Repolishing *in situ* eroded CAD/CAM restorative materials and human enamel

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Abstract

This study aimed to evaluate the effects of repolishing on the surface microhardness (SMH), color change (ΔE), and translucency parameter (TP) of previously *in situ* eroded computer-aided design/computer-aided manufacturing (CAD/CAM) restorative materials and human enamel. Each of 8 volunteers wore an intraoral appliance containing 3 CAD/CAM restorative material specimens (IPS e.max CAD lithium disilicate ceramic, Lava Ultimate hybrid ceramic, and poly (methyl methacrylate) (PMMA) block) and 1 human enamel specimen. The specimens were subjected to *in situ* erosion cycles by rinsing with a cola drink (4×5 min/day) for 14 days. After erosion, the specimens were polished with a silicone polishing system (Ceramister, Shofu Inc, Kyoto, Japan). The SMH and color of the specimens were determined at baseline (T1), after erosion (T2), and after repolishing (T3). The ΔE and TP values of the specimens were further calculated. The data were statistically analyzed using repeated-measures analysis of variance (ANOVA) and Bonferroni's test ($\alpha=0.05$). After erosion, a decrease in the SMH of the restorative materials and enamel was observed (all $P<0.001$), and a decrease in the TP of the enamel was observed ($P=0.016$). The ΔE values of the enamel ($\Delta E=7.32$) and Lava Ultimate ($\Delta E=3.19$) exceeded the clinically unacceptable threshold after erosion. After repolishing, the SMH of the restorative materials and enamel at T3 was significantly higher than that at T2 (all $P<0.001$). No significant difference was found in the TP and ΔE values of the restorative materials and enamel between T2 and T3. In conclusion, erosion negatively affected the surface properties and appearance of the CAD/CAM restorative materials and human enamel. Repolishing contributed to restoring the

compromised SMH of the eroded restorative materials and enamel to a certain extent.

However, repolishing did not restore the color of the eroded restorative materials and enamel.

Keywords: Erosion; in situ; Restorative material; Enamel; Repolishing

1. Introduction

The worldwide prevalence of dental erosion in permanent teeth is estimated to be 30.4% for children and adolescents (Salas et al., 2015). Erosive tooth wear (ETW) is tooth wear that is primarily caused by dental erosion (Schlueter et al., 2020). In advanced stages, ETW often leads to the loss of more than 50% of the surface area of dental hard tissues, resulting in tooth hypersensitivity and a low chewing efficiency (Lussi et al., 2011; Schlueter et al., 2020). Computer-aided design/computer-aided manufacturing (CAD/CAM) restorative materials, including resin composites, ceramics, and hybrid ceramics (Lawson et al., 2016; Spitznagel et al., 2018), are increasingly adopted to reestablish the tooth contour and minimize further dental hard tissue loss by acid exposure (Harryparsad et al., 2014; Lussi et al., 2011; Sulaiman et al., 2015).

It is critical to eliminate the pathogeny of ETW before restoration placement (Attin and Wegehaupt, 2014; Peutzfeldt et al., 2014). However, due to the negative attitudes and poor knowledge of ETW of the public, restorations may be exposed to acidic conditions over time (Attin and Wegehaupt, 2014; Hong et al., 2020). Erosion leads to surface degradation of human enamel (Attin et al., 2001; Francisconi et al., 2008; Wongkhantee et al., 2006; Yang et al., 2020) and affects the clinical behavior of CAD/CAM restorative materials (Alencar-Silva et al., 2019; Backer et al., 2017; Cruz et al., 2019; Gao et al., 2015; Kukiattrakoon et al., 2010; McKinney, 1985; Sagsoz and Polat Sagsoz, 2019; Yu et al., 2009; Yu et al., 2017). The surface microhardness (SMH) of human enamel decreased after acidic attack, and the etching pattern of enamel showed obvious demineralization (Attin et al., 1997; Yu et al., 2018). In addition, significant surface softening of ceramic materials was evident after immersion in fruit juice (Kukiattrakoon

et al., 2010). The SMH of hybrid ceramics and resin composites were also reduced by exposure to cola drinks (Colombo et al., 2019). Additionally, the color stability and translucency of restorative materials and enamel can be negatively affected by the adsorption of colorants derived from commercially available beverages with varied erosive potentials, such as cola, coffee, and red wine (Abdalkadeer et al., 2020; Acar et al., 2016; Borges et al., 2014; Dos Santos et al., 2017; Eldwakhly et al., 2019; Enan et al., 2019; Quek et al., 2018; Ribeiro et al., 2017; Sarikaya et al., 2018). However, no *in situ* study has been conducted to evaluate the effect of erosion on the optical properties of CAD/CAM restorative materials and human enamel.

The negative effects of erosive or acidic substances on restorative materials are limited to very superficial layers (Hannig et al., 2007). Thus, it might be conceivable that the compromised surface properties could be eliminated by a polishing procedure, which is conducive to improving the function of dental materials by removing superficial surface defects (Guazzato et al., 2004; Guazzato et al., 2005); the polishing process also provides esthetic benefits and a smooth restoration appearance and reduces the risk of surface breakdown and corrosion (Jefferies, 2007). Several studies have been conducted to evaluate the effects of repolishing on discolored direct restorative materials and human enamel (Borges et al., 2014; Mundim et al., 2010; Rutkunas and Sabaliauskas, 2009; Türkün and Türkün, 2004). Repolishing improved the esthetic appearance of discolored resin composites and enamel by partially removing surface staining (Borges et al., 2014; Rutkunas and Sabaliauskas, 2009). It is conceivable that repolishing contributes to reversing the negative erosive effects on the SMH and appearance of CAD/CAM

restorative materials and human enamel. However, there is little direct evidence to confirm this hypothesis.

Therefore, the aim of this study was to determine the influence of repolishing following *in situ* erosion on the SMH, color change (ΔE), and translucency parameter (TP) of CAD/CAM restorative materials and human enamel. The following null hypotheses were tested: 1) the SMH, ΔE , and TP values of CAD/CAM restorative materials and human enamel would not be affected by an acid challenge; and 2) the SMH, ΔE , and TP values of eroded CAD/CAM restorative materials and human enamel would not be affected by repolishing.

2. Material and methods

2.1. Sample size calculation

The sample size was calculated using G-power software (University of Düsseldorf, Düsseldorf, Germany). The power necessary to detect the primary outcome (SMH) was calculated based on a two-sided *t*-test with a significance level of 5% and statistical power of 80%. The results showed that the minimum sample size per group was 6 participants. Assuming an anticipated dropout rate of 10%, the final sample size was determined to be 8 participants per group (Chen et al., 2020; Yang et al., 2020).

2.2. Specimen preparation

In this study, human enamel and three types of CAD/CAM restorative materials were tested (Table 1). For each type of CAD-CAM block, disk-shaped specimens (4 mm diameter and 2 mm thickness) were fabricated using CAD/CAM (Zenotec, Wieland, Germany). The IPS e.max CAD specimens were crystallized (Programat P300, Ivoclar Vivadent) according to the instructions from the manufacturer. Buccal enamel blocks (4 mm diameter and 1 mm thickness) were cut from extracted human molars and embedded into acrylic resin (dentin shade, New Century, Shanghai Kangqiao Dental Product, China) to achieve the same dimensions of the restorative material specimens. The specimens were polished with a three-step silicone polishing system (Ceramister, Shofu Inc, Kyoto, Japan) using a slow-speed handpiece by a single blinded operator. The operator was calibrated by a wireless dynamometer (T-measurement, Dalian Taijia Technology Co., Ltd, Dalian, China) and a press-on force of 2 N pressure was applied during polishing. For each polisher, all specimens were polished for 30 s in the same direction (left to right)

(Vrochari et al., 2017). The polisher was discarded after being used once (Chavali et al., 2017). The specimens were then ultrasonically cleaned in distilled water for 10 min.

2.3. *In situ* experiment

The study received ethical approval from the local university (Approval no. 18-FMUSS-21) and was designed according to the Declaration of Helsinki and the Guidelines of Good Clinical Practice. The *in situ* protocol has been previously described in detail (Yang et al., 2020; Yu et al., 2018). Briefly, 8 healthy volunteers (4 men and 4 women, average age 23.4 years) met the inclusion criteria and participated in this study. Each volunteer wore an intraoral appliance containing four specimens (one specimen per experimental group) throughout the experimental period and only removed the intraoral appliance during meals and routine oral hygiene. The appliances were stored in artificial saliva when not in the mouth. The erosive challenge consisted of rinsing with 150 ml of a freshly opened cola drink (Coca-Cola, Quanzhou, China, pH=2.3) for 5 min and 4 times per day for 14 consecutive days. After each erosive challenge, the volunteers rinsed the appliance with distilled water for 10 s, and the appliances were then replaced into the mouth.

2.4. *Repolishing procedure*

After *in situ* erosion, the specimens were removed from the appliance and repolished with Ceramister as previously described. The repolished specimens were then ultrasonically cleaned in distilled water for 10 min. The substance loss due to repolishing was measured using a micrometer with a detection limit of 2 μm (Mitutoyo, Tokyo, Japan).

2.5. *SMH measurement*

The SMH values were measured using a Vickers microhardness tester with a microscopic lens (HXD-1000TM/LCD, Taiming Optical Instrument Co. Ltd, Shanghai, China). For the PMMA block, the SMH was measured with a 100 g load and 10-s dwell time. For the IPS e.max CAD, Lava Ultimate, and enamel, the SMH was measured with a 1000 g load and 10-s dwell time. Five indentations per specimen (500 µm from each other) were performed, and the mean value of each specimen was calculated. SMH analyses were conducted at the following time points: at baseline (T1), after the *in situ* erosion (T2), and after the repolishing treatment (T3).

2.6. Color and translucency measurement

The color and translucency of the specimens were analyzed by using a spectrophotometer (Easyshade, VITA Zahnfabrik, Bad Säckingen, Germany) based on the CIE L*a*b* system. The color was measured on a white background, and the translucency was measured on white and black backgrounds (Alencar-Silva et al., 2019; Sulaiman et al., 2015). The color and translucency measurements of each specimen were performed at T1, T2, and T3. Three measurements were made per specimen, and the mean CIE L*, a*, b* values were calculated.

The ΔE was calculated for each specimen using the following formula (Chen et al., 2020):

$$\Delta E = \sqrt{(L_b^* - L_t^*)^2 + (a_b^* - a_t^*)^2 + (b_b^* - b_t^*)^2}$$

where the subscript b indicates measurement at T1, and subscript t indicates measurement at T2 or T3.

The TP was calculated for each specimen using the following formula (Sulaiman et al.,

2015):

$$TP = \sqrt{(L_b^* - L_w^*)^2 + (a_b^* - a_w^*)^2 + (b_b^* - b_w^*)^2}$$

where the subscripts b and w indicate measurements against white and black backgrounds, respectively.

2.7. Statistical analysis

The Kolmogorov-Smirnov test and the Levene's test were used to determine the normality and homogeneity of the data, respectively. A two-way repeated-measures analysis of variance (ANOVA) was performed to identify the effects of the materials and treatment on the SMH, ΔE and TP of the tested materials. The Bonferroni's test was used for post-hoc analysis. The data were analyzed using the SPSS statistical software package (SPSS 22.0 for Windows, SPSS, Chicago, IL, USA) at a significance level of 0.05.

3. Results

The substance losses due to repolishing of the restorative materials and human enamel were all immeasurable with the micrometer. The results of the Kolmogorov-Smirnov test and Levene's test were non-significant, indicating that the assumptions of a normal distribution in the data and homogeneity of variance are not violated. The ANOVA results are shown in Table 2. For the SMH, significant differences were found among the materials ($P<0.001$) and treatments ($P<0.001$). For the TP and ΔE , significant differences were found among the materials ($P<0.001$).

The SMH of the restorative materials and enamel significantly decreased after *in situ* erosion (all $P<0.001$) (Table 3). After repolishing, a significantly higher SMH was found at T3 than at T2 for all groups (all $P<0.001$). The SMH of IPS e.max CAD at T3 was significantly lower than that at T1 ($P=0.002$), while no significant difference in the SMH of the PMMA block, Lava Ultimate, and enamel was observed between T1 and T3. Significant differences in the SMH were found among the restorative materials and enamel at all time points (IPS e.max CAD>enamel> Lava Ultimate>PMMA block).

The TP value of the enamel significantly decreased after *in situ* erosion ($P=0.016$) (Table 4). For the restorative materials, no significant difference in the TP values was found between T1 and T2. For all groups, no significant differences in the TP values were observed between T2 and T3 and between T1 and T3. Significant differences in the TP values were found among the restorative materials and enamel at T1 (Lava Ultimate>IPS e.max CAD>PMMA block>enamel).

A very large ΔE value was observed after *in situ* erosion in the human enamel

($\Delta E=7.32$), followed by that in the Lava Ultimate ($\Delta E=3.19$), PMMA block ($\Delta E=2.08$), and the IPS e.max CAD ($\Delta E=1.36$) (Table 5). For all groups, no significant difference in the ΔE values was observed between T2 and T3.

4. Discussion

Based on the present findings, the null hypotheses that the SMH, ΔE and TP of the CAD/CAM restorative materials and human enamel would not be affected by acid challenge and that the SMH, ΔE and TP of eroded CAD/CAM restorative materials and human enamel would not be affected by repolishing were rejected.

Most *in vitro* studies cannot fully replicate the clinical situation. In the present study, an *in situ* model was used to simulate the acid challenge intraorally (Yang et al., 2020; Yu et al., 2018). A cola drink was chosen due to its popularity and high erosive potential (Yu et al., 2009). Moreover, due to the presence of caramel color in cola drinks, the effects of the drinks as an extrinsic factor on the optical properties of restorative materials have been widely studied (Alencar-Silva et al., 2019; Aydin et al., 2020). The present study did not evaluate the effect of repolishing on the surface roughness of eroded restorative materials and enamel, although the surface roughness may be directly related to the substance loss, color, and translucency of the restorative materials and enamel. In a previous *in situ* study, a similar erosion protocol showed no effect on the surface roughness of CAD/CAM restorative materials but significantly roughened the enamel ($\Delta R_a=0.59 \mu m$) (Yang et al., 2020). However, this level of change in the surface roughness of enamel could easily be eliminated by polishing procedures (Meredith et al., 2017).

Various techniques have been used to evaluate the surface degradation of dental materials by acid challenges. Microindentation techniques have been used extensively to investigate the early stages of ETW by measuring the SMH (Barbour and Rees, 2004;

Colombo et al., 2019; Kukiattrakoon et al., 2010). The SMH of the tested restorative materials and enamel significantly decreased after *in situ* erosion, which was in agreement with previous studies (Backer et al., 2017; Kukiattrakoon et al., 2010; Yu et al., 2018). It is well known that erosion causes demineralization of enamel crystals, thereby softening the enamel surface (Lussi et al., 2011; Wongkhantee et al., 2006). Regarding the CAD/CAM restorative materials, the acid challenge significantly reduced the SMH, which was in accordance with previous studies (Alencar-Silva et al., 2019; Alnasser et al., 2019; Kukiattrakoon et al., 2010; Sagsoz and Polat Sagsoz, 2019). A possible explanation could be that the presence of an acid led to the dissolution of the superficial glassy matrix and the ceramic silicate network (Si-O-Si) in the IPS e.max CAD (Cruz et al., 2019; Kukiattrakoon et al., 2010). In addition, the surrounding resin matrix or silane coupling agent of the PMMA block and Lava Ultimate could be hydrolyzed by acid, leading to the softening of the surface (Backer et al., 2017; Gao et al., 2015). The softened surface of restorative materials and enamel appears to be more susceptible to mechanical forces than the original inert surface (McKinney, 1985; Yu et al., 2009).

Although repolishing softened materials has been recommended to improve their physical properties (Lima et al., 2008), there is no available evidence to support this recommendation. For the first time, the current study showed that a repolishing treatment was effective in regaining the compromised SMH of eroded restorative materials and enamel without removing substantial amounts of the surface material. After repolishing, the SMH of the eroded materials returned to the baseline values, except for the IPS e.max CAD (77.3% of the SMH regained). This may indicate that the softened parts of the

restorative materials and enamel due to cola drinks were limited to a superficial region.

The TP can be described as the difference in color values obtained between the light reflected by a material with a defined thickness on white and black backgrounds (Hu and Johnston, 2011). The TP changes are regarded as perceptible if ΔTP exceeds the limit of 2 (Eldwakhly et al., 2019). After the *in situ* erosion process, the TP values of the human enamel decreased ($\Delta TP=2.71$), and the ΔE value was greater than 2.70, representing clinically unacceptable color differences according to the ISO standard (ISO28642-2016, 2016) and indicating that the human enamel became more opaque, and the color change was clinically unacceptable. This finding was consistent with previous studies (Carlos et al., 2019; Enan et al., 2019). The tooth discoloration may be related to the adsorption of pigments on the enamel surface (Chen et al., 2020). After considering the protection provided by the saliva flow and acquired pellicle, it was determined that the erosive effects of the cola drinks on the substance loss of the human enamel were approximately 3 to 4 times greater *in vitro* than *in situ* (Yang et al., 2020). However, regarding the tooth discoloration caused by cola drinks, the data obtained from *in vitro* (Enan et al., 2019) and *in situ/ex vivo* studies (Carlos et al., 2019) were similar to the *in situ/in vivo* data in the present study.

The TP values and ΔE values of the repolished human enamel were similar to those of the eroded human enamel. The color changes of the repolished human enamel (ΔE value=5.32) were still above the unacceptability threshold. This may indicate that the surface staining of human enamel can only be partially removed by repolishing procedures. This statement was supported by a previous study (Borges et al., 2014). The

acidic solutions may lead to the dissolution of calcified tooth structure and an increased permeability of enamel, which promote the penetration of pigments through enamel structures (Azer et al., 2010; Rezende et al., 2013).

Regarding the CAD/CAM restorative materials, the acid challenge and repolishing treatment did not affect the TP values. Sarikaya et al. (Sarikaya et al., 2018) concluded that 48 hours of immersion in cola drinks had no detectable effect on the TP values of Lava Ultimate and Vita Enamic. It was also reported that cola exposure for 28 days did not influence the TP values of IPS e.max CAD (Alencar-Silva et al., 2019). After the acid challenge, the color changes in the IPS e.max CAD and PMMA block were all clinically acceptable (all ΔE values < 2.70), which was consistent with the results of previous studies (Alencar-Silva et al., 2019; Quek et al., 2018). However, Dos Santos et al. (Dos Santos et al., 2017) reported that clinically unacceptable ΔE values of IPS e.max CAD were observed after immersion in cola drinks (ΔE values = 2.95). This controversy might be attributed to differences in the protocol of the acidic challenges (immersion in an acidic solution alone vs. thermocycling using an acidic solution and artificial saliva). In the present study, the discoloration of Lava Ultimate after the acid challenge was clinically unacceptable (ΔE value = 3.19). It was reported that hydrophilic triethylene glycol dimethacrylate (TEGDMA) and bisphenol A-glycidyl methacrylate (Bis-GMA) present in Lava Ultimate exhibit a high water absorption and therefore allow hydrophilic colorants to infiltrate the resin matrix (Acar et al., 2016). On the other hand, the ΔE values of the tested CAD/CAM restorative materials remained unchanged after the repolishing treatment, indicating that the discoloration of the restorative material may be attributed

to subsurface degradation. This finding is in contrast with previous studies of resin composites (Mundim et al., 2010; Türkün and Türkün, 2004). The diversity of opinions was most likely due to difference in the microstructures and polishability values between the direct and indirect restorative materials (Mundim et al., 2010). However, this hypothesis needs to be clarified in future studies.

The present study included a relatively short *in situ* erosion duration and did not consider the effect of toothbrush abrasion, which were considered to be limitations. Although the dental restorative materials showed surface degradation over time under acidic conditions, the CAD/CAM restorative materials were more acid resistant than the human enamel and can be used as a suitable material to rehabilitate erosive dentition (Yang et al., 2020). Moreover, the decreased SMH of the CAD/CAM restorative materials can be restored, at least to a certain extent, by a repolishing treatment during maintenance visits. Nevertheless, surface degradation would occur again if patients maintain their unhealthy lifestyle (Yu et al., 2017). Moreover, clinicians should be aware that repolishing is not effective in improving the compromised appearance of eroded CAD/CAM restorative materials and enamel.

5. Conclusions

Erosion negatively affected the surface property and appearance of CAD/CAM restorative materials and human enamel. Repolishing contributed to restoring the compromised SMH of the eroded restorative materials and enamel to a certain extent. However, repolishing did not restore the color of the eroded restorative materials and enamel.

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Table 1Materials evaluated in the study.

Material	Type	Composition*	Manufacturer	Lot number
IPS e.max CAD	Lithium disilicate ceramic	57%-80% SiO ₂ , 11%-19% Li ₂ O, 0%-13% K ₂ O, 0%-11% P ₂ O ₅ , 0%-8% ZrO ₂ , 0%-8% ZnO, 0%- 5% Al ₂ O ₃ , 0%-5% MgO	Ivoclar Vivadent, Schaan, Liechtenstein	Y40748
Lava Ultimate	Resin nanoceramic	80% nanoceramic (69% SiO ₂ , 31% ZrO ₂) 20% resin matrix (BisGMA, UDMA, BisEMA, TEGDMA)	3M-ESPE, St Paul, MN, USA	NC08349
PMMA block	poly (methyl methacrylate) (PMMA) resin	Poly (methyl methacrylate)	Aidite Technology Co., Ltd., Qinhuangdao, China	20191025A3

* information provided by the manufacturers

Table 2

Two-way ANOVA results of the comparison of the surface microhardness (SMH), translucency parameter (TP), and color change (ΔE) values.

Parameters	Source	F	P
SMH	Material	17300.72	<0.001*
	Treatment	134.45	<0.001*
	Material×Treatment	30.36	<0.001*
TP	Material	169.20	<0.001*
	Treatment	2.70	0.073
	Material×Treatment	1.95	0.082
ΔE	Material	19.70	<0.001*
	Treatment	0.53	0.468
	Material×Treatment	1.28	0.292

* indicates a significant difference ($P<0.05$).

Table 3

Means and standard deviations (SD) of the surface microhardness (SMH) of each material.

Material	SMH		
	T1	T2	T3
IPS e.max CAD	636.27 (12.72) ^{A, a}	552.37 (9.51) ^{E, b}	617.26 (5.05) ^{I, c}
Lava Ultimate	101.61 (8.12) ^{B, d}	84.62 (2.61) ^{F, e}	97.80 (1.82) ^{J, d}
PMMA block	19.62 (0.60) ^{C, f}	17.12 (0.37) ^{G, g}	19.35 (0.20) ^{K, f}
Enamel	328.35 (17.64) ^{D, h}	280.84 (20.02) ^{H, i}	321.11 (8.45) ^{L, h}

Different superscripted uppercase letters in a column indicate significant differences for different groups of tested material ($P < 0.05$). Different superscripted lowercase letters in a row indicate significant differences at each time point ($P < 0.05$).

T1: baseline

T2: after the *in situ* erosion

T3: after the repolishing treatment

Table 4

Means and standard deviations (SD) of the translucency parameter (TP) of each material.

Material	TP		
	T1	T2	T3
IPS e.max CAD	14.11 (0.54) ^{A, a}	14.19 (0.44) ^{E, F, a}	13.95 (0.54) ^{H, I, a}
Lava Ultimate	15.84 (1.40) ^{B, b}	15.16 (1.11) ^{E, b}	15.45 (1.42) ^{I, b}
PMMA block	12.13 (0.95) ^{C, c}	12.28 (0.85) ^{F, c}	12.03 (1.54) ^{H, c}
Enamel	8.47 (1.56) ^{D, d}	5.76 (2.31) ^{G, e}	7.29 (2.06) ^{J, d, e}

Different superscripted uppercase letters in a column indicate significant differences for different groups of tested material ($P < 0.05$). Different superscripted lowercase letters in a row indicate significant differences at each time point ($P < 0.05$).

T1: baseline

T2: after the *in situ* erosion

T3: after the repolishing treatment

Table 5

Means and standard deviations (SD) of color change (ΔE) for each material.

Material	ΔE	
	T2	T3
IPS e.max CAD	1.36 (0.93) ^{A, a}	1.41 (0.44) ^{C, a}
Lava Ultimate	3.19 (1.15) ^{A, b}	3.56 (0.80) ^{C, D, b}
PMMA block	2.08 (1.15) ^{A, c}	2.24 (0.32) ^{C, c}
Enamel	7.32 (4.17) ^{B, d}	5.32 (2.94) ^{D, d}

Different superscripted uppercase letters in a column indicate significant differences for different groups of tested material ($P < 0.05$). Different superscripted lowercase letters in a row indicate significant differences at each time point ($P < 0.05$).

T2: after the *in situ* erosion

T3: after repolishing treatment