




## Article

# Toothbrushing Wear Resistance of Stained CAD/CAM Ceramics

Amanda Maria de Oliveira Dal Piva <sup>1</sup> , Marco Antonio Bottino <sup>1</sup>, Lilian Costa Anami <sup>1</sup>, Arie Werner <sup>2</sup>, Cornelis Johannes Kleverlaan <sup>2</sup>, Roberto Lo Giudice <sup>3,\*</sup> , Fausto Famà <sup>3</sup>, Laís Regiane da Silva-Concilio <sup>4</sup> and João Paulo Mendes Tribst <sup>4</sup> 

- <sup>1</sup> Post-Graduate Program in Restorative Dentistry (Prosthodontic), Department of Dental Materials and Prosthodontics, Institute of Science and Technology, São Paulo State University (Unesp/SJC), Engenheiro Francisco José Longo Avenue 777, São José Dos Campos 12245-000, Brazil; amodalpiva@gmail.com (A.M.d.O.D.P.); marco.bottino@unesp.br (M.A.B.); lianami@gmail.com (L.C.A.)
- <sup>2</sup> Department of Dental Materials Science, Academic Centre for Dentistry Amsterdam (ACTA), Universiteit van Amsterdam and Vrije Universiteit. Gustav Mahlerlaan #3004, 1081 Amsterdam, The Netherlands; a.werner@acta.nl (A.W.); c.kleverlaan@acta.nl (C.J.K.)
- <sup>3</sup> Department of Human Pathology in Adulthood and Childhood “G. Barresi”, University Hospital “G. Martino” of Messina, Via Consolare Valeria 1, 98125 Messina, Italy; fausto.fama@unime.it
- <sup>4</sup> Department of Dentistry, Postgraduate Program in Dentistry, University of Taubaté (UNITAU), Operários Street 09, São Paulo 12020-340, Brazil; regiane1@yahoo.com (L.R.d.S.-C.); joao.tribst@gmail.com (J.P.M.T.)
- \* Correspondence: roberto.logiudice@unime.it; Tel.: +39-3934399197



**Citation:** Dal Piva, A.M.d.O.; Bottino, M.A.; Anami, L.C.; Werner, A.; Kleverlaan, C.J.; Lo Giudice, R.; Famà, F.; Silva-Concilio, L.R.d.; Tribst, J.P.M. Toothbrushing Wear Resistance of Stained CAD/CAM Ceramics. *Coatings* **2021**, *11*, 224. <https://doi.org/10.3390/coatings11020224>

Academic Editor:  
Eugenio Velasco-Ortega

Received: 19 January 2021  
Accepted: 10 February 2021  
Published: 13 February 2021

**Publisher’s Note:** MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

**Abstract:** Aim: To investigate the effect of toothbrushing on different stained Computer Aided Design/Computer Aided Manufacturing CAD/CAM ceramics. Materials and Methods: Fifty specimens (high translucency zirconia, YZHT; zirconia-reinforced lithium silicate, ZLS; feldspathic, FDL; hybrid ceramic, HC; all from Vita Zahnfabrik) were divided into five groups according to their staining technique. ZLS allowed the composition of two groups: ZLS1 with crystallization and staining together; ZLS2, with the stain firing after the crystallization. YZHT received a stain firing after the sintering process, and FDL received stain firing directly. The HC was stained with acrylic staining, and was light-cured on its surface. The specimens were brushed in total for 150,000 cycles at 2.45 N with 180 strokes/min. Surface measurements to obtain Rz were performed after 50,000, 100,000 and 150,000 cycles, with five evaluation lines (5 mm) per specimen orthogonal to the brushing direction, covering brushed and unbrushed areas. The wear was analyzed using two-way ANOVA and Tukey tests ( $\alpha = 5\%$ ). Scanning electron microscopy (SEM) was performed to access the surface profile. The wear was affected by material ( $p < 0.001$ ) and time ( $p = 0.139$ ). Superior wear ( $\mu\text{m}$ ) was observed for HC ( $6.6 \pm 4.4$ ;  $6.8 \pm 4.0$ ;  $9.2 \pm 3.5$ ) compared to ZLS2 ( $1.0 \pm 0.3$ ;  $1.2 \pm 2.2$ ;  $1.3 \pm 0.3$ ), YZHT ( $1.0 \pm 0.1$ ;  $1.2 \pm 0.3$ ;  $1.2 \pm 0.3$ ), ZLS1 ( $0.9 \pm 0.1$ ;  $1.1 \pm 0.5$ ;  $1.2 \pm 0.3$ ) and FDL ( $0.9 \pm 0.1$ ;  $0.9 \pm 0.1$ ;  $1.0 \pm 0.2$ ) after 5, 10 and 15 years of simulation, respectively. SEM showed different wear patterns for HC with the removal of the glaze layer. HC showed a higher staining wear rate compared to the glass-based and polycrystalline ceramics after 15 years. The extrinsic characterization of feldspathic ceramic showed its superior longevity compared to the evaluated high-translucency zirconia, zirconia-reinforced lithium silicate, and hybrid ceramic.

**Keywords:** dental ceramics; toothbrushing; wear depth; friction and wear; dental materials

## 1. Introduction

Computer-aided design/computer-aided manufacturing (CAD/CAM) technology allows different ceramic materials to be machined and indicated for the manufacturing of indirect dental restorations [1]. Ceramic blocks for digital workflow are available in different colors. However, monolithic restorations without further processing do not meet high aesthetic demands, and therefore, are more often used in the posterior region [2,3]. The staining of ceramic restorations is a common procedure used to mimic the nuances

and colors of natural teeth [4–6]. To improve the aesthetic limitations of glass-based and polycrystalline ceramics, stain firing was performed on this materials [5]. However, for hybrid ceramics, which present a polymeric matrix associated with an inorganic ceramic matrix [7,8], the staining procedure involves a surface treatment and the photoactivation of the stain material [3].

The staining or extrinsic characterization is applied at the end of the restoration manufacturing process, and is described as the superficial application of stains to the outermost ceramic layer [2]. This procedure has been reported as a routine technique for restorations made in ceramics with different composition, e.g., glass-based ceramic [9], reinforced glass ceramic [2], polycrystalline ceramic [10] or polymer infiltrated ceramic [3]. Unfortunately, the stain layer can be removed by deteriorating processes [2,4,5], which can cause discomfort for the patient due to the perception of unsatisfactory aesthetics [11]. There are no reports in the literature that demonstrate the wear rate of the extrinsic stain layer applied on different ceramics. Therefore, the information of which ceramic material allows the maintenance of the staining layer for a longer period could help the clinicians to plan which material should be used in cases of great aesthetic demand.

Toothbrushing is the most common oral hygiene method. This procedure can be simulated in vitro to demonstrate how the dental tissues [12], direct materials [13], indirect materials [14] and staining layer [2,3,9] could behave in the long term. The literature is concise in showing that toothbrushing has an effect on the extrinsic staining layer [2,3,9]. However, there are no investigations on the effects of toothbrushing on the staining surface wear of different CAD/CAM ceramics. Therefore, this study aimed to evaluate the wear of different CAD/CAM ceramics extrinsically stained after 5, 10 and 15 years of simulated toothbrushing. The null hypothesis was that no difference would exist between the stain wearing of different materials.

## 2. Materials and Methods

### 2.1. Specimen Preparation

Fifty specimens ( $n = 10$  per group) with standard dimension of 10 (length)  $\times$  8 (thickness)  $\times$  6 (height) mm<sup>3</sup> were obtained from five ceramic blocks from the same manufacturer (Vita Zahnfabrik, Bad Säckingen, Germany). The materials' information and compositions are summarized in Table 1. The blocks were cut using a precision cutting machine (Isomet<sup>®</sup> 1000, Precision Sectioning Saw, Buehler, Lake Bluff, IL, USA) with a diamond disc (Series 15LC Diamond Blade wafering, Buehler, Lake Bluff, IL, USA) under constant water cooling. Next, the specimens were polished under water cooling using an orbital polishing machine with silicon carbide papers grits up to P1200 [15]. The specimens were cleaned in an ultrasonic bath with isopropyl alcohol for 5 min, and then ZLS2 was subjected to crystallization firing and zirconia was subjected to sinterization firing, according to the manufacturer's recommendations using specific ovens.

**Table 1.** Materials information and staining techniques.

Brand Name/Material	Composition
High-translucency zirconia. Vita YZ HT block, Vita Zahnfabrik, Bad Säckingen, Germany. Batch 46243. YZHT	(90.9–94.5% of ZrO <sub>2</sub> , 4–6% of Y <sub>2</sub> O <sub>3</sub> , 1.5–2.5 of HfO <sub>2</sub> , 0–0.3 of Al <sub>2</sub> O <sub>3</sub> , 0–0.5 of Er <sub>2</sub> O <sub>3</sub> and 0–0.3 of Fe <sub>2</sub> O <sub>3</sub> )
Feldspathic ceramic. Vitablocs Mark II, Vita Zahnfabrik. Batch 57370. FLD	(20–23% of Al <sub>2</sub> O <sub>3</sub> , 6–9% of Na <sub>2</sub> O, 6–8% of K <sub>2</sub> O, 0.01% of TiO <sub>2</sub> , 56–64% of SiO <sub>2</sub> , 0.3–0.6% of CaO)
Zirconia-reinforced lithium silicate based ceramic. Vita Suprinity, Vita Zahnfabrik. Batch 57370. ZLS1 and ZLS2	(56–64% of SiO <sub>2</sub> , 15–21% of Li <sub>2</sub> O, 1–4% of K <sub>2</sub> O, 3–8% of P <sub>2</sub> O <sub>5</sub> , 1–4% of Al <sub>2</sub> O <sub>3</sub> , 0–4% of CeO <sub>2</sub> , 0–6% of pigments and 10% of ZrO <sub>2</sub> )
Hybrid ceramic. Vita Enamic, Vita Zahnfabrik. Batch 74750. HC	(58–63% of SiO <sub>2</sub> , 20–23% of Al <sub>2</sub> O <sub>3</sub> , 6–11% of Na <sub>2</sub> O, 4–6% of K <sub>2</sub> O, 0.5–2% of B <sub>2</sub> O <sub>3</sub> , <1% of CaO and <1% of TiO <sub>2</sub> ).

Table 1. Cont.

Brand Name/Material	Composition
Vita Akzent® Plus	Effect Stains ES14 (52–68% of silicon dioxide, 4–6% of sodium oxide, 6–7% of aluminum oxide, 3–4% of potassium oxide, 4–5% of calcium oxide, 9–11% of boron trioxide, 1–2% of barium oxide, 3–4% of tin dioxide, 2–4% of zirconium dioxide, <1% of zinc dioxide, <1% of titanium dioxide, <1% of cerium (IV) dioxide, <1% of magnesium oxide and <1% of iron (III) oxide; Batch 60840).
	Power Fluid (Polyhydric alcohol; Batch 79641).
Vita Enamic Stains®	Stain (Silicon dioxide, Benzoyl peroxide, Titanium peroxide and Ferric oxide; Batch 32300)
	Stains Liquid (Aliphatic urethane dimethacrylate; Methyl methacrylate; Camphoroquinone; Phosphine oxide, dinphenyl-trimethylbenzoyl; Ethyl-4-dimethylaminobenzoate; Butylated hydroxytoluene; Batch 74180).

Information according to the manufacturer's data.

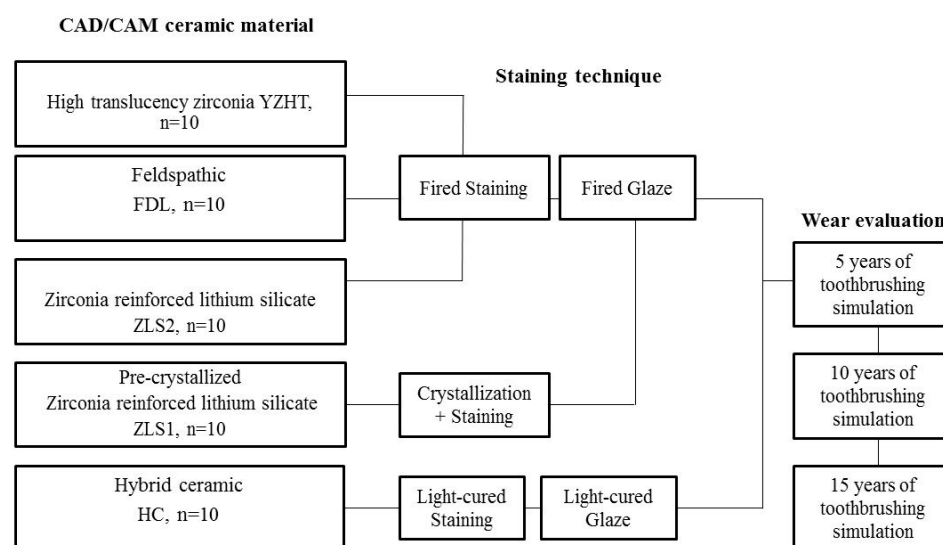
The polycrystalline YTZP HT was sintered in a ZYRCOMAT 6000 MS furnace (Vita Zahnfabrik, Bad Säckingen, Germany) at 1450 °C using the universal program, while the reinforced glass ceramic (Vita Suprinity, Vita Zahnfabrik) was crystallized in the Vacumat 40T furnace (Vita Zahnfabrik, Bad Säckingen, Germany). The crystallization firing schedule was the following: pre-dry: 400 °C; time at the initial temperature: 4 min; time for temperature elevation: 8 min; temperature elevation rate: 55 °C/min; maximum temperature: 840 °C; time at the maximum temperature: 8 min.

The low-fusing ceramic (Vita Akzent, Vita Zahnfabrik) was sintered in the Vacumat 40T furnace (Vita Zahnfabrik, Bad Säckingen, Germany). The stain fixation firing schedule was the following: initial temperature: 500 °C; time at the initial temperature: 4 min; time for temperature elevation: 3:15 min; temperature elevation rate: 80 °C/min; maximum temperature: 760 °C; time at the maximum temperature: 1 min. The glaze fixation firing schedule was the following: initial temperature: 400 °C; time at the initial temperature: 6 min; time for temperature elevation: 5:36 min; temperature elevation rate: 80 °C/min; maximum temperature: 850 °C; time at the maximum temperature: 1 min.

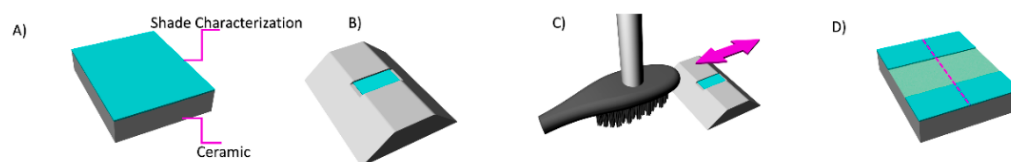
The groups distributions are presented in the flowchart (Figure 1). ZLS1 was submitted to crystallization, including stains, in one unique step, followed by the glaze application, whereas the HC surface, prior to staining, was treated with 5% hydrofluoric acid etching for 60 s (Condac Porcelana, FGM, Joinville, Brazil), cleaned (ultrasonic bath with distilled water for 10 min) and silanized (Vita Adiva® C-Prime, Vita Zahnfabrik, Bad Säckingen, Germany; Batch 82680). The stain layer was applied and light-cured for 30 s. After, the glaze was applied and light-cured for 60 s (800 mW/cm<sup>2</sup>). The average thickness for each material was YZHT (40.5 µm), FDL (33.5 µm), ZLS1 (26.7 µm), ZLS2 (34.4 µm) and HC (36.1 µm).

## 2.2. Specimen Preparation

Soft brushes (Slim Soft, Colgate-Palmolive Indústria e Comércio, 00779B) were coupled in the parallel arms of the brushing machine, perpendicular to the ceramic surface. Each reservoir was filled with a solution made of 250 g of low-abrasion (RDA = 63) toothpaste (Colgate Sensitive, Colgate-Palmolive Indústria e Comércio) suspended in 1 L of distilled water [16]. To restrict the wear in the ceramic center, a metallic device was used restricting the brushing area. The no-brushed sides were used as reference for the measurement of profile variation [17] (Figure 2). Each specimen was brushed (2.45 N, 80 strokes per min) for 50,000, 100,000, and 150,000 cycles, proportionally corresponding to 5, 10, and 15 years of toothbrushing in the oral environment [18]. Both brushes and solution were changed after each 5000 cycles.



**Figure 1.** Study flowchart shows four Computer-aided design/computer-aided manufacturing CAD/CAM ceramics submitted to staining procedures prior to the toothbrushing simulation.



**Figure 2.** Schematic illustration of toothbrushing simulation. (A) Extrinsically stained ceramic specimen. (B) Metallic device positioned to restrict the brushing area. (C) Brushing direction during the test. (D) Inspection path of the wear profile of brushed specimen.

### 2.3. Surface Topography Analysis

To measure the abrasive wear rate during the different brush intervals, the surface analysis was performed with a contact profilometer (SJ 400, Mitutoyo, Tokyo, Japan). Each specimen was analyzed in 5 random different areas at a speed of 0.2 mm/s [19], with a range of 5 mm, to reach the worn and not worn areas. The test parameter was the average of the 5 highest peaks and the average of the 5 deepest valleys (Rz). The analysis was performed following the Gaussian low-pass filter and the cut-off wavelength value was 0.8 mm [19].

Representative specimens from each group were ultrasonically cleaned in distilled water (5 min), gold sputtered, and analyzed under scanning electron microscopy (SEM) (Evo LS15, Oberkochen, Carl Zeiss, Germany) to identify the group's wear profile.

### 2.4. Statistical Analysis

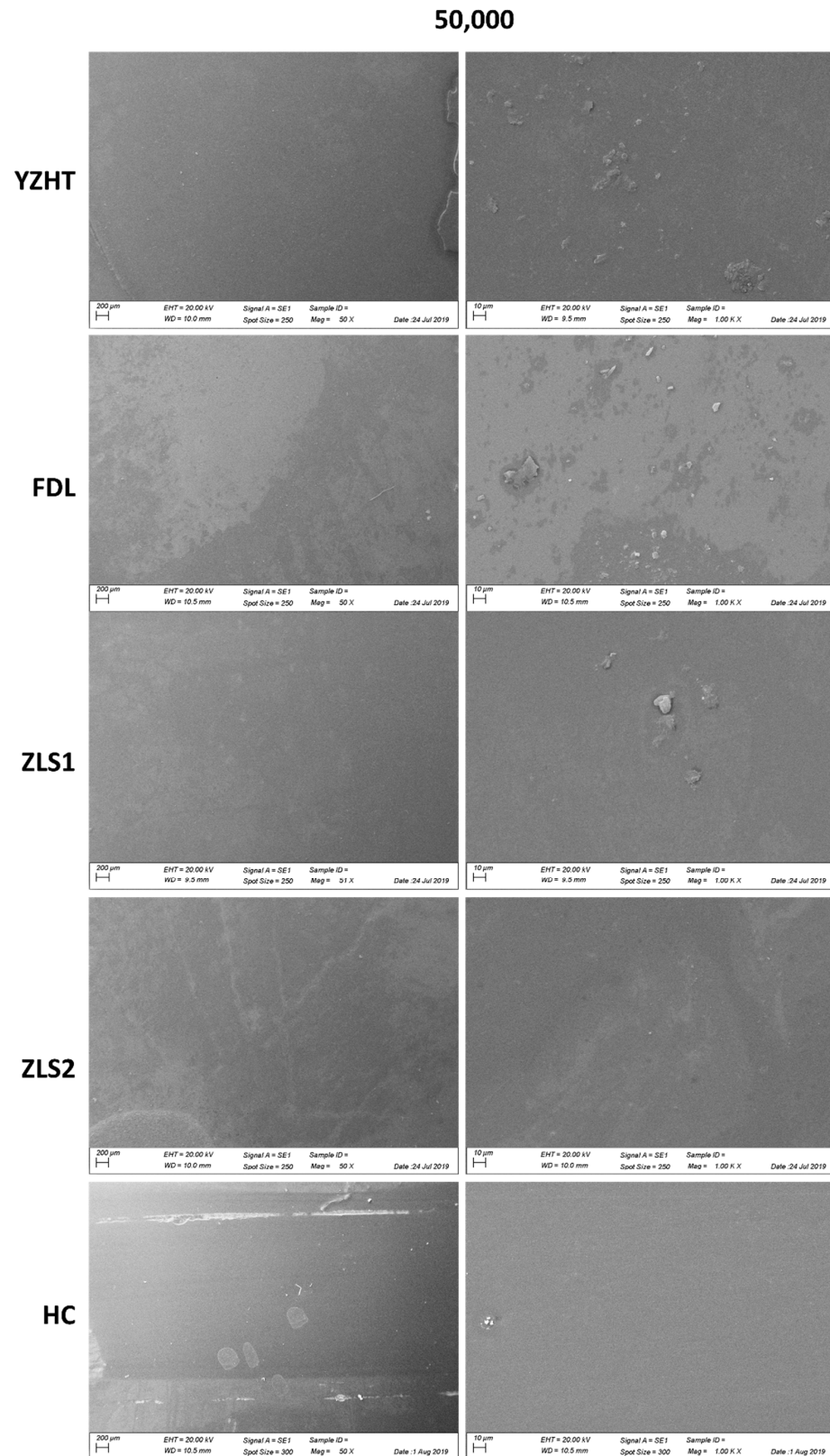
Two-way analysis of variance (ANOVA) and post-hoc Tukey tests were used to evaluate the wear rate (all with  $\alpha = 0.05$ ) according to the materials and number of cycles (time).

## 3. Results

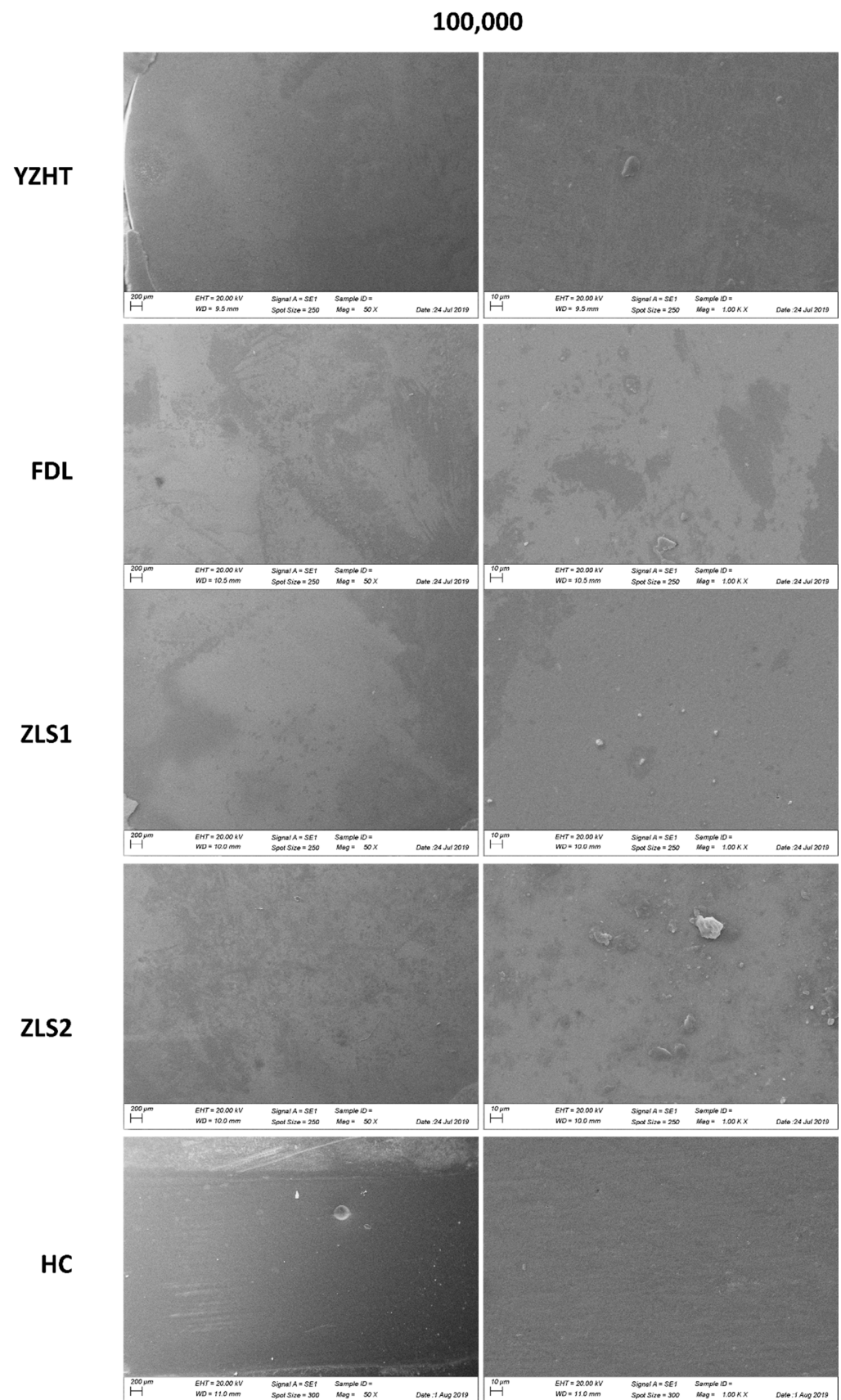
Two-way ANOVA (Table 2) showed the significant influence of the material on the abrasion wear rate. Observing only the material factor, HC showed a higher mean wear rate ( $7.5 \pm 4.0 \mu\text{m}$ ) than ZLS2 ( $1.2 \pm 0.3 \mu\text{m}$ ), YZHT ( $1.1 \pm 0.3 \mu\text{m}$ ), ZLS1 ( $1.1 \pm 0.3 \mu\text{m}$ ) and FDL ( $0.9 \pm 0.1 \mu\text{m}$ ). No difference was observed between the simulations of 5 ( $2.1 \pm 2.9 \mu\text{m}$ ), 10 ( $2.3 \pm 2.8 \mu\text{m}$ ) and 15 ( $2.8 \pm 3.5 \mu\text{m}$ ) years. Table 3 presents the descriptive statistical analysis, and shows the groupings according to the ceramic material and years of simulated toothbrushing on the staining surface wear. Scanning electron microscopy of surface topography revealed similar worn surface profiles for YZHT, FDL, ZLS1 and ZLS2, regardless of the time (5, 10 and 15 years). However, the HC group showed a higher wear rate, confirmed by the difference between the worn and the non-worn surface



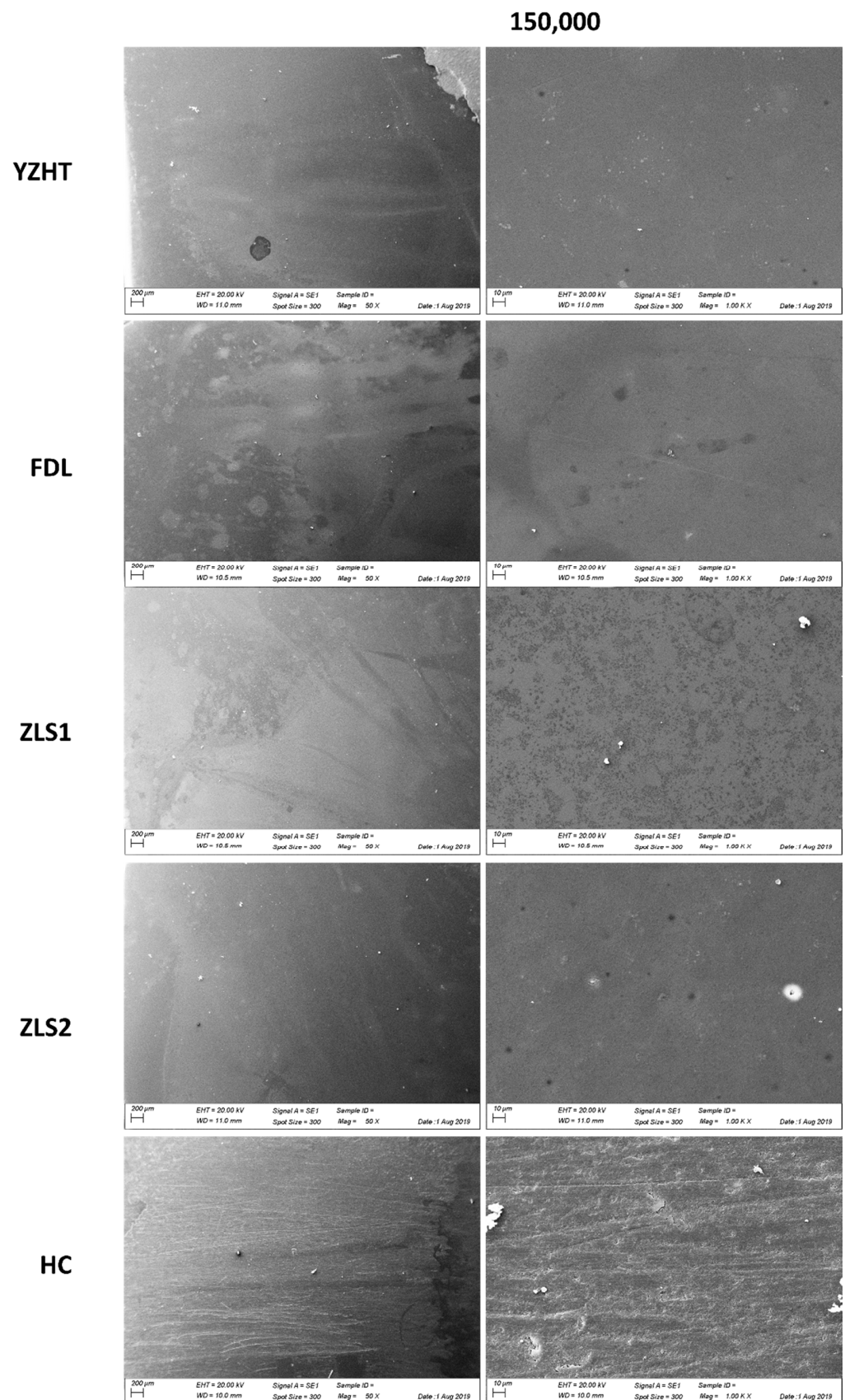
(Figures 3–5). The worn surface profiles have been evaluated between periods, and since the wear behavior was proportional, Figure 6 shows the representative wear profile after 15 years (the most aggressive profile) of simulated toothbrushing for each ceramic material.



**Figure 3.** Scanning electron microscopy of surface topography with 50× and 1000× magnification according to the ceramic material (YZHT, FDL, ZLS1, ZLS2 and HC) for 5 years of evaluation.



**Figure 4.** Scanning electron microscopy of surface topography with 50× and 1000× magnification according to the ceramic material (YZHT, FDL, ZLS1, ZLS2 and HC) for 10 years of evaluation.



**Figure 5.** Scanning electron microscopy of surface topography with 50× and 1000× magnification according to the ceramic material (YZHT, FDL, ZLS1, ZLS2 and HC) for 15 years of evaluation.



**Figure 6.** Surface profiles of representative specimens from according to the ceramic material (YZHT, FDL, ZLS1, ZLS2 and HC) after 15 years of toothbrushing simulation.

**Table 2.** Two-way ANOVA of abrasive wear according to the factors: ceramic material and time.

Source	DF	Adj SS	Adj MS	F-Value	p-Value
Ceramic	4	1001.89	250.472	75.90	<0.001
Time	2	13.23	6.617	2.01	0.139
Interaction	8	29.29	3.661	1.11	0.361
Error	135	445.52	3.300	-	-
Total	149	1489.94	-	-	-
Ceramic	4	1001.89	250.472	75.90	<0.001

Information according to the statistical analysis.

**Table 3.** Staining surface mean wear (Rz values in  $\mu\text{m}$ )  $\pm$  standard deviation and grouping according to the material and time (period of evaluation).

Group	5 Years	10 Years	15 Years
YZHT	$1.0 \pm 0.1^B$	$1.2 \pm 0.3^B$	$1.2 \pm 0.3^B$
FDL	$0.9 \pm 0.1^B$	$0.9 \pm 0.1^B$	$1.0 \pm 0.2^B$
ZLS1	$0.9 \pm 0.1^B$	$1.1 \pm 0.5^B$	$1.2 \pm 0.3^B$
ZLS2	$1.0 \pm 0.3^B$	$1.2 \pm 2.2^B$	$1.3 \pm 0.3^B$
HC	$6.6 \pm 4.4^A$	$6.8 \pm 4.0^A$	$9.2 \pm 3.5^A$

Information according to the statistical analysis. Similar capital letters correspond to no statistical difference.

#### 4. Discussion

The goal of this study was to investigate the effect of toothbrushing on different stained CAD/CAM ceramics. According to the results, the ceramic material influenced the extrinsic characterization of the wear. Based in this finding, the null hypothesis was rejected, because the wear rate of tooth brushing was significantly higher for hybrid ceramic (HC) compared to the other ceramics investigated.

With the increased use of monolithic restorations, the use of pigments also increases for shade corrections, and natural tooth color is mimicked through extrinsic staining [2]. In addition, materials with different compositions were developed to be used in the monolithic form, most of the time, for the same indication [20]. However, the extrinsic staining is exposed to the oral environment and can be worn by toothbrushing in the long term [9,21]. Most of the abrasive simulation using toothbrushing evaluated the surface roughness quality [2,3,18,22] instead of wear rate [21,23,24]. In addition, limited data are available regarding the effect of toothbrushing on the staining layer of CAD/CAM ceramics over time [3,9,18,21].

The results showed that the hybrid ceramic (HC) presented a significantly higher wear rate of the stain compared to the evaluated polycrystalline (high-translucency zirconia,



YZHT), feldspathic ceramic (FDL), and glass-based ceramic (zirconia-reinforced lithium silicate, ZLS). The mechanism to bond the stain layer on an HC is different from the staining firing used for YZHT, FDL and ZLS materials. The manufacturer's instructions consist of a surface treatment prior to the staining layer, whereas the staining layers for YZHT, FDL and ZLS are applied on a polished surface followed by staining firing [2]. Hydrofluoric acid etching followed by a silane layer was the surface treatment selected for HC in this study.

The surface treatment creates irregularities on the ceramic due to the glass matrix dissolution and resinous matrix exposure [25]. Because of the high amount of composite matrix in the HC's composition, the stain layer that is not suitable for firing with high temperatures should be light-cured [26]. Even though the HC showed the highest wear rate after 15 years of simulated toothbrushing, the possibility of reapplying this material inside the mouth should be investigated in further studies. This alternative could attenuate the disadvantage observed in this study, of using stained HC compared to the other ceramics. It is important to emphasize the novelty of this material composition in comparison with the other evaluated glass ceramics and zirconia. The hybrid ceramic (58–63% of  $\text{SiO}_2$ , 20–23% of  $\text{Al}_2\text{O}_3$ , 6–11% of  $\text{Na}_2\text{O}$ , 4–6% of  $\text{K}_2\text{O}$ , 0.5–2% of  $\text{B}_2\text{O}_3$ , <1% of  $\text{CaO}$  and <1% of  $\text{TiO}_2$ ) that contains a polymer infiltrated matrix has been developed to improve glass ceramics' (without reinforcement) mechanical properties, while maintaining the aesthetic properties [27]. Studies have observed the promising mechanical behavior of this material due to the polymer infiltration, which can dampen the stresses inside the material [28,29]. However, in terms of stain characterization durability against food bolus, there is some evidence of reduced wear resistance when compared to glass and polycrystalline ceramics [26,27]. The same pattern can be observed in the present results for the toothbrushing simulation. According to the literature, the surface roughness does not seem to be related to the staining durability; however, the bonding or union between ceramic and staining seems to be the determinant factor for staining longevity [27]. Therefore, the content of 58–63% of  $\text{SiO}_2$  in the hybrid ceramic is not enough to guarantee the strong adhesion with the methylmethacrylate stain layer, as occurs when a porcelain-fused staining layer is associated with the other ceramic materials. Further studies should be carried out to evaluate the polymeric stain layer's bond strength, and whether the polymerization shrinkage residual stress on the restoration's surface is relevant to this hypothesis.

For YZHT, FDL and ZLS (stained in one or two steps), no differences were observed in their staining layer abrasive wear rates, suggesting that, regardless of the material, all extrinsic characterization would remain in the restoration surface after 15 years. All these ceramics received the VITA AKZENT Plus stain kit that consists of feldspathic ceramic powder, butanediol and glycerin [3]. Additionally, after the staining firing, the ceramics received a glaze firing [30]. This study simulated two different staining techniques for the ZLS material. For the one-step firing (ZLS1), the specimens were crystallized and stained together [31,32] to accelerate the final color achievement, and for the conventional two-steps firing (ZLS2), the specimens were stained and then glazed. However, the influence of the staining technique on ZLS's properties has not been studied yet. The results show that both the staining techniques available for ZLS have the same wear rate under toothbrushing simulation. A previous study evaluated the effects of different staining techniques (stain and glaze separate or together) on the surface roughness and color change of a leucite-based and a lithium silicate glass ceramic. The authors observed that after 12 years of simulated toothbrushing, no difference was observed for the leucite-based ceramic, while for the lithium silicate, the surface roughness values were only different between the baseline and 3 years of toothbrushing [2]. The authors did not evaluate the abrasive wear rates of those materials. For that, the use of a roughness depth parameter is suggested in order to determine the wear rate based on the lateral no-brushed references [3,17,21].

The staining layer on the feldspathic ceramic is suggested to be removed after 10–12 years of simulated toothbrushing. This result could be improved if a glaze layer is applied over the stain, protecting it from wear [2,9,21,23,33]. A previous study evaluated the influence of load and brushing time on the surface roughness and gloss of a composite resin and



two leucite-based ceramics. The authors observed little or no deterioration on the ceramics' surface [34], which confirms the results of this study. However, the authors limited the toothbrushing time to 6 years. Other investigations also did not find any influence of toothbrushing on lithium disilicate surface roughness, microhardness or color stability after 5 (simulated) years [22]. However, the authors did not evaluate the stained specimens. Mühlemann et al. (2019) investigated the surface roughness of HC and FDL materials (using the same stain kits) after 5 years of toothbrushing simulation [3,35]. They found that the abrasion increased the surface roughness of HC, but not of the FDL material. Those findings suggest a tendency of HC towards deterioration, which was similar to the behavior observed by Flury et al. (2017) [36] and the present study. Based on this and other in vitro studies, it was shown that non-stained HC is more prone to degradation by artificial toothbrushing compared to FDL.

Yuan et al. (2018) [18] evaluated the influence of toothbrushing and/or thermocycling on the extrinsic staining color stability and surface roughness of a lithium disilicate and a zirconia. The authors simulated 5, 10 and 15 years of toothbrushing, and observed that for both materials, the color changes were below the clinically established perceptible level, and that lithium disilicate's average roughness increased over time, while this decreased for zirconia. The results did not show a surface modification sufficient to make the restoration unfeasible. The results present in this investigation are in agreement with the longitudinal clinical trials/reports that concluded no significant aesthetic modification for the restorations of ceramics, with a high percentage of success at six years [37].

The glaze available for the hybrid ceramic has low viscosity, contains methyl methacrylate, and should be light-cured on the ceramic surface. After 15 years of simulation, the glaze layer was removed, and this fact was confirmed after SEM analysis. However, for the other materials, the glaze layer is suggested as a protective barrier to avoid staining wear and color changes [2,21,38]. One previous paper evaluated the color stability on stained ZLS, and reinforced the importance of the glaze layer for color stability [38]. In terms of wear rate, a previous study performed three body-wear simulations in different stained dental ceramics [26]. The author concluded that the feldspar ceramic presented superior staining durability, and the least durable was the hybrid ceramic material. The present study corroborates this statement, and complements it by showing that even a less aggressive abrasion, such as toothbrushing, can negatively affect the hybrid ceramic stain durability. According to a previous report that evaluated the wear resistance of surface treatments prior to staining and glazing a hybrid ceramic, the use of acid etching is one of the most promising treatments [25,39]. Therefore, the present study simulated one of the most promising surface treatments for this material; however, conventional glass staining is still superior in terms of wear resistance.

The literature affirms that toothpaste abrasiveness is measured with relative dentin abrasivity (RDA), and previous studies have shown that toothbrushing can affect the porcelain restorations [2]. There are reports that used medium abrasive (70 RDA) toothpaste [2], and found that the glass ceramic shade characterization (Ra value) can be affected by toothbrushing simulation. Another report [3] used a slurry containing toothpaste with 100 RDA to simulate a total of 5 years of clinical service. The authors showed surface structural changes (SEM images) for the resin-ceramic CAD/CAM materials. The present study corroborates that, showing that even with a low abrasive toothpaste (63 RDA), some structural changes will be present after the same period of evaluation for a hybrid ceramic.

Another investigation [16] used a low-abrasiveness toothpaste during the same period of time of this study, and found that, for CAD/CAM ceramic materials, all mean surface roughness values were below 0.2 mm. However, the authors used the roughness average parameter (Ra), and did not fix an unworn reference, as in the present study. Therefore, the values cannot be compared between the present and the reported results; however, the general information that low-abrasiveness toothpaste can affect reinforced glass ceramic is common in both studies.

As far as the surface wear is concerned, a direct comparison between studies is difficult, since differences in the results are observed and can be justified by the use of different toothpaste slurries, loadings, numbers of strokes and toothbrush bristle hardness, and differences in specimen preparation and the quantification of the wear rate. As concerns the limitations of this study, we used a low-abrasion toothpaste and a soft brush, as is commonly suggested by clinicians for ordinary patients, due to the lower probability of causing damage [40–42]. In addition, the toothbrushing simulation did not consider all complex oral mediums that include pH variation, masticatory load, and saliva. The use of a harder brush and non-unidirectional movement could also influence the results. Despite these limitations, this investigation has the main purpose of informing the clinicians regarding the staining layer wear. In addition, a maintenance appointment should be performed to guarantee the restoration's success in the long term.

## 5. Conclusions

Within the limitations of this study, the following conclusion can be drawn: (i) the extrinsic staining wear caused by toothbrushing simulation is different for the hybrid ceramic compared to high-translucency zirconia, feldspathic and reinforced glass ceramic CAD/CAM materials; (ii) the hybrid ceramic showed a significantly higher wear rate, resulting in the disappearance of the glaze layer at less than 150,000 toothbrushing cycles (simulated 15 years), which was also confirmed by the SEM analysis of the specimens.

**Author Contributions:** Conceptualization, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K., R.L.G., L.R.d.S.-C. and J.P.M.T.; methodology, A.M.d.O.D.P., A.W., C.J.K. and J.P.M.T.; software, A.M.d.O.D.P., A.W., C.J.K. and J.P.M.T.; validation, A.M.d.O.D.P., A.W., C.J.K. and J.P.M.T.; formal analysis, A.M.d.O.D.P., A.W., C.J.K. and J.P.M.T.; investigation, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K., R.L.G., L.R.d.S.-C. and J.P.M.T.; resources, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K., R.L.G., L.R.d.S.-C. and J.P.M.T.; data curation, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K., R.L.G., L.R.d.S.-C. and J.P.M.T.; writing—original draft preparation, A.M.d.O.D.P., A.W., C.J.K. and J.P.M.T.; writing—review and editing, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K., R.L.G., L.R.d.S.-C. and J.P.M.T.; visualization, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K., R.L.G., L.R.d.S.-C. and J.P.M.T.; supervision, M.A.B. and C.J.K.; project administration, A.M.d.O.D.P., M.A.B., C.J.K. and J.P.M.T.; funding acquisition, A.M.d.O.D.P., M.A.B., L.C.A., A.W., C.J.K. and J.P.M.T. Resources, writing—review and editing, visualization and funding acquisition, F.F. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research was funded by São Paulo Research Foundation (FAPESP), grant number #2018/03984-5, #2018/05908-4, #2018/07404-3 and #2017/11535-3.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Acknowledgments:** The authors thank São Paulo Research Foundation (FAPESP) with the grants #2018/03984-5, #2018/05908-4, #2018/07404-3 and #2017/11535-3.

**Conflicts of Interest:** The authors declare no conflict of interest.

## References

1. Papadiochou, S.; Pissiotis, A.L. Marginal adaptation and CAD-CAM technology: A systematic review of restorative material and fabrication techniques. *J. Prosthet. Dent.* **2018**, *119*, 545–551. [[CrossRef](#)]
2. Garza, L.A.; Thompson, G.; Cho, S.H.; Berzins, D.W. Effect of toothbrushing on shade and surface roughness of extrinsically stained pressable ceramics. *J. Prosthet. Dent.* **2016**, *115*, 489–494. [[CrossRef](#)]
3. Mühlemann, S.; Bernini, J.M.; Sener, B.; Hämmerle, C.H.; Özcan, M. Effect of aging on stained monolithic resin-ceramic CAD/CAM materials: Quantitative and qualitative analysis of surface roughness. *J. Prosthodont.* **2019**, *28*, 563–571. [[CrossRef](#)] [[PubMed](#)]
4. Bativa, F.; Weiner, S.; Berendsen, P.; Vincent, G.R.; Ianzano, J.; Harris, W.T., Jr. The microscopic appearance and effect of toothbrushing on extrinsically stained metal-ceramic restorations. *J. Prosthet. Dent.* **1987**, *57*, 47–52. [[CrossRef](#)]
5. Anil, N.; Bolay, S. Effect of toothbrushing on the material loss, roughness, and color of intrinsically and extrinsically stained porcelain used in metal-ceramic restorations: An in vitro study. *Int. J. Prosthodont.* **2002**, *15*, 483–487. [[PubMed](#)]

6. Guess, P.C.; Selz, C.F.; Steinhart, Y.N.; Stampf, S.; Strub, J.R. Prospective clinical split-mouth study of pressed and CAD/CAM all-ceramic partial-coverage restorations: 7-year results. *Int. J. Prosthodont.* **2013**, *26*, 21–25. [\[CrossRef\]](#) [\[PubMed\]](#)
7. Chirumamilla, G.; Goldstein, C.E.; Lawson, N.C. A 2-year retrospective clinical study of enamic crowns performed in a private practice setting. *J. Esthet. Restor. Dent.* **2016**, *28*, 231–237. [\[CrossRef\]](#)
8. de Carvalho Ramos, N.; Campos, T.M.; de La Paz, I.S.; Machado, J.P.; Bottino, M.A.; Cesar, P.F.; De Melo, R.M. Microstructure characterization and SCG of newly engineered dental ceramics. *Dent. Mater.* **2016**, *32*, 870–878. [\[CrossRef\]](#)
9. Aker, D.A.; Aker, J.R.; Sorensen, S.E. Toothbrush abrasion of color-corrective porcelain stains applied to porcelain-fused-to-metal restorations. *J. Prosthet. Dent.* **1980**, *44*, 161–163. [\[CrossRef\]](#)
10. Souza, R.; Barbosa, F.; Araújo, G.; Miyashita, E.; Bottino, M.A.; Melo, R.; Zhang, Y. Ultrathin monolithic zirconia veneers: Reality or future? Report of a clinical case and one-year follow-up. *Oper. Dent.* **2018**, *43*, 3–11. [\[CrossRef\]](#) [\[PubMed\]](#)
11. Gordan, V.V.; Garvan, C.W.; Blaser, P.K.; Mondragon, E.; Mjör, I.A. A long-term evaluation of alternative treatments to replacement of resin-based composite restorations: Results of a seven-year study. *J. Am. Dent. Assoc.* **2009**, *140*, 1476–1484. [\[CrossRef\]](#)
12. Ionta, F.Q.; Dos Santos, N.M.; Mesquita, I.M.; Dionísio, E.J.; Cruvinel, T.; Honório, H.M.; Rios, D. Is the dentifrice containing calcium silicate, sodium phosphate, and fluoride able to protect enamel against chemical mechanical wear? An in situ/ex vivo study. *Clin. Oral Investig.* **2019**, *23*, 3713–3720. [\[CrossRef\]](#) [\[PubMed\]](#)
13. Augusto, M.G.; Borges, A.B.; Pucci, C.R.; Mailart, M.C.; Torres, C.R.G. Effect of whitening toothpastes on wear and roughness of ormocer and methacrylate-based composites. *Am. J. Dent.* **2018**, *31*, 303–308.
14. Azevedo, S.M.; Kantorski, K.Z.; Valandro, L.F.; Bottino, M.A.; Pavanelli, C.A. Effect of brushing with conventional versus whitening dentifrices on surface roughness and biofilm formation of dental ceramics. *Gen. Dent.* **2012**, *60*, e123–e130. [\[PubMed\]](#)
15. Tribst, J.; Anami, L.C.; Özcan, M.; Bottino, M.A.; Melo, R.M.; Saavedra, G. Self-etching primers vs. acid conditioning: Impact on bond strength between ceramics and resin cement. *Oper. Dent.* **2018**, *43*, 372–379. [\[CrossRef\]](#)
16. Rosentritt, M.; Sawaljanow, A.; Behr, M.; Kolbeck, C.; Preis, V. Effect of tooth brush abrasion and thermo-mechanical loading on direct and indirect veneer restorations. *Clin. Oral Investig.* **2015**, *19*, 53–60. [\[CrossRef\]](#)
17. Vieira, A.; Overweg, E.; Ruben, J.L.; Huysmans, M.C. Toothbrush abrasion, simulated tongue friction and attrition of eroded bovine enamel in vitro. *J. Dent.* **2006**, *34*, 336–342. [\[CrossRef\]](#)
18. Yuan, J.C.; Barão, V.A.R.; Wee, A.G.; Alfaro, M.F.; Afshari, F.S.; Sukotjo, C. Effect of brushing and thermocycling on the shade and surface roughness of CAD-CAM ceramic restorations. *J. Prosthet. Dent.* **2018**, *119*, 1000–1006. [\[CrossRef\]](#)
19. Dal Piva, A.; Contreras, L.; Ribeiro, F.C.; Anami, L.C.; Camargo, S.; Jorge, A.; Bottino, M.A. Monolithic ceramics: Effect of finishing techniques on surface properties, bacterial adhesion and cell viability. *Oper. Dent.* **2018**, *43*, 315–325. [\[CrossRef\]](#)
20. Dal Piva, A.M.O.; Tribst, J.P.M.; Borges, A.L.S.; Souza, R.O.A.E.; Bottino, M.A. CAD-FEA modeling and analysis of different full crown monolithic restorations. *Dent. Mater.* **2018**, *34*, 1342–1350. [\[CrossRef\]](#) [\[PubMed\]](#)
21. Chi, W.J.; Browning, W.; Looney, S.; Mackert, J.R.; Windhorn, R.J.; Rueggeberg, F. Resistance to abrasion of extrinsic porcelain esthetic characterization techniques. *US Army Med. Dep. J.* **2017**, *2*, 71–79.
22. Alencar-Silva, F.J.; Barreto, J.O.; Negreiros, W.A.; Silva, P.G.B.; Pinto-Fiamengui, L.M.S.; Regis, R.R. Effect of beverage solutions and toothbrushing on the surface roughness, microhardness, and color stainability of a vitreous CAD-CAM lithium disilicate ceramic. *J. Prosthet. Dent.* **2019**, *121*, 711.e1–711.e6. [\[CrossRef\]](#) [\[PubMed\]](#)
23. Ishikiriyama, S.K.; Ordonez-Aguilera, J.F.; Maenosono, R.M.; Volú, F.L.A.; Mondelli, R.F.L. Surface roughness and wear of resin cements after toothbrush abrasion. *Braz. Oral Res.* **2015**, *29*, 1–5. [\[CrossRef\]](#)
24. Yin, R.; Jang, Y.S.; Lee, M.H.; Bae, T.S. Comparative evaluation of mechanical properties and wear ability of five CAD/CAM dental blocks. *Materials* **2019**, *12*, 2252. [\[CrossRef\]](#)
25. Campos, F.; Almeida, C.S.; Rippe, M.P.; de Melo, R.M.; Valandro, L.F.; Bottino, M.A. Resin bonding to a hybrid ceramic: Effects of surface treatments and aging. *Oper. Dent.* **2016**, *41*, 171–178. [\[CrossRef\]](#) [\[PubMed\]](#)
26. Tribst, J.P.M.; Dal Piva, A.M.O.; Werner, A.; Anami, L.C.; Bottino, M.A.; Kleverlaan, C.J. Durability of staining and glazing on a hybrid ceramics after the three-body wear. *J. Mech. Behav. Biomed. Mater.* **2020**, *109*, 103856. [\[CrossRef\]](#) [\[PubMed\]](#)
27. Dal Piva, A.M.O.; Tribst, J.P.M.; Werner, A.; Anami, L.C.; Bottino, M.A.; Kleverlaan, C.J. Three-body wear effect on different CAD/CAM ceramics staining durability. *J. Mech. Behav. Biomed. Mater.* **2020**, *103*, 103579. [\[CrossRef\]](#) [\[PubMed\]](#)
28. Tribst, J.P.M.; Dal Piva, A.M.O.; Borges, A.L.S.; Anami, L.C.; Kleverlaan, C.J.; Bottino, M.A. Survival Probability, Weibull Characteristics, Stress Distribution, and Fractographic Analysis of Polymer-Infiltrated Ceramic Network Restorations Cemented on a Chairside Titanium Base: An In Vitro and In Silico Study. *Materials* **2020**, *13*, 1879. [\[CrossRef\]](#)
29. Dal Piva, A.M.O.; Tribst, J.P.M.; Benalcázar Jalkh, E.B.; Anami, L.C.; Bonfante, E.A.; Bottino, M.A. Minimal tooth preparation for posterior monolithic ceramic crowns: Effect on the mechanical behavior, reliability and translucency. *Dent. Mater.* **2020**. [\[CrossRef\]](#)
30. Da Cunha, L.F.; Mondelli, J.; Auersvald, C.M.; Gonzaga, C.C.; Mondelli, R.F.; Correr, G.M.; Furuse, A.Y. Endocrown with leucite-reinforced ceramic: Case of restoration of endodontically treated teeth. *Case Rep. Dent.* **2015**, *2015*, 750313. [\[CrossRef\]](#)
31. Reich, S.; Fiedlar, K. Cerec anterior crowns: Restorative options with monolithic ceramic materials. *Int. J. Comput. Dent.* **2013**, *16*, 295–315.
32. Willard, A.; Chu, G.T.M. The science and application of IPS e.Max dental ceramic. *Kaohsiung J. Med. Sci.* **2018**, *4*, 238–242. [\[CrossRef\]](#)

- 
33. Mohammadi, Z.; Giardino, L.; Palazzi, F.; Shalavi, S.; Alikhani, M.Y.; Giudice, G.L.; Davoodpour, N. Effect of sodium hypochlorite on the substantivity of chlorhexidine. *Int. J. Clin. Dent.* **2013**, *6*, 173–178.
  34. Heintze, S.D.; Forjanic, M.; Ohmiti, K.; Rousson, V. Surface deterioration of dental materials after simulated toothbrushing in relation to brushing time and load. *Dent. Mater.* **2010**, *26*, 306–319. [[CrossRef](#)]
  35. Giudice, R.L.; Famà, F. Health care and health service digital revolution. *Int. J. Environ. Res. Public Health* **2020**, *17*, 4913. [[CrossRef](#)] [[PubMed](#)]
  36. Flury, S.; Diebold, E.; Peutzfeldt, A.; Lussi, A. Effect of artificial toothbrushing and water storage on the surface roughness and micromechanical properties of tooth-colored CAD-CAM materials. *Prosthet. Dent.* **2017**, *117*, 767–774. [[CrossRef](#)] [[PubMed](#)]
  37. Della Bona, A.; Kelly, J.R. The clinical success of all-ceramic restorations. *J. Am. Dent. Assoc.* **2008**, *139*, 8S–13S. [[CrossRef](#)] [[PubMed](#)]
  38. Kanat-Ertürk, B. Color Stability of CAD/CAM Ceramics Prepared with Different Surface Finishing Procedures. *J. Prosthodont.* **2019**, *29*, 166–172. [[CrossRef](#)]
  39. Lo Giudice, R.; Tribst, J.P.M. Dental Materials Coatings: Effect on the Clinical Behavior. *Coatings* **2020**, *10*, 1229. [[CrossRef](#)]
  40. Zanatta, F.B.; Bergoli, A.D.; Werle, S.B.; Antoniazzi, R.P. Biofilm removal and gingival abrasion with medium and soft toothbrushes. *Oral Health Prev. Dent.* **2011**, *9*, 177–183.
  41. Valkenburg, C.; Slot, D.E.; Bakker, E.W.P.; Van der Weijden, F.A. Does dentifrice use help to remove plaque? A systematic review. *J. Clin. Periodontol.* **2016**, *43*, 1050–1058. [[CrossRef](#)]
  42. Giudice, R.L.; Lipari, F.; Puleio, F.; Alibrandi, A.; Giudice, F.L.; Tamà, C.; Sazonova, E.; Giudice, G.L. Spectrophotometric evaluation of enamel color variation using infiltration resin treatment of white spot lesions at one year follow-up. *Dent. J.* **2020**, *8*, 35. [[CrossRef](#)] [[PubMed](#)]