

## Surface Discoloration of 3D Printed Resin-Ceramic Hybrid Materials against Various Stain Beverages

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### Abstract

This study investigated color ( $\Delta E_{00}$ ) and translucency ( $\Delta TP$ ) changed of 3D printing VarseoSmile Crown plus® and milling VITA Enamic® CAD/CAM hybrid material. The 126 rectangular specimens from each material were divided randomly (n=42) to immersed in distilled water, coffee, and cola.  $\Delta E_{00}$  and  $\Delta TP$  were computed before and after immersing at 1 week, 2 weeks, and 1 month using a spectrophotometer, following ISO/TR 28642:2016 standards. Two-way ANOVA followed by Tamhane post-hoc test and a one-way repeated measures ANOVA were performed (P=0.05). The result revealed that the types of material and beverages significantly affected  $\Delta E_{00}$  and  $\Delta TP$ , except for  $\Delta E_{00}$  in the first week was not significantly affected by types of material (P = 0.84). The  $\Delta E_{00}$  of VS showed more color change than EN after being immersed in coffee and cola at all time points. Also, the  $\Delta TP$  of VS shows more translucency change statistically significant than EN after being immersed in coffee and water at all time points. Coffee showed the highest discoloration of materials over 30 days. Time is a crucial factor increasing in discoloration. The results concluded that 3D printed resin-ceramic hybrid material prone to have more color and translucency change than milled resin-ceramic hybrid material.

Experimental article (J Int Dent Med Res 2022; 15(4): 1465-1471)

**Keywords:** Resin-ceramic hybrid material, 3D printing, milling, CAD/CAM, color change, translucency change.

**Received date:** 15 July 2022

**Accept date:** 14 August 2022

### Introduction

Nowadays, computer-aided design (CAD) and computer-aided manufacturing (CAM) system has been lightning development and it is becoming more general in dentistry. In the CAM system, there are subtractive (milling) and additive (printing) for restorative manufacturing.<sup>1</sup> Unfortunately, limitations of the motion range of cutting devices, size of cutting burs, unrecyclable of wasted material, and wear of milling tools seem to be problems in the milling system.<sup>1,2</sup> Causing the development of three-dimensional (3D) printing for dental applications to overcome the limitations of the milling system.<sup>3</sup> It is

processed by multiple layers of material that are added one by one layer under computer control to create the final desired 3D objects without wasting materials.<sup>1-5</sup>

The success of restoration depends not only on mechanical and physical properties but also on esthetic appearances. Restoration should feature excellent color match, together with high color and translucency stability during clinical service especially related to beverage stains.<sup>6-8</sup> Several studies show that resin-based restorations, as well as hybrid materials, are a tendency to discolor in an oral environment due to their part of the polymer matrix, filler particle size, and distribution more than other materials.<sup>6,7,9</sup>

Hybrid materials are an alternative dental esthetic restoration that has been improving in their properties to get better in both ceramic and composite resin.<sup>10-12</sup> It is worldwide available in a form of machinable mono blocks for milling but not for printing. Till late 2020, VarseoSmile

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Crown plus® (BEGO, Bremen, Germany) is the first ceramic-filled hybrid material that has been launched for 3D printed technology.<sup>13,14</sup> There is no publication on the color stability of this material so far, researchers are interested to examine the material's tendency to discolor. Thus, the purpose of this study was to investigate the effects of common beverages on the surface discoloration of 3D printed compared to milled resin-ceramic hybrid materials. The null hypotheses are (1) Translucency and color stability are not statistically different among hybrid materials in each staining beverage and point of time. (2) Translucency and color stability of each hybrid material are not statistically different among staining beverages at each point of time. (3) Translucency and color stability of each hybrid material and staining beverages are not statistically different in the differences point of time.

### Materials and methods

#### Specimen preparation

CAD/CAM resin-ceramic hybrid materials with different fabrication methods were selected for this study: VITA Enamic® (EN) represents the milling CAD/CAM system, and VarseoSmile Crown plus® (VS) represents the 3D printing CAD/CAM system. The materials and their specifications used in this in-vitro investigation are shown in Table 1.

The total sample size of 252 was estimated using G\*Power 3.1.9.3 software (Heinrich-Heine-Universität Düsseldorf, Germany), at significance level = 0.05, effect size = 0.25 and power = 0.95. 126 rectangular specimens size 12 x 14 x 1.5 mm ± 0.01 mm. of each material were prepared. For EN, mono-blocks were sliced with a micro-cutting machine (Accuton-50 wafer cutting machine, Struers, Ohio, United States). For VS, rectangular specimens were designed by 3D modeling software (Revit 2021, Autodesk, California, United States) and exported as a file in Standard Tessellation Language (.STL) format. The file was imported into the Asiga 3D printer software interface (Asiga Composer, Asiga, Sydney, Australia), and set the thickness of each printing layer to 50 µm. Then the specimens were printed with Digital light processing (DLP) 3D printer (MAX UV, Asiga, Sydney, Australia) with a 385 nm. ultraviolet light-emitting diode light

(high power UV LED). According to the manufacturer's instructions, ethanol was used to remove the uncured liquid resin. The final-curing process was achieved by light polymerization with protective gas for 2x1500 flash. (Otoflash G171-6, Whip mix, Kentucky, United States).

System	Hybrid Material	Composition	Manufacturer
Milling	VITA Enamic®-EN (Shade 2M2-T, Block size EM-14, lot No. 93140)	Aluminum oxide-enriched. Fine-structure feldspar matrix (86% wt, 75% vol) infused by a polymer material consisting of UDMA and TEGDMA (14%wt, 25% vol) (Polymer infiltrated ceramic) <sup>34, 37, 38</sup>	VITA Zahnfabrik Bad Sackingen, Germany
3D printing	VarseoSmile Crown plus®-VS (Shade A2 Dentin, liquid resin, lot No. 600236)	4,4'-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid. Silanized dental glass, methyl benzoylformate, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide. Inorganic fillers (particle size 0.7 µm) 30–50%wt (Ceramic filled hybrid material) <sup>14</sup>	BEGO, Bremen, Germany
Abbreviation UDMA = Urethane dimethacrylate TEGDMA = Triethylene glycol dimethacrylate			

**Table 1.** Restorative materials were used in the study.<sup>13</sup>

The specimen surfaces were polished by an automatic microprocessor-controlled machine for grinding and polishing (Struers, Ohio, United States) with sequential use of silicon carbide paper P1200 and P4000 grit15 (Water Proof SiC Paper; Struers, Ohio, United States) under running water with 5 N pressure at 300 rpm of polishing disk and co-rotation 150 rpm of holder plate for 60 seconds. The final thickness was controlled by a vernier caliper with 0.01 mm. resolution (Mitutoyo Corp, Kanagawa, Japan). The polished specimens were cleaned in an ultrasonic bath (GA008G-60W, Thai C.L.H., Bangkok, Thailand) with distilled water for 10 minutes and allowed to air dry at room temperature before testing.

Specimen immersion in beverage solutions

126 specimens of each material were randomly divided into 3 subgroups of 42 specimens immersed in 100 ml of water (control), coffee (NESCAFÉ Americano House Blend Ready-to-Drink Coffee, Nestlé), and Cola (Coca-cola original taste soft drink can, Coke). The specimens were placed in a specimen holder to avoid specimens overlapping while immersed in the solution. Storing in an incubator at 37 °C for 1 month with a condition of renewing the solution daily to avoid bacterial contamination and maintain their concentration and pH as well.

#### Color and translucency measurement

The color and translucency of specimens were measured and computed after surface

polishing (baseline) and immersion in solutions at 1 week (1w), 2 weeks (2w) and 1 month (1m), by using a spectrophotometer (VITA Easyshade® V, VITA Zahnfabrik, Bad Säckingen, Germany)

Before measurement, specimens were rinsed with distilled water for 10 seconds and dried with mild air pressure for 5 seconds. To reduce bias, the spectrophotometer was performed by a trained examiner and calibrated every time before being used in a tooth single-mode setting. To eliminate errors in color assessment, the area of measurement was performed under viewing boost with a specimen holder to control the measuring area exactly in the same region of every specimen. Moreover, affected conditions were set following ISO/TR 28642:2016 standards<sup>16</sup>, under CIE standard illumination D65 with light intensity 10,000 lux (controlled by digital lux meter (LX-1330B)), illumination and observation geometry for the reflectance measurements was a 45 and 0-degree optical configuration. The measurement was performed three times for each specimen to improve accuracy, and the average value was recorded. The change of color and translucency can be defined as a numerical comparison.

Color changes were calculated according to the CIEDE2000 definition ( $\Delta E_{00}$ ) against white backgrounds and determined by using the following formula.

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L^*}{K_L S_L}\right)^2 + \left(\frac{\Delta C^*}{K_C S_C}\right)^2 + \left(\frac{\Delta H^*}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C^*}{K_C S_C}\right) \left(\frac{\Delta H^*}{K_H S_H}\right)}$$

Where  $L^*$  represents lightness,  $a^*$  represents the point on a red-green scale, and  $b^*$  represents the point on a yellow-blue scale,  $C^*$  represents chroma, and  $H^*$  represents the hue angle. The change in color coordinates values were calculated from values before and after immersion and were shown as Delta ( $\Delta$ ),  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ ,  $\Delta C^*$ , and  $\Delta H^*$ .  $S_L$ ,  $S_C$  and  $S_H$  are the functions to calibrate the absence of visual uniformity of the CIELAB formula on the direction of lightness, chroma, and hue respectively.  $K_L$ ,  $K_C$ , and  $K_H$  were the correction parameters of the environment and were set at 1.<sup>17</sup>  $R_T$  is the rotation function, which indicates the interaction between chroma and hue differences in the blue region. The clinical significance of  $\Delta E_{00}$  was evaluated by using CIEDE2000 50:50% acceptability and perceptibility thresholds.<sup>16,18</sup>

The acceptability threshold was set at 1.8

$\Delta E_{00}$  units, and the perceptibility threshold was set at 0.8  $\Delta E_{00}$  units.<sup>10,12,19,20</sup>

Translucency was measured by using the translucency parameter (TP) method. The color parameters were recorded according to the CIELAB system against white and black backgrounds. The translucency parameter difference ( $\Delta TP$ ) was calculated from the difference of TP before and after immersion by using the following formula.<sup>6</sup>

$$TP = \sqrt{(L^*_w - L^*_B)^2 + (a^*_w - a^*_B)^2 + (b^*_w - b^*_B)^2}$$

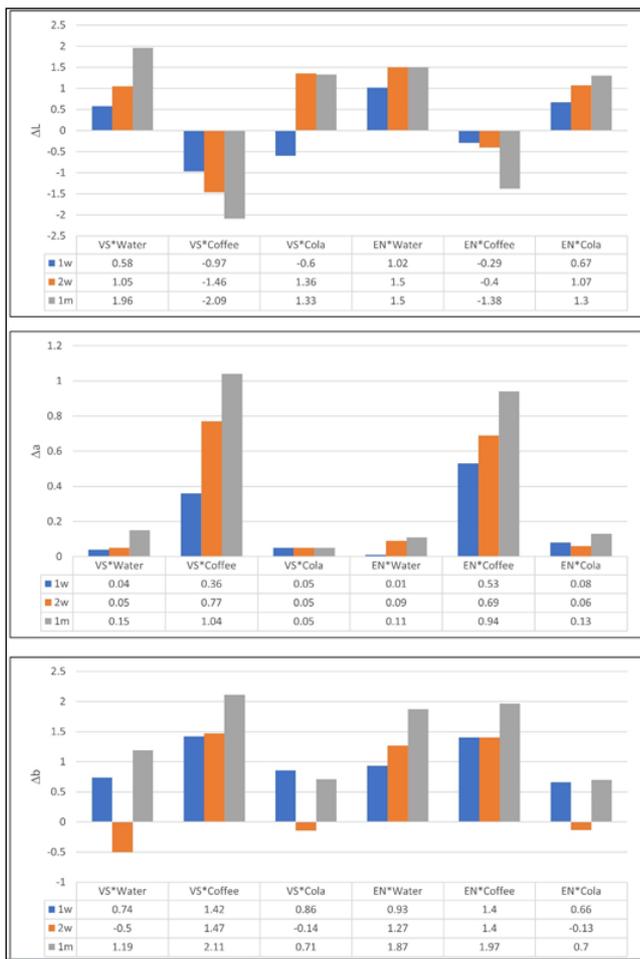
Where B and W represent color coordinates over the black and white background respectively. The translucency parameter value is between 0 to 100 where 0 indicates the complete opacity of the material, and 100 indicates a completely transparent material.<sup>21</sup>

#### Statistical analysis

$\Delta E_{00}$  and  $\Delta TP$  were statistically analyzed using SPSS (Statistical Package for Social Sciences, version 26.0; SPSS, Chicago, IL, USA) program. As the type of material and type of staining beverages are the main factor, the data were statistically analyzed by two-way and one-way ANOVA followed by Tamhane post-hoc multiple comparison test ( $\alpha = 0.05$ ). For the main factor of the time interval of immersion, one-way repeated ANOVA was performed ( $\alpha = 0.05$ ).

#### Results

The specimens were evaluated for  $\Delta E_{00}$  and  $\Delta TP$  when immersion in different beverage solutions at 1w, 2w and 1m. Two-way ANOVA revealed the types of material and staining beverages as well as the two-factor interaction between material and staining beverage effects on  $\Delta E_{00}$  and  $\Delta TP$  in all groups as shown in Table 2, except for  $\Delta E_{00}$  at 1w was not affected by types of material ( $P = 0.84$ ). The difference in mean and standard deviations (SD) of  $\Delta E_{00}$  and  $\Delta TP$  were statistically analyzed using one-way ANOVA (compare differences between materials and beverages) and repeated ANOVA (compare differences between the point of time) as shown in Table 3 and Table 4. The difference in color coordinates values over a white background,  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  are shown in Figure 1.



**Figure 1.** Change in lightness ( $\Delta L^*$ ), red-green scale ( $\Delta a^*$ ) yellow-blue scale ( $\Delta b^*$ ) over white background.

## Discussion

CAD/CAM resin-ceramic hybrid material is an alternative dental esthetic restoration that is available in a machinable form of milling and printing.<sup>13</sup> Resin-ceramic hybrid material has properties lie between ceramics and composites resin which physical properties are very close to those of natural teeth, causing less abrasion of antagonist's tooth and less wear than composite resin.<sup>11,22</sup> However, composite resin-composed materials, as well as hybrid materials, were more prone to discolor than other materials which limit the longevity and quality of esthetic restorations.<sup>6,7,9</sup> The color and translucency stability of materials is a crucial property and unacceptable discoloration of materials is a reason for the failure of the esthetic restoration.<sup>6-8</sup>

$\Delta E_{00}$  and  $\Delta TP$  are parameters representing the color and translucency change of materials which were affected by many factors such as types of material, types of staining intervention, exposure time, etc.<sup>6,9-12,23</sup> In this study, water, coffee, and Cola were chosen as beverage stain solutions because of the frequent daily intake. The main purpose of this study was to investigate the effects of common beverages on surface discoloration by comparing 3D printed and milled resin-ceramic hybrid materials when immersed in 3 common beverages (distilled water, coffee, and Cola) for 1w, 2w, and 1m. In the present study,  $E_{00}$  and color-coordinated parameters according to CIE  $L^*a^*b^*$  coordinate system were measured following ISO/TR 28642:2016 standard<sup>16</sup> by using analytical equipment to obtain more accurate, reproducible, and objective assessments than visual.<sup>7,23</sup> The VITA Easyshade<sup>®</sup>V was used to analyze because it had more than 90% reliability and validity as previously reported.<sup>11,24</sup> The result of the study showed that all null hypotheses were rejected.

Factors	P-value					
	$\Delta E_{00}$			$\Delta TP$		
	1w	2w	1m	1w	2w	1m
Materials (VS vs EN)	0.84	<0.01*	<0.01*	<0.01*	<0.01*	<0.01*
Beverages	<0.01*	<0.01*	<0.01*	<0.01*	<0.01*	<0.01*
Materials*Beverages	<0.001*	<0.01*	<0.01*	<0.01*	<0.01*	<0.01*

\*Significantly different at  $p < 0.05$

**Table 2.** The effect of materials, staining beverages, and interaction of material and staining beverage on  $\Delta E_{00}$  and  $\Delta TP$  at different times.

Materials	Beverages	Mean (SD) of $\Delta E_{00}$		
		1w	2w	1m
VS	Water	0.53(0.03)Aa	0.74(0.03)Ab	1.38(0.03)Ac
	Coffee	0.94(0.04)Ba	1.33(0.03)Bb	1.85(0.04)Bc
	Cola	0.72(0.04)Ca	0.95(0.05)Cb	1.04(0.05)Cb
EN	Water	0.82(0.02)Ba	1.14(0.02)Db	1.29(0.04)Ac
	Coffee	0.83(0.03)Ba	0.87(0.04)AcB	1.43(0.03)Ac
	Cola	0.55(0.02)Aa	0.72(0.04)Ab	0.93(0.03)Cc

Different uppercase and lowercase letters respectively represent statistically significant differences among groups (within columns, 1-way ANOVA) and time points (within rows, repeated ANOVA) ( $p < 0.05$ ).  
 Abbreviation:  $\Delta E_{00}$  = color change, VS = VarseoSmile Crown plus<sup>®</sup>, EN = VITA Enamic<sup>®</sup>

**Table 3.** Mean and standard deviations of  $\Delta E_{00}$  when immersion in different staining beverages and times.

When comparing  $\Delta E_{00}$  and  $\Delta TP$  between VS and EN, The  $\Delta E_{00}$  of VS was higher (more color change) than EN after being immersed in coffee and cola. The  $\Delta TP$  of VS was a more negative value (more translucency change to reduced translucent) and was statistically

significant than EN after being immersed in coffee and water. These may be explained by several reasons as followed (1) the compositions in filler and organic matrix (2) the manufacturer method and (3) the interface between the filler particle of VS and EN. (1) The difference in inorganic filler and organic matrix of VS and EN as the result of a study by Wojciech et. al<sup>13</sup>, the amount and size of filler of VS (19-24%vol., 430 nm to 3 µm) are lower than that of EN (75% vol., 1 µm to 11 µm). VS with a lower amount of filler is suspected to be unstable in color and translucency when exposed to extrinsic factors that might be attributed to the greater organic resin matrix volume allowing higher water sorption.<sup>7,11,25-28</sup> For EN, in addition to higher filler volume, UDMA, and TEGDMA are inorganic matrices that are less hydrophilicity and less water absorption than Bis-GMA may result in more resistance to color or translucency change.<sup>9, 12, 23</sup> However, to the best of our knowledge, there is no information on resin monomer type in VS due to the company have patents to protect detailed ingredient combinations. (2) the manufacturer method. For VS, layer in the microstructure of 3D printed that occurred based on the additive manufacturing method. Although 3D printing resins undergo post-curing processes after printing, their polymerization rates are relatively low.<sup>3</sup> Resulting in the remaining residual resin monomer on the surface from insufficient washing and the post-curing, which might lead to more susceptibility to staining.<sup>3,29</sup> Whereas, the EN is a polymer-infiltrated ceramic network (PICN) that is manufactured through a dense homogeneity microstructure.<sup>11-13</sup> (3) the interface between the filler particle.

According to the study of Wojciech et. al<sup>13</sup> found the SEM observation revealed bridging between filler particles of EN and not found in VS. This may result in an external stain at the interface between the filler of VS more than EN because of the binding of filler-matrix insufficiency.

The increase of  $\Delta E_{00}$  and more negative  $\Delta TP$  values of materials after immersion into water (no colorant as the control), coffee, and cola can be reasonably explained by the water absorption characteristics. Water absorption capable should be considered because if the material can absorb water, it also absorbs other fluids, such as coffee or cola, resulting in the discoloration of the material.<sup>7,8,27,28</sup> The result shows that the distilled water immersion affects to discoloration of both VS and EN by the positive value of  $\Delta L^*$  (lighter) and  $\Delta b^*$  (shift to yellow) may be due to intrinsic discoloration from debonding of the polymeric component. While coffee and cola affect to discoloration of both VS and EN may be due to intrinsic discoloration from debonding of the polymeric component and extrinsic discoloration from external stain colorants.<sup>11,30</sup> The study shows that coffee causes the highest discoloration of both VS and EN which is similar to the result of Lauvahutanon S et al., also Yerliyurt K. and Sarikaya I.<sup>6,26</sup> Coffee is composed of yellow colorant more than the others and can diffuse into the resin polymer matrix of hybrid ceramic.<sup>31</sup> Therefore it resulted in the highest positive value of  $\Delta a^*$  (shift to red),  $\Delta b^*$  (shift to yellow), and negative  $\Delta L^*$  (darker) of the materials as shown in figure 1. According to the result, both VS and EN in cola have relatively less color and translucency change compared to coffee at almost time point. Although cola had phosphoric acid and acidic pH that might damage the surface integrity of the materials as reported by many studies, it did not produce as much discoloration as coffee, which may be explained by the less of pigment in cola as previous report.<sup>7,8,31,32</sup>

The previous study reported that in vitro 7 days and 28 days of immersion in the staining solution can be represented for approximately 7 months (0.5 years) and 28 months (2.5 years), respectively.<sup>33</sup> Thus, this laboratory study evaluated immersion periods of 1w, 2w, and 1m for corresponding to the clinical situation aging of material at 0.5 years, 1.25 years, and 2.5 years, respectively. When immersion both VS and EN in

Materials	Beverages	Mean (SD) of $\Delta TP$		
		1w	2w	1m
VS	Water	-0.87(0.33)Aa	-0.94(0.29)Aa	-0.92(0.28)Aa
	Coffee	-0.45(0.21)Ba	-0.79(0.25)Ab	-1.26(0.31)Bc
	Cola	-0.18(0.35)Ca	-0.14(0.47)Bb	-0.07(0.43)Cc
EN	Water	-0.19(0.38)Ca	-0.14(0.28)Ba	-0.19(0.72)Ca
	Coffee	-0.37(0.14)BCa	-0.57(0.24)Cb	-0.87(0.24)Ac
	Cola	-0.38(0.23)BCa	-0.13(0.29)Bb	-0.13(0.33)Cb

Different uppercase and lowercase letters respectively represent statistically significant differences among groups (within columns, 1-way ANOVA) and time points (within rows, repeated ANOVA) ( $p < 0.05$ ).  
 Abbreviation:  $\Delta TP$  = translucency change, VS = VarseoSmile Crown plus®, EN = VITA Enamic®

**Table 4.** Mean and standard deviations of  $\Delta TP$  when immersion in different beverage solutions and time.

coffee and cola, the color change gradually increased as the period of immersion. The coffee and cola immersed specimen, as shown by the greater values of  $\Delta E_{00}$  and  $\Delta TP$  over time, also had the highest value in 1 month of immersion. Yellow colorant in coffee is a low polarity component that was released and absorbed gradually with time into the organic matrix part of the materials.<sup>31,34</sup> While, cola has more polarity caramel colorant than coffee that can adsorption only into the surface due to gradually matrix degradation by acidic pH (2.62) and adsorption.<sup>35,36</sup> The clinical significance of  $\Delta E_{00}$  was interpreted based on CIEDE2000 human perception threshold.  $\Delta E_{00} \leq 0.8$  represented excellent color stability (50:50% perceptibility threshold, PT) While,  $\Delta E_{00} > 0.08$  and  $\leq 1.8$  represented acceptability color stability (50:50% acceptability threshold, AT).<sup>10,12,19,20</sup> Besides the result, only VS immersed in coffee at 1m (approximately 2.5 years in vivo) has the unacceptable color change ( $\Delta E_{00} = 1.85$ ).

This study was in vitro laboratory conditions, color and translucency change of materials may effect by another environment in the oral cavity. Some of the clinical simulations were not included in this study, such as the cleaning effect by polishing or toothbrushing and the combined effect of various pH and thermal of other daily beverage stains, etc. And also, the available published data on newly introduced 3D printing resin ceramic hybrid material was limited. Therefore, further study of this material in many aspects should be performed.

## Conclusions

This study concludes that 3D printed resin-ceramic hybrid material prone to have more color and translucency change than milled resin-ceramic hybrid material. The discoloration gradually increases with time and the highest discoloration occurs when immersion of the material into the coffee.

## Acknowledgements

This study was funded and supported by the Thammasat University Research Unit in Restorative and Esthetic Dentistry and the Division of Restorative Dentistry, Faculty of Dentistry, Thammasat University.

## Declaration of Interest

The authors declare no conflict of interest.

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