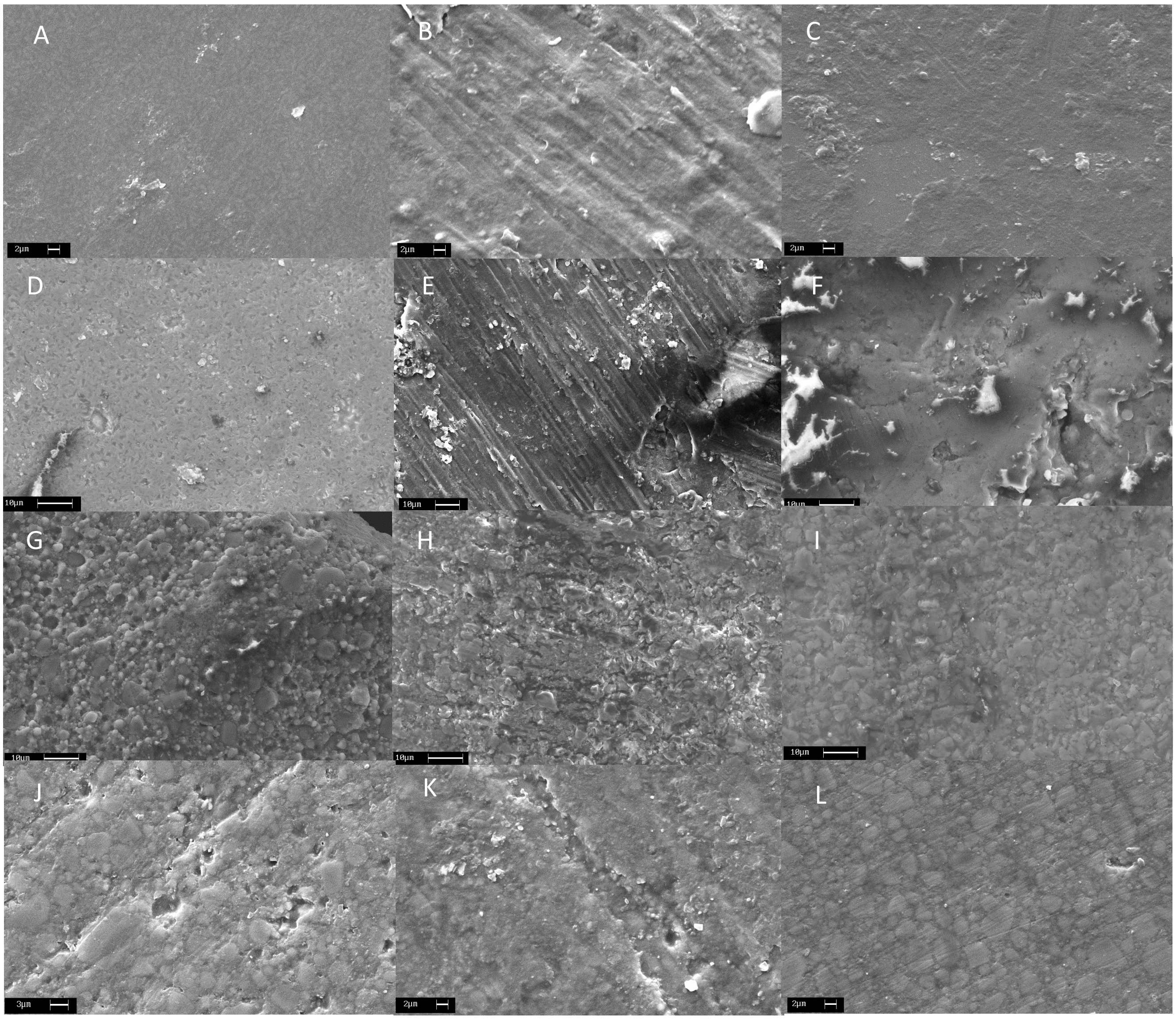


Figure 3. Scanning electron microscope images (original magnification ×3000) of control, ground, and polished groups. A-C, IPS e.max CAD; D-F, Vita Suprinity; G-I, Vita Enamic; J-L, Lava Ultimate.



TABLES

Table 1. Materials used

Materials (Manufacturer)	Composition	Type of block
IPS e.max CAD (Ivoclar Vivadent AG)	Lithium disilicate	HT A2 / C14
Vita Suprinity (Vita Zahnfabrik)	Glass ceramic reinforced with zirconium dioxide	A2-HT LS-14
Vita Enamic (Vita Zahnfabrik)	Hybrid Ceramic with resin polymers	2M2 – HT – EM-14
Lava Ultimate (3M ESPE)	Nanoceramic resin	A1-LT/14L

Table 2. Roughness: Mixed repeated-measures ANOVA

	SS	df Num	SQ Error	df Den	F	*P*
Intercept	215.204	1	1.3454	28	4478.887	<.001
Material	18.484	3	1.3454	28	128.234	<.001
Treatment	20.993	2	2.5833	56	227.542	<.001
Material×treatment	21.435	6	2.5833	56	77.442	<.001

Table 3. Roughness results in log mean (Rq) ±standard error

	Control	Ground	Polished
IPS e.max CAD	-3.242 ±0.077 bC	-969 ±0.076 bcA	-1.575 ±0.077 bB
Vita Suprinity	-3.025 ±0.077 bC	-1.213 ±0.076 cA	-1.584 ±0.077 bB
Lava Ultimate	-1.013 ±0.077 aB	-0.612 ±0.076 aA	-1.699 ±0.077 bC
Vita Enamic	-0.934 ±0.077 aA	-0.844 ±0.076 abA	-1.257 ±0.077 aB

Multiple comparisons of log averages (Rq) of materials and treatments. Different lowercase letters in columns indicate statistical differences (*P*<.05). Uppercase letters indicate statistical differences between treatments.

Table 4. Fracture resistance: 3-way ANOVA

Source	SS	df	MS	F	*P*
Corrected Model	7972417.464	23	346626.846	12.892	<.001
Intercept	563728169.669	1	563728169.669	20966.519	<.001
Material	2195372.097	3	731790.699	27.217	<.001
Treatment	1603381.739	2	801690.869	29.817	<.001
Aging	1147967.336	1	1147967.336	42.696	<.001
Material×treatment	379869.661	6	63311.610	2.355	.031
Material×aging	1295300.119	3	431766.706	16.059	<.001
Treatment×aging	558413.706	2	279206.853	10.384	<.001
Material×treatment×aging	792112.806	6	132018.801	4.910	<.001
Error	9034053.867	336	26887.065		
Total	580734641.000	360			
Corrected Total	17006471.331	359			

Table 5. Compression test results in N ±standard deviation and statistical comparisons

IPS e.max CAD/control/aged	1670 ±115	A
Vita Suprinity/control/aged	1481 ±225	AB
IPS e.max CAD/ground/non-aged	1455 ±127	ABC
Vita Suprinity/ground/aged	1405 ±232	BCD
IPS e.max CAD/ground/aged	1396 ±178	BCD
Vita Enamic/control/aged	1316 ±186	BCDE
IPS e.max CAD/control/non-aged	1286 ±112	BCDEF
Vita Enamic/ground/non-aged	1284 ±150	BCDEF
Vita Suprinity/polished/aged	1276 ±252	BCDEF
IPS e.max CAD/polished/non-aged	1264 ±110	BCDEFG
Lava Ultimate/control/aged	1263 ±191	BCDEFG
Vita Enamic/control/non-aged	1260 ±149	CDEFG
Vita Enamic/polished/aged	1254 ±191	CDEFG
Lava Ultimate/ground/non-aged	1198 ±186	DEFG
IPS e.max CAD/polished/aged	1187 ±172	DEFGH
Vita Suprinity/control/non-aged	1166 ±114	EFGH
Lava Ultimate/control/non-aged	1164 ±110	EFGH
Vita Enamic/polished/non-aged	1160 ±197	EFGH
Vita Enamic/ground/aged	1154 ±227	EFGH
Lava Ultimate/polished/aged	1145 ±112	EFGH
Lava Ultimate/ground/aged	1145 ±138	EFGH
Vita Suprinity/ground/non-aged	1077 ±81	FGH
Lava Ultimate/polished/non-aged	1053 ±86	GH
Vita Suprinity/polished/non-aged	972 ±103	H

Table 6. Weibull modulus and 90% confidence interval

		Control	Ground	Polished
IPS e.max CAD	non-aged	12.4 (7.2 – 17.4)	13.5 (7.8 – 18.9)	11.9 (6.9 – 16.6)
	aged	17.1 (9.9 – 23.9)	8.9 (5.1 – 12.4)	8.1 (4.7 – 11.3)
Vita Suprinity	non-aged	11.7 (6.8 – 16.4)	15.2 (8.8 – 21.3)	11.1 (6.4 – 15.5)
	aged	7.6 (4.4 – 10.6)	6.8 (3.9 – 9.5)	5.8 (3.3 – 8.1)
Vita Enamic	non-aged	9.0 (5.2 – 12.6)	9.5 (5.5 – 13.2)	6.1 (3.5 – 8.5)
	aged	8.5 (4.9 – 11.9)	5.7 (3.3 – 8.0)	7.7 (4.4 – 10.7)
Lava Ultimate	non-aged	12.5 (7.2 – 17.5)	7.3 (4.2 – 10.2)	14.6 (8.5 – 20.5)
	aged	7.8 (4.5 – 11.0)	9.7 (5.6 – 13.6)	12.0 (6.9 – 16.8)

Weibull modulus not influenced by material, surface treatment, or aging.

Final Considerations

4 Final considerations

In the first part of this thesis, we evaluated the 2-body wear produced by CAD-CAM monolithic materials on bovine enamel and on themselves.

In general, Lava Plus and IPS e.max CAD wore the antagonists more than did Vita Enamic and Lava Ultimate, and no significant difference was found in wear potential, either between IPS e.max CAD and Suprinity (except for Lava Ultimate antagonist) or between Vita Enamic and Lava Ultimate. In particular, we observed that the materials with higher hardness than that of enamel, presented higher enamel wear than those with lower hardness. It was also observed that a relationship between roughness, coefficient of friction, and wear behavior was not occurring as Lava Plus and IPS e.max CAD exhibited significantly higher wear potential but lower roughness than Vita Enamic and Lava Ultimate and a lower coefficient of friction than the Lava Ultimate.

During cyclic sliding, some materials may undergo changes in surface topography, which might modify their abrasiveness, in fact, the presence of material debris between the rubbing surfaces might increase the friction and accordingly the wear rate, both from the glass ceramic and enamel.

Therefore, some materials may have their surface roughness and coefficient of friction changed during the wear process, explaining the poor relationship between these 2 properties and wear potential.

These statements agree only partially to the some of the initial considerations, where roughness, coefficient of friction and hardness seemed to play a role in material wear potential. This could be due to the different methodology used in this study, which allowed us to test different materials at the same time. Indeed, it is realistic to assume that what occurred “in vitro”, which is that materials superficial properties change during their test, befall “in vivo”, which is that, after years of usage, a prosthetic material roughness will be different than when it was cemented at the beginning. For what concern hardness, this is an intrinsic property for ceramic materials, which does not change over time. It could be suggested to recall patients periodically to polish their prosthetic artefacts to maintain surface roughness over time.

A limitation of this study was that the enamel was not used as abrader (not possible with the methodology used); therefore, qualitative and quantitative information about its potential to wear itself and the evaluated antagonist materials is lacking.

In the second part of this thesis, we investigated if occlusal adjustment and classical polishing protocol affect initial material roughness of CAD-CAM monolithic materials, if those protocols and mechanical aging influences materials flexural strength, and if the studied materials reliability is maintained after those treatments (Weibull Modulus).

Regarding roughness, for both glass-ceramics, the polishing after grinding was not able to recover the smoothness given by the glaze, while for the Lava Ultimate and Vita Enamic, a higher smoothness was achieved by polishing after grinding, showing that even though these two materials do not require clinical adjustments, they should be polished to obtain a smooth surface.

In the present study, grinding and polishing affected negatively the fracture resistance of the IPS e.max CAD only in the aged groups. However, this behavior was due to the expressive increase in IPS e.max CAD/control/aged group fracture resistance in comparison with the IPS e.max CAD/control/non aged group, for which we find no explanation in the related literature.

Vita Suprinity showed an increase in the fracture resistance after aging, regardless of the surface treatment condition. It was a surprise since it was expected that the aging would reduce the fracture resistance. Also, it was found that Vita Suprinity presents significantly higher flexural strength than the IPS e.max CAD.

Regarding the maintenance of the fracture resistance of the Vita Enamic and Lava Ultimate, even after grinding and mechanical aging, due to a combination of lower hardness and lower elastic modulus, these materials present a high resistance to crack initiation and crack growth, with no estimated potential loss in their strength.

In our study, thanks to the great mechanical properties of the studied materials, confirmed by the Weibull modulus test, neither mechanical aging nor grinding procedures were able to influence the tested materials reliability. As a matter of facts, both the resin based and the glass-ceramics materials investigated in this study are made to be reliable and to resist in mouth, which is a hostile environment. It can be supposed that a longer aging time is necessary to decrease mechanical properties of these materials.

A limitation of this study was that evaluations were performed in flat specimens, since it does not closely reproduce what may happen in the oral cavity. Moreover, different aging methods and diamond burs may be required to reach a clearer image of the behavior of the studied materials submitted to clinical adjustments.

Conclusion

5 Conclusion

- The nanofilled composite resin and polymer-infiltrated ceramic were the most antagonist-friendly materials when sliding against enamel and any other material.
- Lithium disilicate, zirconia-reinforced lithium silicate, and zirconia caused high wear rates on the enamel and materials, with the difference that zirconia did not damage the surface of the materials, except for the enamel.
- Hardness should be considered in the selection of materials, especially in patients with bruxism.
- The grinding increased the roughness of the materials, except for the Vita Enamic. The smoothness of the glazed glass-ceramics was not achievable by the common polishing kits, while the Lava Ultimate and Vita Enamic showed a smoother surface compared with the baseline.
- The grinding followed or not by polishing did not affect adversely the fracture resistance of the materials.
- Mechanical aging did not reduce the fracture resistance of the materials, not even in the ground groups.
- The reliability of the evaluated materials under the scenario of the current study was not influenced by the material, surface treatment or aging.

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Attachment 1
First page manuscript 1

7 First page manuscript 1

THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

Wear resistance and abrasiveness of CAD-CAM monolithic materials

Francesco Saverio Ludovichetti, DDS,[a] Flávia Zardo Trindade, DDS, MSc, PhD,[b] Arie Werner, Ing,[c] Cornelis Johannes Kleverlaan, PhD,[d] and Renata Garcia Fonseca, DDS, MSc, PhD[e]

ABSTRACT

Statement of problem. Computer-aided design and computer-aided manufacturing (CAD-CAM) restorations are in contact with the antagonist tooth, either a natural tooth or a restoration. Therefore, clinicians should be aware of the wear resistance of CAD-CAM materials and the wear behavior of the antagonist.

Purpose. The purpose of this in vitro study was to evaluate the wear resistance and abrasiveness of CAD-CAM materials.

Material and methods. In a 2-body wear test, the materials IPS e.max CAD (Ivoclar Vivadent AG), Vita Suprinity (Vita Zahnfabrik), Lava Ultimate (3M ESPE), Vita Enamic (Vita Zahnfabrik), and Lava Plus (3M ESPE) acted as abraders and, together with bovine enamel, also as antagonists. Each antagonist wheel ran against each abrader wheel for 200 000 cycles, with a spring force of 15 N, and at a rotational speed of 1 Hz in distilled water. The wear rate was determined with a surface profilometer. The surfaces were observed with scanning electron microscopy, and their hardness, coefficient of friction, and roughness were evaluated.

Results. Lava Plus and IPS e.max CAD exhibited the highest potential for wear of Lava Ultimate. These 2 materials, together with Vita Suprinity, provided the highest wear of enamel and Vita Enamic. Vita Suprinity and IPS e.max CAD had higher wear than Lava Plus, and the inverse was also true. Vita Enamic and Lava Ultimate were among the materials that caused the lowest wear of enamel and all other evaluated materials. Scanning electron microscopy images revealed that except for Lava Ultimate, all other materials damaged enamel, in which Vita Suprinity and IPS e.max CAD were more aggressive when sliding against the materials. Lava Plus had the greatest hardness, followed by Vita Suprinity and IPS e.max CAD, Vita Enamic, and then Lava Ultimate. The coefficient of friction varied from 0.42 to 0.53. The Vita Enamic and Lava Ultimate showed the highest surface roughness.

Conclusions. The nanofilled composite resin and polymer-infiltrated ceramic were more antagonist-friendly (whether enamel or CAD-CAM material) than glass-ceramics and zirconia. Care should be taken when selecting the material that will contact mainly with glass-ceramics. Hardness should also be considered when selecting a material. (J Prosthet Dent 2018;120:318.e1-e8)

An improvement in the esthetics of lithium disilicate and yttria-stabilized tetragonal zirconia ceramics has led to monolithic restorations, eliminating the problem of fracture and chipping of veneering porcelain.[1] In addition, the advent of computer-aided design and computer-aided manufacturing (CAD-CAM) technology has enabled restorations to be provided in a single session.[2] More recently, other esthetic CAD-CAM monolithic materials with similar indications as lithium disilicate and yttria-stabilized tetragonal zirconia ceramic have been introduced. These include zirconia-reinforced lithium silicate ceramic, polymer-infiltrated ceramic, and nanofilled composite resin.

In the absence of veneering porcelain, these materials are in contact with the antagonist, which can be a natural tooth or

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Attachment 2
First page manuscript 2

8 First page manuscript 2

The effect of grinding and polishing on the roughness and fracture resistance of cemented CAD/CAM monolithic materials submitted to mechanical aging

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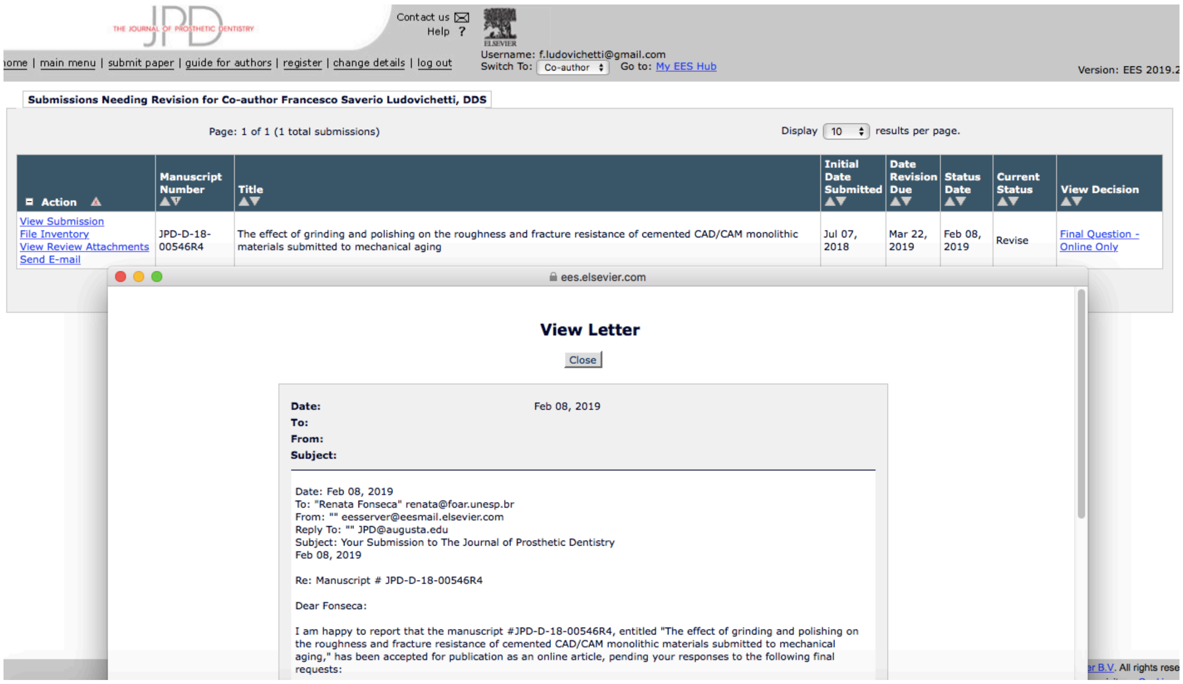
9 Manuscript 2 – Accepted



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