

## ORIGINAL RESEARCH ARTICLE

# Color Stability and Surface Roughness of Resin Based Direct and Indirect Restorative Materials

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## Abstract

**Purpose:** This study investigated discoloration and surface roughness (Ra) of resin-based computer aided design (CAD)/computer aided manufacturing (CAM) blocks and direct and indirect resin composites after staining with coffee. **Materials & Methods:** Using a Teflon mold, 60 disc shaped specimens (8×2 mm<sup>2</sup>) were fabricated from direct (Estelite Asteria and GrandioSO) and indirect (Gradia Plus) resin composites, and 30 resin CAD/CAM specimens (12×14×2 mm<sup>3</sup>) were prepared from resin composite CAD/CAM blocks (Vita Enamic, Brilliant Crios, and Cerasmart) (n=10). A contact type profilometer was used to determine the Ra of all 60 polished samples. All specimens were thermocycled between 5 and 55°C for 3000 cycles, and immersed in coffee solution (37°C) for 1 and 7 days. A spectrophotometer was used to determine the color change ( $\Delta E_{00}$ ) with the CIEDE2000 formula after the specimens were re-polished. One-way analysis of variance (ANOVA) and Tukey multiple comparison test was performed to analyze the data (p<0.05). **Results:** The lowest degree of discoloration among the materials tested was found in Vita Enamic resin CAD/CAM block (p<0.001), whereas the highest degree of discoloration was found in the Gradia Plus indirect composite (p<0.001). There was no significant difference was seen between the initial surface roughness values (Ra) among the groups (p=0.249). No significant improvement was observed in the color of the groups except for the Cerasmart resin block after after polishing. **Conclusion:** Direct resin composites have a higher discoloration potential over resin-based CAD/CAM blocks, whereas they are less prone to discoloration than indirect resin composites

**Key words:** aesthetic dentistry; discoloration; resin-based CAD/CAM materials; roughness

## Introduction

A successful resin restoration should offer excellent wear resistance and simultaneously provide a satisfactory esthetic result. Despite advances in resin monomer and filler particle technology, discoloration in time in resin composites still constitutes a problem.<sup>1</sup> Changes in the color or translucency of the restorations caused by intraoral conditions or drinks cause loss of the esthetic properties of the restorations. There are many studies on the coloring of composite materials, but there are few studies comparing the color stability properties of CAD/CAM resins and other composite materials.<sup>2</sup> For this purpose this study aims to determine the initial surface roughness (Ra) of resin-based CAD/CAM blocks, detect changes in color after exposure to aging and the staining solution ( $\Delta E$ ), and evaluate

the degree of discoloration removal after polishing.

With advances made in the field of computer aided design CAD/CAM technology, manufacturers have fabricated resin based ceramic restorative materials that combine the advantages of composites and ceramics.<sup>3</sup> Hybrid ceramics, polymer infiltrated ceramic network materials, and resin nano ceramic materials are currently available.<sup>4</sup> These materials, unlike ceramic restorations, do not require firing in the finishing and polishing process, and restoration can be completed in a single visit after manual polishing or light curing with resin glaze.<sup>5</sup> Although their mechanical and esthetic properties are not as superior as ceramic restorations, ease of manufacturing and repair makes these materials preferable.<sup>6</sup> Owing to their strong polymer crosslinks that form under high temperature and pressure, resin-based CAD/CAM blocks are considered to have su-

perior properties over direct and indirect laboratory composites.<sup>7</sup> However, like direct and indirect composites, these materials may be sensitive to discoloration when exposed to changes in temperature, humidity, beverages containing coloring pigments, and smoking.<sup>8</sup> In the literature, limited number of studies have investigated discoloration and surface roughness (Ra) of resin-based CAD/CAM blocks. However, it is important to know the advantages and disadvantages of these up-to-date materials compared to resin composites that are frequently used in clinical conditions. For this purpose, we investigated the color changes and the surface roughness of these materials in our study.

The null hypothesis of this study were that; there is no difference (1) between the Ra of direct and indirect resin composites and resin-based CAD/CAM blocks, (2) between the discoloration sensitivity of direct and indirect resin composites, and resin-based CAD/CAM blocks, (3) among three materials in terms of discoloration removal after polishing.

## Materials and Methods

For this study, three resin-based CAD/CAM blocks 1- Vita Enamic (Vita Zahnfabrik, Bad Säckingen, Germany), 2- Brilliant Crios (Coltene/Whaledent, Langenau, Germany), 3- Cerasmart (GC, Leuven, Belgium), two direct resin composites 1- Estelite Asteria (Tokuyama Dental Corporations, Tokio, Japan), 2- GrandioSO (Voco, Cuxhaven, Germany), and one indirect resin composite (Gradia Plus, GC, Leuven, Belgium) were selected. Technical characteristics of the materials and their manufacturers are shown in Table 1.

Specimen size was calculated using a power analysis program ( $\alpha=0.05$  and  $\beta=0.80$ ), and from each group, 10 specimens were prepared to afford a total of 60 specimens. Disc-shaped direct and indirect resin composite specimens with the diameter of 8 mm and the thickness of 2 mm were prepared using a Teflon mold (n=10, per group). To secure smooth composite surfaces, the specimens were polymerized for 20 s using a light-emitting diode curing unit (DTE LUX-E Plus, Guilin Woodpecker Medical Instrument, Guilin, Guangxi, China; 1200 mW/cm<sup>2</sup>) over a 1-mm glass slide after placing transparent strips at the top and bottom of the Teflon mold. CAD/CAM specimens (n=10, per group) of 12×14×2 mm<sup>3</sup> were cut out from blocks using a diamond bladed water-cooled low speed precision cutting device (Microcut 201 High Speed Precision Cutter, Metkon, Bursa, Turkey) (n=10). The specimens were then cleaned using an ultrasonic cleaning device (VGT-1740, Guang Dong, China) The all specimens were stored for 24h in distilled water at 37°C to complete the polymerization of the composite. As per the previous studies, the water-cooled specimens were grinded using a P600-to-P1200 silicon carbide grit for 30s (Metkon; Gripo 2v Grinder-Polisher, Bursa, Turkey). The specimens were cleaned for 10min with distilled water in an ultrasonic cleaner and dried.<sup>9</sup> The specimen thickness was determined by a digital micrometer (Marcal 16 ER, Perthen, Mahr, Germany). Additional finishing and polishing procedures were applied to the specimens. A two-step rubber polishing wheel kit (Clearfil Twist Dia, Kuraray, Tokyo, Japan) was used by applying low pressure for 20s below 10,000 rpm. The specimens were then polished using a diamond polishing paste (Diapolisher Paste; GC Dental Products Europe) and a cotton brush (Renfert GmbH, Hilzingen, Germany) at a speed of 10,000 rpm for 15 s with low pressure. After polishing, the specimen thickness was measured for standardization purposes, and an average reduction of 0.15 mm was recorded (2±0.15 mm). Each tool used was replaced with a new one after every 5 specimens. After manual polishing, the specimens were cleaned in an ultrasonic cleaner with distilled water for 10min and dried. All

procedures were performed by the same expert. Using a contact type profilometer, three measurements were taken from the center of each specimen to obtain Ra values after polishing (Mahr Perthometer SD 26, Perthen, Mahr, Germany). Ra values were recorded at a sample length of 1.75 mm with a cut-off value of 0.25. All specimens were thermocycled between 5 and 55°C for 3000 cycles with 60 s dwell time.

As the staining agent, a coffee solution was prepared with 2 g of instant coffee powder dissolved in 200 ml of distilled boiled water (Nescafé Classic; Nestlé). No cream and sugar were added to the mixture. After the coffee solution cooled down to room temperature, it was poured into opaque stainless-steel containers, and specimens were placed in the containers away from each other. To avoid physical contact, each group was placed in a completely permeable holder and the specimens were kept in a vertical position during 24 h for 7 days.<sup>10</sup> The coffee solution was daily changed over 7 days. After the samples were removed from the coffee solution, they were washed with distilled water for 10 s and dried using an air water spray. Then the specimens were polished with two-step rubber polishing wheel kit (Clearfil Twist Dia, Kuraray, Tokyo, Japan) at a low pressure for 20 s below 10,000 rpm. Then the specimens were polished using a diamond polishing paste (Diapolisher Paste; GC Dental Products, Tokyo, Japan) and a cotton brush at a speed of 10,000 rpm for 15 s with low pressure. After this final polishing step the degree of discoloration removal in the specimens were examined with a spectrophotometer (VITA Easyshade V 4.0®; VITA Zahnfabrik, Bad Säckingen, Germany) and values were recorded.

To measure changes in color, a spectrophotometer was used before each measurement, the device was calibrated on a standard white calibration surface. Three measurements were taken for each specimen and the average values of the L\*, a\*, and b\* data were calculated. The color differences ( $\Delta E_{00}$ ) in the 3-dimensional L\*, a\*, and b\* color space were calculated after thermocycling, after 1 and 7 days of immersion, and after polishing, in comparison to the initial color with the CIEDE2000 formula.<sup>11</sup>

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right) \left(\frac{\Delta H'}{K_H S_H}\right)} \quad (1)$$

For this study, each  $K_L$ ,  $K_C$ , and  $K_H$  were set to 1.0. The clinically accepted 50%:50% color change threshold was determined at  $\Delta E_{00}=1.8$ .<sup>12</sup>

## Statistical analysis

Statistical analysis of the data was performed using the statistical software SPSS 22.0 (SPSS Inc.). Normality test (Kolmogorov-Smirnov Normality Test) was applied to the surface roughness and color change data. Parametric tests were preferred as the data showed normal distribution. Two-way analysis of variance (ANOVA) and Tukey multiple comparison method were deployed to evaluate initial Ra values, thermocycling, 1 and 7 days after immersion in the staining solution, and color change values after the polishing (p<0.05).

## Results

No statistically significant difference was found between groups in terms of initial Ra values of direct and indirect resin composites and resin-based CAD/CAM blocks, as seen in Table 2 (p=0.249). The color changes in the direct and indirect resin composites and the resin-based CAD/CAM blocks ( $\Delta E_{00}$ ) are shown in Figure 1. Brilliant Crios ( $\Delta E_{00}=0.6$ ) and Vita Enamic

**Table 1.** Resin restorative materials used in the study.

Type	Brand	Manufacture	Shade	Batch Number	Monomer	Filler	Filler ratio (%)
Resin composite nanoceramic	Cerasmart	GC, Leuven, Belgium	A2HT	14092	Bis-MEPP, UDMA, DMA	Silica (20 nm), barium glass (300 nm)	71
Polymer infiltrated ceramic network (PICN)	Vita Enamic	Vita Zahnfabrik, Bad Säckingen, Germany	2M2	39440	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86
Reinforced resin composite	Brilliant Crios	Coltene/Whaledent, Langenau, Germany	A2HT	G46616	Cross-linked methacrylates, Bis-GMA, BIS-EMA, TEGDMA	barium glass, amorphous silica particles	71
Supra-nano spherical hybrid composite	Estelite Asteria	Tokuyama Dental Corporations, Tokio, Japan	A2B	W128	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	Silica-zirconia spherical (200 nm) filler, composite filler	82
Nano-hibrit composite	GrandioSO	Voco, Cuxhaven, Germany	A2	1029391	Bis-GMA, Bis-EMA, TEGDMA	0.5–10 µm particles; 20 nm particles	89
Microhybrid (Indirect composite)	Gradia Plus	GC, Leuven, Belgium	LE Light Enamel	190917A	UDMA, dimethacrylate,	SiO <sub>2</sub> , fumed SiO <sub>2</sub> , Sr and lanthanoid F, Al F silicate (prepolymerized)	71

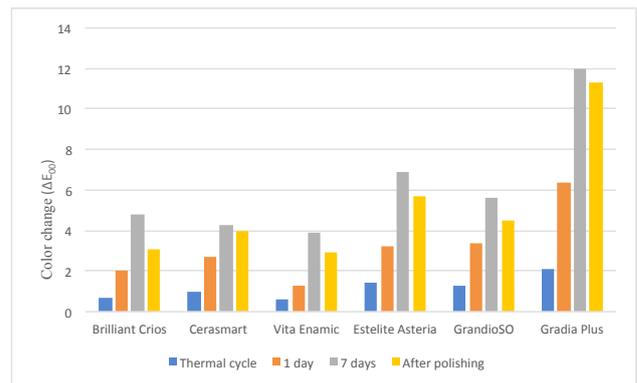
Abbreviations: Bis-EMA; ethoxylated bisphenol-A dimethacrylate, Bis-GMA; bisphenol-A diglycidylether methacrylate, Bis-MEPP; 2,2-bis (4 methacryloxy-polyethoxyphenyl) propane, TEGDMA; triethylene glycol dimethacrylate, UDMA; urethane dimethacrylate, bis-MPEPP; bisphenol-A polyethoxy methacrylate.

( $\Delta E_{00}=0.6$ ) showed the minimum change in color after the thermocycling, and the maximum color change was seen in the Gradia Plus group ( $\Delta E_{000}=2.3$ ) (Table 3).

In terms of discoloration in restorative materials after 1 and 7 days immersion in the staining solution, Vita Enamic showed the least color change among the CAD/CAM blocks. However, no significant difference was present between Vita Enamic and the other two CAD/CAM blocks, namely Brilliant Crios, and Cerasmart (Figure 1) ( $p<0.001$ ). After 1 and 7 days immersion, direct resin composites showed more color change than resin based CAD/CAM blocks ( $p<0.001$ ), while the discoloration was less over the indirect resin composites ( $p<0.001$ ). Among the resin composite groups, the supra-nano resin composite (Estelite Asteria) showed more color change than the nanohybrid resin composite (GrandioSO) (Table 3).

**Table 2.** Initial surface roughness values (Ra) of samples.

Material	Roughness Values (Mean±SD)	P
Brilliant Crios	0.149±0.19	0.249
Cerasmart	0.158±0.29	
Vita Enamic	0.151±0.12	
Estelite Asteria	0.165±0.38	
GrandioSO	0.132±0.38	
Gradia Plus	0.162±0.44	

**Figure 1.** Color differences ( $\Delta E_{00}$ ) of materials

Polishing led to significant color improvement in all groups except for Cerasmart ( $p<0.001$ ). The best color correction among groups was seen for Vita Enamic and Brilliant Crios, while the least color improvement was seen for Gradia Plus (Table 3).

**Table 3.** Color difference ( $\Delta E_{00}$ ) values (Mean $\pm$ SD) of tested materials after thermocycling, storage in coffee solution for 1 and 7 days and after polishing ( $\Delta E_{00} = 1.8$ ).

Material	After Thermocycling	1 Day	7 Days	After polishing
Brilliant Crios	0.6 $\pm$ 0.2 <sup>a,A</sup>	1.7 $\pm$ 0.2 <sup>a,B</sup>	4.8 $\pm$ 0.2 <sup>ab,C</sup>	3.1 $\pm$ 0.9 <sup>ab,D</sup>
Cerasmart	1 $\pm$ 0.7 <sup>ab,A</sup>	1.3 $\pm$ 0.4 <sup>ab,A</sup>	4.3 $\pm$ 1.1 <sup>a,B</sup>	4 $\pm$ 0.4 <sup>bc,B</sup>
Vita Enamic	0.6 $\pm$ 0.4 <sup>a,A</sup>	1 $\pm$ 0.3 <sup>b,A</sup>	3.9 $\pm$ 0.2 <sup>a,B</sup>	2.9 $\pm$ 0.6 <sup>a,C</sup>
Estelite Asteria	1.3 $\pm$ 0.2 <sup>b,A</sup>	3.1 $\pm$ 0.6 <sup>c,B</sup>	6.9 $\pm$ 0.6 <sup>c,C</sup>	5.7 $\pm$ 0.6 <sup>d,D</sup>
GrandioSO	0.7 $\pm$ 0.1 <sup>ab,A</sup>	2.3 $\pm$ 0.4 <sup>c,B</sup>	5.6 $\pm$ 0.5 <sup>b,C</sup>	4.5 $\pm$ 0.8 <sup>c,D</sup>
Gradia Plus	2.3 $\pm$ 0.5 <sup>c,A</sup>	5.1 $\pm$ 0.4 <sup>c,B</sup>	12 $\pm$ 1.1 <sup>d,C</sup>	11.3 $\pm$ 1.2 <sup>e,C</sup>

\*A-D shows column comparisons, a-e shows statistical difference between lines  $p < 0.001$ .

## Discussion

Since no statistically significant difference was present between the initial Ra values of the specimens, the first null hypothesis of the study was accepted. However, since resin-based CAD/CAM blocks showed less discoloration than direct and indirect resin composites, the second null hypothesis of the study was rejected. The third null hypothesis of the study was also rejected as statistically significant differences were present between the specimens in color correction after polishing.

Free radicals of monomer in resin composites tend to react with oxygen during the polymerization of resin restorations. This results in the formation of nonreactive peroxide radicals, and an “oxygen inhibition layer” is created on the surface of the composite, which needs to be removed.<sup>13</sup> To remove this layer, the specimens in this study were initially polished with a two-step rubber polishing wheel kit and a diamond polishing paste. The critical value for the average Ra values is 0.2  $\mu\text{m}$ , and above this level, the bacterial colonization increases.<sup>14</sup> In studies examining the effect of polishing systems on the surface roughness of different composites, it has been reported that there is no difference.<sup>15</sup> Similarly, in the present study, no statistically significant difference was present between initial Ra among groups after polishing, and the Ra in all materials was below the critical value of 0.2  $\mu\text{m}$ .

In this study, CIEDE2000 formula was used for color measurement before and after coloring in the colorant solutions of the samples. It has been found that the CIEDE2000 formula performed better than the CIELab formula in measuring the changes in color as perceived by the naked eye and that it is frequently preferred in clinical observation.<sup>16</sup> According to the CIEDE2000 system, changes in color are caused by shifts in lightness, chroma, and hue, and these parameters are the most affected by thermocycling and immersion in the staining solution. Thermocycling causes hydrolytic and thermal decomposition in resin composites when they are subjected to repeated and sudden temperature changes.<sup>17,18</sup>

Tea and coffee are known to cause discoloration in dental composites. Tea is rich in tannins, and coffee contains chromogens.<sup>19,20</sup> Previous studies have found that the average consumption time of a cup of tea or coffee is 15 min.<sup>21</sup> Assuming that an average individual consumer of tea or coffee drinks a total of three cups of either of the beverages, keeping specimens in the staining solution for 7 days will correspond to 7 months of daily consumption of these drinks in real life. In another study, different resin composite samples to thermocycling (between 5 and 55  $^{\circ}\text{C}$ ; 3000 cycles) and stored the specimens in coffee solution for 1 and 7 days. At the end of day 1, visible color change was seen on the composite samples, and the discoloration significantly increased on day 7 of immersion.<sup>10</sup> Similarly, this study also observed significant discoloration in composite specimens after the samples were thermocycled (between 5 and 55 $^{\circ}\text{C}$ , 3000 cycles) and immersed in the coffee solution for 1 and 7 days. Similar to the results of the study, herein,

samples with resin content were discolored in the coffee solution on day 1, and the discoloration significantly increased on day 7 ( $\Delta E_{00} > 1.8$ ).

Furthermore, it has been found that resin-based CAD/CAM blocks are less prone to discoloration by red wine than direct and indirect resin composites.<sup>22</sup> Similarly, in this study, resin CAD/CAM specimens exhibited lower color change values than direct and indirect resin composites. Moreover, on day 1, for all specimens, except for resin based CAD/CAM samples, the color change values were over the acceptable threshold ( $\Delta E_{00} > 1.8$ ). On day 7, the discoloration values for all specimen groups were higher than the acceptable threshold of  $\Delta E_{00} = 1.8$ . Lower overall discoloration values in resin-based CAD/CAM blocks can be explained by the high levels of crosslinks, reduced porosity defects, and more homogeneous polymerization of resin CAD/CAM blocks.<sup>22</sup>

No significant difference reported in  $\Delta E$  values of resin-based CAD/CAM blocks investigating the discoloration of resin based CAD/CAM blocks (Shofu HC Block, LavaTM -Ultimate, and Vita Enamic).<sup>23</sup> Similarly, in this study, no significant difference was observed in color change values among resin CAD/CAM specimens. It has been showed that the discoloration may be associated with water absorption. Water acts as a penetration vehicle for staining agents in the process of water sorption of the material, and in fact, the discoloration occurs in the first week after the contact of the restoration with water.<sup>24,25</sup>

In a study investigating two nano-hybrid fillers containing resin matrix (Cerasmart and LavaTM -Ultimate) and a polymer-infiltrated ceramic-network material (Vita Enamic); Vita Enamic exhibited the lowest degree of water absorption.<sup>26</sup> When Vita Enamic and Cerasmart, tested together with Lava Ultimate containing Bis-GMA, Lava Ultimate showed the highest water absorption.<sup>27</sup>

Direct and indirect resin composites containing Bis-GMA show higher water absorption than those containing UDMA and TEGDMA. Most of the resin-based CAD/CAM blocks contain UDMA and TEGDMA, but not Bis-GMA.<sup>28</sup> Therefore, we believe that high water absorption and discoloration associated with water absorption may be due to Bis-GMA. In this study, the polymer-infiltrated ceramic-network Vita Enamic, which does not contain Bis-GMA, exhibited the lowest degree of discoloration.

In a study which three resin composites tested, showed that the ones with lower filler ratio were more sensitive to discoloration.<sup>29</sup> Similarly, in our study, the supra-nano composite Estelite Asteria (82 %) was colored more than the GrandioSO composite (89 %) due to the low filler ratio. This, we believe, is due to the higher Ra value of Estelite Asteria. This shows that Ra values contribute to extrinsic discoloration. However, according to the results of the present study, the correlation between Ra values and discoloration does not hold for each and every resin material. Ra is not the only factor involved in the discoloration of resin composites; the degree of discoloration also hinges on the physicochemical properties of the

material.<sup>30</sup>

Previous studies have shown that extrinsic discoloration of resin-based CAD/CAM materials can be reversed by an acceptable degree by finishing and polishing applications.<sup>31</sup> It has been reported significant improvement in stain removal in CAD/CAM blocks after polishing eight CAD/CAM blocks and four resin composites with prophylaxis paste following immersion in a coffee solution for 1 day, 1 week, and 1 month.<sup>32</sup> In this study, a two-step rubber polishing wheel kit and a diamond polishing paste were applied on the specimens to remove the discoloration caused by the coffee solution. A significant improvement in stain removal was observed for all the groups except for the resin-based CAD/CAM block Cerasmart. However, the discoloration was not reduced the values below the acceptable threshold of  $\Delta E_{00} = 1.8$  in any group. Resin based CAD/CAM blocks Vita Enamic and Brilliant Crios showed the best color correction, while Gradia Plus showed the lowest degree of improvement. Intrinsic discoloration in hybrid restorative materials does not penetrate as deeply as in composite materials. Therefore, hybrid resin-based CAD/CAM blocks better afforded the closest result to the initial color than the composite materials.

Today, the problem of discoloration in restorations has not been solved yet. This study was done to help choosing of materials according to the patient and personal habits. Considering the study's results, for individuals who have the habit of using coloring products such as tea, coffee, wine, and cigarettes, the use of CAD/CAM materials may be a priority when choosing materials. We can conclude that when the necessary oral hygiene is provided, the formation of external stains can be prevented to a great extent. In addition, we can conclude that the formation of extrinsic stains can be prevented to a great extent when the necessary oral hygiene is attained.

This study has some limitations. This is an in vitro study, and the staining solution contacted both sides of the specimens; moreover, the materials tested here can be considered to have simulated a Class 3 restoration, which corresponds to the largest surface area in the mouth. Future studies may be planned with longer immersion periods and longer dwell times for thermocycling.

## Conclusion

Within the limitations of this study, the following conclusions can be drawn.

1. No significant difference was present between the initial roughness (Ra) values among the groups.
2. Resin based CAD/CAM blocks were less discolored than both direct and indirect resin composites. Vita Enamic showed the lowest degree of discoloration, and Gradia Plus showed the highest discoloration.
3. After final polishing, a significant improvement was observed in all groups except for the resin-based CAD/CAM block Cerasmart.

## Author Contributions

BE and NA participated in designing the study. SK and NA participated in generating the data for the study. NA and BE participated in gathering the data for the study. NA participated in the analysis of the data. BE wrote the majority of the original draft of the paper. SK, NA and EAO participated in writing the paper. All authors approved the final version of this paper.

## Conflict of Interest

The authors declare no competing interests.

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