Effect of Tooth Brushing on Surface Color of Ceramic-Polymer Materials: An In Vitro Study

Purpose: The purpose of this study was to examine the effects of tooth brushing on the change in color of extrinsic characterization of ceramic-polymer materials. **Materials and Methods:** Two ceramic-polymer materials (CeraSmart, GC; Enamic, Vita) and one lithium-disilicate material (IPS e.max CAD; Ivoclar Vivadent) were tested. Specimens of each material in size 14 blocks were prepared, characterized and glazed per manufacturer's instructions. The treated surface of the blocks were then brushed in a toothpaste slurry with artificial saliva using a toothbrush machine with a soft toothbrush. Commission Internationale de L'Eclairage (CIE) L*a*b* values were recorded with a spectrophotometer at baseline and at 3, 6, 9, and 12 simulated years of brushing (7,300 strokes/yr). A mean change in color (ΔE*) and standard deviation were determined for each group and brushing interval. Data were analyzed with a two-way repeated measures ANOVA examining the effects of tooth brushing the ceramic materials on ΔE* over time (alpha = 0.05). **Results:** The difference in the ΔE* between CeraSmart and Enamic was significant at 3 years, while the differences between them were not significant at 6 years, 9 years, and 12 years of simulated brushing. The ΔE* of IPS e.max CAD was significantly smaller than CeraSmart and Enamic at all time points (all $p < 0.0001$) except for the comparison with Enamic at 3 years. **Conclusions:** The extrinsic stains on the ceramic-polymer materials may be more susceptible to change from simulated tooth brushing compared to the lithium-disilicate material.

KEYWORDS: Extrinsic stains, tooth brushing, ceramic polymers, lithium disilicate, surface color

Ceramic restorations are usually characterized via superficial application of pigments (commonly known as “extrinsic staining”). These pigments are strategically applied using a fine brush to create desired effects, colors, and shades.[1-3] Unlike intrinsic characterization of ceramic restorations, extrinsic characterization is in direct contact with the oral environment and abrasives, and therefore at higher risk of fading.[3] Consequently, it has been recommended that extrinsic stains be placed as deeply as possible to enhance longevity.[4]

Tooth brushing with fluoride-containing toothpaste is the most effective approach for plaque removal and dental-caries prevention.[5-7] It is therefore recommended to brush at least twice a day for a minimum of two minutes each.[5] Unfortunately, abrasives are an integral component of toothpastes and may have an adverse effect on surface characterization of restorations. Garza *et al.* demonstrated that twelve years of tooth brushing did not produce a clinically relevant shade change or increase in the surface roughness of extrinsically stained and/or glazed IPS e.max Press (Ivoclar Vivadent, Amherst, NY) a pressable lithium-disilicate glass-ceramic.[8] Aker *et al.* indicated the use of a normal toothbrush with a common toothpaste could wear away surface characterization applied to feldspathic porcelain restorations in a period of 10-12 years. However, colors lasted longer when a layer of protective glaze was applied.[3]

There are three main groups of dental ceramic and ceramic-like materials available – glass-matrix, polycrystalline, and ceramic polymers.[9] Glass-matrix ceramics contain nonmetallic inorganic ceramic materials that present with a glass phase. Examples include feldspathic ceramics, leucite-based ceramics, and lithium disilicate and derivatives. These materials tend to be the most esthetic, but have lower flexural strengths, and they are relatively brittle.[10]

Polycrystalline ceramics like alumina and stabilized zirconia contain nonmetallic inorganic ceramic materials that do not present any glass phase. These materials usually

exhibit high flexural strength, but may lack esthetics without extrinsic staining or esthetic ceramic veneering. Ceramic polymers are made up of predominantly inorganic refractory compounds that usually include porcelains, glasses, ceramics, glass-ceramics, in addition to organic polymers. These materials have a modulus of elasticity close to that of dentin, and they are not as brittle as the glass-matrix and polycrystalline ceramics. Nevertheless, ceramic polymers may require extrinsic staining to achieve acceptable clinical esthetics.[9]

The new ceramic-polymer materials appear to fall on a spectrum between polymer and ceramic, with some materials, such as Lava Ultimate (3M ESPE, St. Paul, MN), CeraSmart (GC America, Alsip, IL), and Shofu HC (Shofu, San Marcos, CA) containing primarily polymer materials. On the other hand, Enamic (Vita, Yorba Linda, CA), reportedly contains more ceramic material.[9,11,12] Two ceramic-polymer materials, CeraSmart and Enamic, represent both ends of the spectrum of ceramic-polymer materials and are marketed with a dedicated proprietary light-polymerized staining kit. CeraSmart is composed of a combination of bis-methacryloxyethoxy phenyl propane (BisMEPP), urethane dimethacrylate (UDMA), and dimethacrylate (DMA) with silica and barium glass fillers at 71% by weight. GC claims CeraSmart is a “force absorbing hybrid ceramic CAD/CAM block”, with the same flexible amorphous nano-ceramic technology as their G-aenial Universal Flo composite material (200-nm strontium glass particles).[13] Enamic is composed of predominately a feldspar-ceramic network enriched with aluminum oxide (86% wt or 75% vol) and a polymethyl-methacrylate (14% wt or 25% vol) that permeates the feldspar-ceramic matrix to yield a restorative material that is both strong and elastic as claimed by the manufacturer.[12]

The glass-ceramic, lithium disilicate (IPS e.max CAD, Ivoclar Vivadent) was chosen as the control because it is a commonly used material that has been well studied and clinically successful with over 96% success rate.[13] The material is milled at an intermediate blue state composed of 40% lithium-metasilicate crystals. After

crystallization, the material crystallizes to approximately 70% fine-grain lithium disilicate, creating increased strength and wear resistance.[14]

Extrinsic characterization of dental restoratives is usually carried out by applying a desired colorant to external surfaces of a restoration to achieve the desired effect. According to Ivoclar Vivadent technical instructions, for glass ceramics such as IPS e.max CAD, the characterized restoration must be placed in a ceramic oven capable of reaching temperatures of 840ºC.[15] In contrast, both Vita and GC recommend extrinsic characterization via visible-light polymerization of their methacrylate-based surface stains.[16,17] Fasbinder et al noted that the polymer chemistry of CAD/CAM composite resin restorations rendered them easier to adjust, polish, stain and repair.[18] However, the longevity of these repairs has not been validated by clinical studies.

No studies have reported the effect of tooth brushing on extrinsically characterized ceramic-polymer materials. The purpose of this *in vitro* study was to evaluate the effect of tooth brushing on the surface of ceramic-polymer materials, specifically surfaces treated with visible light-cured stains. The null hypothesis suggested there would be no difference in the change in color based on type of ceramic material over 3, 6, 9, and 12 equivalent years of simulated tooth brushing.

MATERIALS AND METHODS

Ten blocks (size C14) from each of the three tested materials (n=10) in shade A2 (CeraSmart, Enamic, and IPS e.max CAD) were polished using an Ecomet 6 (Buehler, Lake Bluff, IL) lapping table in conjunction with silicon-carbide paper which ranged from 320-grit through 420-grit. Each block's thickness was measured with a digital caliper (GA182, Grobet Vigor, Carlstadt, NJ) and then covered with 120µm-thick tape (Masking Tape, 3M, Maplewood, MN) with a window cut in the area to be stained and glazed for standardization. The metal stubs of the IPS e.max CAD blocks were removed and the

blocks were crystallized followed by staining (IPS e.max Crystall./Shade 1, Ivoclar Vivadent) and glazing (IPS e.max Crystall./Glaze and Glaze Liquid, Ivoclar Vivadent) in a ceramic oven to 840°C (Programat P500, Ivoclar Vivadent) as per the manufacturer's recommendation utilizing the same masking tape method as stated above. The metal stubs were then reattached to the IPS e.max CAD blocks with a commercial adhesive (Gorilla Glue, Gorilla Glue Co., Cincinnati, OH). Using the digital caliper, the thickness of the stain and glaze was measured to assure standardization. The stain and the glaze were each approximately 60µm thick for each sample.

Enamic specimens were treated with the Enamic Stain Kit (Vita) according to manufacturer's instructions.[16] The surface was etched with a 5% hydrofluoric acid gel (Ceramics Etch, Vita) for 60 seconds and then steam cleaned (i700B, Reliable, Toronto, Ontario). The cleaning solution included in the kit (Cleaner, Vita) was applied and air-dried. Masking tape was used in the same manner described above for standardization of the extrinsic stain and glaze. Next, silane (VitaSil, Vita) was applied and air-dried. Stain 3 Brown was applied and light cured for 30 seconds with Bluephase G2 LED Curing Light (Ivoclar Vivadent) in the "high" setting. Irradiance of the curing light was monitored with a radiometer (LED Radiometer, Kerr, Orange, CA) to verify irradiance levels above 1,200 mW/cm^2. Glaze was applied with a clean disposable brush and light polymerized for an additional 60 seconds. The digital caliper was used to assure standardization of the stain thickness.

CeraSmart blocks were stained and glazed using Optiglaze (GC America) according to the manufacturer's instructions.[17] First, the CeraSmart blocks were sandblasted (AccuFlo, Comco Inc., Burbank, CA) with 50µm of aluminum oxide at a distance of 10mm at 80 PSI and steam cleaned. Then, the Optiglaze Ceramic Primer II was applied and air-dried. Stain A-plus brown was applied and light cured for 40 seconds with the curing light as before. Finally, Optiglaze Clear was applied and light cured for 60

seconds. Baseline surface color measurements for individual blocks (CIE - L*a*b*), were recorded with the spectrophotometer (Easyshade, Vita). A 3D printed index (Eden 260V 3-D printer, Stratasys, Eden Prairie, MN) created with a resin material (VeroDent MED670, Stratasys) was utilized to hold each block specimen and the spectrophotometer in the same orientation every time the color was measured. See Figure 1.

The ten specimens per group were mounted in a 10-station automated toothbrush machine (Sabri Dental Enterprises, Downers Grove, IL) using 3D-printed indexes. Soft filament toothbrush heads were sectioned from brush handles (GUM 311 Classic Toothbrush, Sunstar, Schaumburg, IL) and fixed to the machine brush arm. Each toothbrush head was applied to the stained surfaces with a force of 3N. All specimens were brushed for one-year intervals up to 12 equivalent-years of brushing with 87,600 strokes at a rate of 60 strokes per minute (1 hz). Toothbrush heads were replaced every 7,300 strokes (one-year equivalent).[19] An abrasive slurry consisting of toothpaste (Crest, Procter and Gamble, Cincinnati, OH) at a mixture of 21g per 80 mL of synthetic mouth fluid was utilized for experimental purposes. The synthetic saliva was prepared with the following solutions: Na_3PO_4 - 3.90 mM $NaCl_2$ - 4.29 mM KCl - 17.98 mM $CaCl_2$ - 1.10 mM $MgCl_2$ - 0.08 mM H_2SO_4 - 0.50 mM $NaHCO_3$ - 3.27 mM, distilled water, with a pH set to a level of 7.2.[20] A pH meter (Accumet XL50, Fisher Scientific, Waltham, MA) was used to measure pH. Specimens were removed from the automated toothbrush machine at prescribed intervals and color measurements were performed using the spectrophotometer and 3D printed index to orient each sample in the same way to the spectrophotometer tip. Changes in color of the ceramic-polymer materials from the tooth brushing process were determined using the CIE L* a* b* color space. L* indicates lightness (L+ = lightness and L- = darkness), the a* coordinate represents the red/green range (a*+ = redness and a*- = greenness) and the b* coordinate represents for the yellow/blue range (b* + = yellowness and b*- = blueness). The L* a* b* system allows the

numeric definition of a color as well as the overall difference between two colors (ΔE^*).[21] The mean of three readings per specimen was recorded for each L*a*b* value and standard deviations were determined for each group at baseline, 3, 6, 9, and 12-year equivalent simulated years of brushing. Color change was measured using the formula: $\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. Two-way repeated measures ANOVA was conducted to test the effects of material and time on the ΔE^*. The sample size of 10 specimens per group provided 80% power to detect a moderate effect size (0.3, or approximately 0.6 standard deviation difference) among means for the main factors of group and time, and a moderate effect (0.35, or approximately 0.7 standard deviation difference) for the interaction term when testing with a two-way repeated measures ANOVA at the alpha level of 0.05 (NCSS PASS 2002).

RESULTS

Two-way repeated measures ANOVA with a Greenhouse-Geisser correction showed that overall main effects of material and time and the interaction effect between material and time were statistically significant: Material [$F(2, 27)= 359.72$, $p < 0.0001$]; Time [$F(2.31, 62.33)= 697.35$, $p < 0.0001$]; Material*Time [$F(4.62, 62.33)= 77.02$, $p < 0.0001$]. The significant interaction between material and time indicates that the differences among the material groups varied at different time points.

Post hoc tests using the Tukey correction for material groups revealed that the ΔE^* of IPS e.max CAD was significantly smaller than the ΔE^* of CeraSmart ($p < 0.0001$) and Enamic ($p < 0.0001$), but the difference in the ΔE^* between Enamic and CeraSmart was not significant ($p = 0.053$). Post hoc tests using the Tukey correction for time revealed that all of the pair-wise comparisons between different time points were statistically significant at $p < 0.0001$. Post hoc tests using the Tukey correction for the material and time interaction indicated that the difference in the ΔE^* between Enamic and IPS e.max

CAD was not significant ($p = 0.46$) at 3 years, while the differences between them were significant ($p < 0.0001$) at 6, 9, and 12 years of simulated brushing. On the other hand, the difference in the ΔE^* between CeraSmart and Enamic was significant ($p < 0.0001$) at 3 years, while the differences between them were not significant at 6 years ($p = 0.94$), 9 years ($p = 1.00$), and 12 years ($p = 0.88$) of simulated brushing. Except for the comparison with Enamic at 3 years, the ΔE^* of IPS e.max CAD was significantly smaller than CeraSmart and Enamic at all time points (all $p < 0.0001$). See Table 1 and Figure 2.

DISCUSSION

Indirect ceramic-polymer materials are created under optimized temperature and pressure settings for a controlled and standardized polymerization. Polymerization in the laboratory allows for enhanced mechanical strength and improved stain resistance when compared to direct resin composite material.[22,23] Additionally, these polymers can be repaired using light-cured composite materials and readily characterized with light-polymerized stains. Extrinsic stains may be necessary to achieve comparable esthetics to ceramic materials.[18] However, these new ceramic-polymer materials may be less resistant to the abrasive effects of tooth brushing compared to ceramic materials.

In this study, significant increases in ΔE^* were observed in the two stained and glazed ceramic-polymer materials over a 12-year equivalent of simulated tooth brushing when compared to extrinsic stains on IPS e.max CAD. Therefore, the null hypothesis was rejected. Garza *et al.* showed that it required 12 years of simulated tooth brushing for noticeable change on extrinsic stains on IPS e.max Press, a pressable lithium-disilicate material.[8] The current study found a minimal effect on IPS e.max CAD with only a 0.8 ± 0.2 increase in ΔE^* after 12 years of simulated tooth brushing. A ΔE^* measure of 1.0 is the most frequently reported threshold value for the perceptibility of color differences.[21] Compared to the IPS e.max CAD extrinsic stains, both CeraSmart and Enamic extrinsic

stains displayed a statistically significant increase in ΔE* starting at 6 years of simulated brushing. At that time, the surface changes with both CeraSmart and Enamic may be clinically noticeable with an increase of ΔE* of 1.2 ± 0.1 and 1.1 ± 0.2 respectively. Conversely, the findings of the current study suggest that changes in the visible-light-polymerized extrinsic stains of ceramic-polymer materials may remain imperceptible to the eye for at least three years of tooth brushing.

In a recent study by Partin-Agarwal et al., the surface roughness of new CAD/CAM ceramic-polymer materials without any surface stains or glazes was evaluated before and after abrasion with a toothbrush and dentifrice. Compared to the tested ceramic-polymer materials, only IPS e.max CAD had no statistically significant increase in surface roughness after 60,000 brush strokes (a simulated 8 years of toothbrush abrasion).[24] This result is not unexpected, as lithium-disilicate ceramic has been shown to exhibit high polishability and polish retention.[25] In a similar study performed by Koizumi et al, both ceramic-polymers tested demonstrated significantly rougher surfaces than a feldspathic porcelain (Vita Mark II, Vita) after toothbrush abrasion.[26]

The focus of the current study was to test extrinsic color longevity when normal tooth brushing was employed. Only two ceramic-polymer materials were evaluated in this study because currently very few companies of ceramic-polymer materials provide a proprietary stain and glaze kit. In addition, only one type of toothbrush and one type of toothpaste was used. There may be large variations in the amount of force that people use while brushing, the abrasiveness of the toothpaste, the softness of the toothbrush bristles, and the number of times per day individuals brush their teeth. Based on studies showing that humans have an average brushing force of 2-3 N, the toothbrush heads in this study were applied to the polished surface with a force of 3 N.[27] Manley determined the average daily brushing interval to be 19.72 strokes per surface per tooth resulting in the 7,300 strokes per simulated year used in this study.[19] The literature reports conflicting

information pertaining to the amount of brush strokes needed to simulate toothbrush abrasion. In various studies, 20,000 simulated toothbrush strokes were defined as the equivalent of 2 years or 7-10 years.[28,29] Based on different definitions, the simulation of 87,600 toothbrush strokes on the glass-ceramic and ceramic-polymer materials in the present study may therefore replicate from 8.75 to 43.75 years of general tooth brushing wear. Furthermore the impact of elements such as patient habits and diet must be further studied. Lastly systemic diseases causing elevated gastric acid levels may negatively affect extrinsic stains and glazes.[30] Therefore additional research is needed to better understand clinical significance of surface color changes demonstrated by ceramic-polymer materials in association with the various factors described.

CONCLUSION

The extrinsic stains on the ceramic-polymer materials may be more susceptible to change from tooth brushing compared to the lithium-disilicate material. However, the change may remain clinically imperceptible to the naked eye ($\Delta E^* > 1.0$) for nearly 6 equivalent years of brushing the ceramic-polymer materials.

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Materials	Mean ΔE* (st dev)			
	3 yrs	**6 yrs**	**9 yrs**	**12 yrs**
CeraSmart (GC)	0.7 (0.1) Aa	1.2 (0.1) Ba	1.8 (0.1) Ca	3.3 (0.2) Da
Enamic (Vita)	0.4 (0.04) Ab	1.1 (0.2) Ba	1.9 (0.4) Ca	3.1 (0.3) Da
IPS e.max CAD (Ivoclar Vivadent)	0.2 (0.1) Ab	0.2 (0.1) Ab	0.3 (0.1) Bb	0.8 (0.2) Cb

Table 1. Mean ΔE* (± one standard deviation) for each of the ceramic materials at each simulated time interval. Groups with the same upper case letter per row or lower case letter by column are not significantly different ($p>0.05$).

Figure Legends

Figure 1. Stained block and spectrophotometer insertion into custom printed alignment jig for recording L*a*b* values.

Figure 2. Mean change in ΔE* of surface stains after automated tooth brushing over time with the various ceramic materials.

Figure 1

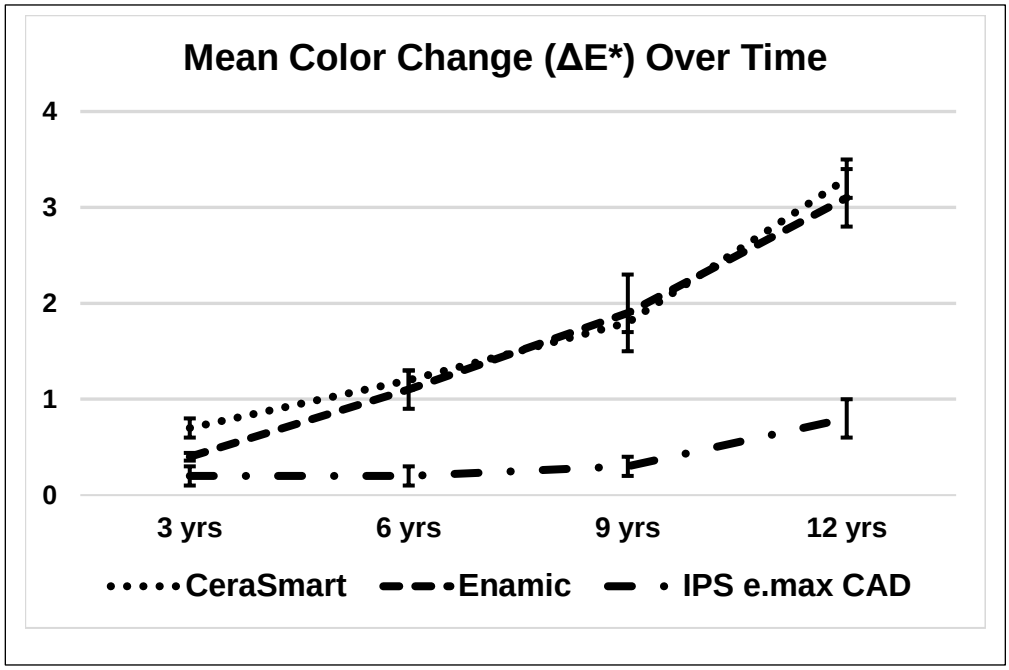
Mean Color Change (ΔE*) Over Time
4
3
2
1
0
3 yrs
6 yrs
9 yrs
12 yrs
CeraSmart
Enamic
IPS e.max CAD

Figure 2