



## Staining Susceptibility of Dental Composite Resins with Various Nano-Filler Technologies

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### ABSTRACT

**Objectives:** The purpose of this study was to evaluate the color stability of polished composite resin material with different filler technologies.

**Materials and Methods:** Three composites were studied; Filtek™ Z350 XT (3M, ESPE, St. Paul, USA) [FXT] that has nano-cluster filler particles, Brilliant EverGlow™ (Coltene/Whaledent® AG, Altstätten, Switzerland) [BEG] which consists of submicron barium glass fillers and pre-polymerized fillers and Ceram.X® Sphere TEC™ (Dentsply, Konstanz, Germany) [CXS] with advanced granulated filler technology composite. Twenty standardized composite discs were prepared with each composite resin, and it was polished with Sof-Lex disks (3M, ESPE, St. Paul, USA). The baseline color was then recorded using a spectrophotometer (X-Rite PANTONE® iPro-2, Michigan). Eight samples from each group were then subdivided and immersed into freshly prepared solutions of turmeric, coffee, and four samples into distilled water for a total period of 3 hours/day for 30 days. Following this, the color was again recorded. The change in color ( $\Delta E$ ) was calculated, and the data obtained were subjected to Kruskal Wallis and Mann Whitney test.

**Results:** A significant difference was seen between the staining characteristics of CXS and FXT. The mean color change in the values was highest in Ceram.X that was clinically unacceptable ( $\Delta E > 3.3$ ).

**Conclusions:** Though all composites revealed color changes after their immersion into the staining solutions, the amount of stain varied based on their constituents and filler characteristics.

**Keywords:** Color, Composite Resin, Filler Particles, Spectrophotometer, Staining.

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### Introduction

Beauty and aesthetics have been one of the most important concerns of people for many centuries. With this increasing demand for aesthetics in the field of smile designing/dentistry, the invention of composites has revolutionized treatment modalities. Composite resin has enabled dentists to perform restorations for patients on not only the anterior teeth but also on the posterior teeth with utmost perfection and ease. However, the color stability of these materials has been one of the most important criteria in determining the longevity and the success of restorations.<sup>1</sup> The quality of the resin restorations has improved greatly with the advent of new technologies in recent years. Composite resin has benefited with the development of smaller particle sizes, better bonding systems, curing refinements, and sealing systems, but color stability remains as an inherent challenge to the material.<sup>2</sup>

The discoloration of dental composites has a wide variety of reasons, both intrinsic and extrinsic. Whereas recent studies have shown no significant color changes in completely polymerized resins due to intrinsic factors post water storage.<sup>3</sup> Hence, discoloration due to extrinsic factors becomes the most significant one, although the etiology is multifactorial, staining due to coffee, tea, turmeric is a few of the causative factors being widely accepted in day-to-day life.<sup>3</sup> This discoloration is mainly caused by the adsorption of the colorant from the staining solutions or beverages on the resin-based restorations. This adsorption occurs mainly due to the surface roughness of a material, the types of resin matrix, and the dimensions of the filler particles. Discoloration of composite resins has been largely found as one of the major reasons for its replacement.<sup>4,5</sup>

Various composite resins have been developed with different physical properties mainly based on the filler particle size. The traditional ones being macrofilled, microfilled, small particle, and hybrid.<sup>6</sup> The newer ones in the market are nanocomposites. These materials are very versatile as they could be used for restoring both anterior and posterior teeth because of their better strength, handling, wear properties, and polishability.<sup>7-9</sup> Nanocomposites can be broadly sub-classified as nano-filled and nano-hybrid based on the filler technology used. Nano-filled composite resin consists nanoparticles and nanocluster filler particles. Whereas nano-hybrid composite contains larger filler particles in addition to the nanoparticles.<sup>10,11</sup> Various nano-hybrid composites are marketed with diverse filler technologies. The discoloration potential of these composites may vary depending on this.<sup>12</sup>

Aim of this study was to evaluate the color stability of various nanocomposite resin restorations having different filler particle technologies on exposure to staining agents.

## Materials and Methods

### Sample Preparation

Three composites that differ in their filler particle size and structure were selected for the study (Table 1). A total of 60 disks were made using a brass mold with a disk size of 2 mm height and 10 mm diameter. Twenty samples (n=20) were prepared from each of the composite resin groups using the mold. The mold was placed on a mylar strip and overfilled with composite resin, the top of the brass mold was again covered with a mylar strip and a glass slide. The composite was then polymerized completely for 20 seconds on one side and 20 seconds on the opposite side (LED- D Woodpecker Curing Light, 850mW/cm<sup>2</sup> -1000 mW/cm<sup>2</sup>). One side of the composite samples was polished using Sof-Lex disks as per manufacturer instructions (3M ESPE, St. Paul, MN, USA).

The non-polished side was marked with an indentation. The samples were stored in distilled water for 24hrs to ensure complete polymerization.

### Staining Procedure

Twenty samples from each group were randomized into three subgroups. Those in subgroup A were immersed in staining solutions of turmeric. It was prepared by mixing 0.5g of turmeric (Everest, S. Narendra Kumar, and Co., Mumbai, India) in 500ml boiling water, simmer for 5 mins, and filtered. Samples in subgroup B were immersed in the coffee solution. This solution was prepared by adding 15g instant coffee powder (Bru, Brook Bond label, Hindustan Unilever, Ltd., Mumbai, India) in 500ml boiling water, simmer for 5 mins, and filtered. Samples in subgroup C were immersed in distilled water, acted as the control group. The samples were immersed in vials containing staining solution in a water bath at 37°C for 3 hours following which the samples were washed and kept in the water bath till the next day. Before immersion, the samples were taken out of the water bath and blotted with blotting paper to prevent dilution and then immersed again for 3 hours. The staining solutions were freshly prepared every day, and this process was repeated for 3 hours/day for 30 days. (Figure 1).

### Staining Characteristics

The color of the samples was recorded at day 1 (T<sub>1</sub>) before their immersion in the staining solutions, and finally, on Day 30 (T<sub>30</sub>). The measurements were recorded using X-Rite i1 Pro Spectrophotometer with a Profile maker pro 5.0.1 software with D65 as the standard illumination under natural daylight conditions. The instrument was calibrated before use according to the manufacturer's instructions. Three readings were taken for each specimen at specific areas marked, and the mean of all the values, L\*, a\*, and b\* were calculated.

Table 1. Composite resin materials used in the study

Composite resin	Code	Shade	Filler technology	Composition	Filler Volume (%)
Filtek™ Z350 XT (3M, ESPE, St. Paul, USA)	FXT	A2 Body	Nanocluster filler particles (Nano-filled)	bis-GMA, bis-EMA, UDMA, with small amount of TEGDMA Aggregated zirconia/silica cluster filler (0.6 -10µm); Silica (20nm); Zirconia (4-11nm)	63.3
Ceram.X® Sphere TEC™ (Dentsply, Konstanz, Germany)	CXS	A2	Advanced granulated nano-ceramic filler technology (Nano-hybrid)	Methacrylate modified polysiloxane Dimethacrylate resin Barium boron aluminum silicate glass (1.1- 1.5µm), Silicon dioxide (2-10nm)	59-61
Brilliant EverGlow™ (Coltene/Whaledent® AG, Altstätten, Switzerland)	BEG	A2	Submicron barium glass fillers, and pre-polymerized fillers (Nano-hybrid)	bis-GMA, TEGDMA, bis-EMA Pre-polymerized filler containing dental glass and nano-silica Colloidal nano-silica aggregated and non-aggregated Barium glass (20-1500 nm)	64

bis-GMA- Bisphenol A-glycidyl methacrylate; TEGDMA- Triethylene glycol dimethacrylate; bis-EMA- Bisphenol A-ethoxylate dimethacrylate; UDMA- Urethane dimethacrylate

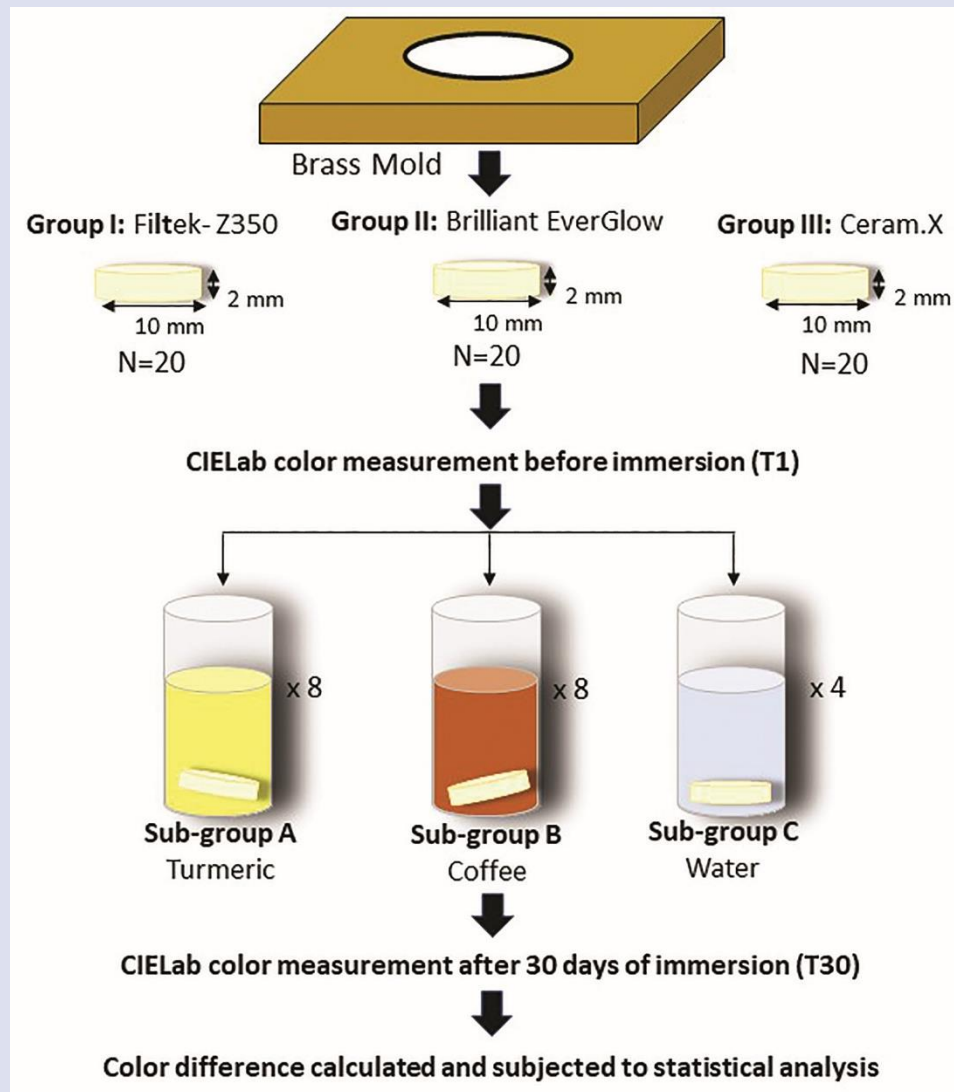


Figure 1. Schematic diagram of the experimental design

The  $L^*$  value mainly refers to the lightness or the darkness of the object and ranges from 0 to 100, 0 being a perfect black to 100 being white.  $a^*$  values show the shift between the color red and green. Positive  $\Delta a^*$  value shows a shift towards red, whereas negative value points towards a shift to green. On the other hand,  $b^*$  value gives a range from yellow to blue, positive  $\Delta b^*$  value indicates a change towards yellow and negative towards blue.<sup>13</sup>

The total color difference ( $\Delta E$ ) that is the variation between the color at baseline, and after 30 days for each disk sample was calculated using the following equation:  $\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$ ; where  $\Delta L^* = (L^*_{30} - L^*_1)$ ,  $\Delta a^* = (a^*_{30} - a^*_1)$ , and  $\Delta b^* = (b^*_{30} - b^*_1)$ .

### Statistical Analysis

The change in color ( $\Delta E$ ) after 30 days was calculated, and the data obtained were subjected to Kruskal Wallis and Mann Whitney test using statistical software (SPSS for Windows, Version 11.0.0, SPSS Inc., Chicago, IL, USA). A p-value of less than 0.05 was set as statistically significant.

### Results

The overall color change of the composite specimens in various staining solutions is shown in Table 2.  $\Delta E$  value, which is obtained with the combination of  $L^*$ ,  $a^*$ , and  $b^*$  should be less than or equal to 1 in order to be inconspicuous. When this value is between 1 and 3, the color change is visually perceptible, and the value beyond 3.3 units in clinically unacceptable.<sup>13,14</sup>

All the three composite samples showed visually perceptible color change after immersion in the staining solution. The amount of color change was greatly affected by the group of composites used as well as the nature of staining solution used. In the turmeric staining solution, the mean color change seen was highest with Ceram.x ( $\Delta E=3.63$ ). This value was statistically significant when compared to the other two composites. In the coffee staining solution, the mean color change of all the composite resins was higher than the clinically acceptable limit, with Ceram.x being significantly higher than the rest ( $\Delta E=5.91$ ).

Table 2. Mean color changes ( $\Delta E$ ) of tested materials

Color parameter	Composite	Turmeric	p value	Coffee	p value	Distilled water	p value
Delta E	Filtek	2.02±0.61 [1.80] <sup>A</sup>	0.02	3.19±0.82 [3.49] <sup>A</sup>	0.02	0.87±0.06 [0.87]	0.56
	Ceram.X	3.34±0.53 [3.63] <sup>B</sup>		5.49±1.05 [5.91] <sup>B</sup>		1.04±0.78 [1.04]	
	Brilliant	2.17±0.70 [2.28] <sup>A</sup>		3.84±0.75 [3.64] <sup>A</sup>		1.01±0.02 [1.01]	
	EverGlow						

Table 3.  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  values among restorative materials

Color parameter	Composite	Turmeric	p value	Coffee	p value	Distilled water	p value
Delta L	F	-1.40±0.66 [-1.50]	0.12	-2.56±0.59 [-2.60] <sup>A</sup>	0.01	-0.10±0.42 [-0.10]	0.24
	C	-2.54±1.18 [-2.80]		-5.15±1.18 [-5.45] <sup>B</sup>		-0.85±0.63 [-0.85]	
	B	-1.54±0.82 [-1.60]		-3.00±0.94 [-3.40] <sup>A</sup>		-0.50±0.00 [-0.50]	
Delta a	F	-0.15±0.22 [-0.10]	0.06	-0.42±0.25 [-0.50] <sup>A</sup>	0.01	0.40±0.00 [0.40]	0.17
	C	-0.08±0.42 [-0.10]		-0.20±0.31 [-0.35] <sup>B</sup>		0.50±0.42 [0.50]	
	B	-0.54±0.24 [-0.60]		-0.72±0.17 [-0.70] <sup>A</sup>		-0.55±0.49 [-0.55]	
Delta b	F	-0.70±1.36 [-1.25]	0.56	-0.86±1.91 [-1.50]	0.14	-0.70±0.14 [-0.70]	0.09
	C	0.18±2.13 [0.20]		0.21±1.97 [-0.20]		0.35±0.21 [0.35]	
	B	-1.30±0.47 [-1.30]		-1.92±1.27 [-2.10]		-0.10±0.00 [-0.10]	

Same uppercase letters denote an insignificant difference between the same columns. The value in the brackets represents the median.

The Kruskal Wallis and Mann Whitney test results for  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  are given in Table 3.  $\Delta L^*$  represents the change in brightness of an object. In the present study, there was a significant difference before and after the polishing and staining of the composite materials. The samples after staining had become dark.  $\Delta L$  value was found to be highest for Ceram.x and lowest for Filtek. The  $\Delta a^*$  value means a shift towards red, which, according to the values, was seen highest in Ceram x. The  $\Delta b^*$ , which is a measure of a shift towards yellow, did not show any significant difference in the value. [ $\Delta a^*$  value <0 means a shift towards green, and  $\Delta a^*$  >0 means a shift towards red.  $\Delta b^*$  value <0 the color of specimens shifts to blue and  $\Delta b^*$  >0 means a shift towards yellow].

## Discussion

The present study was based on the assessment of the color stability of 3 different nanocomposites with different filler technologies when subjected to the action of food colorants. Color perception is highly subjective and prone to individual variation. To match the restoration to the color of any tooth, Culpepper<sup>15</sup> has noted a disagreement not only between different dentists but also the same dentist at different occasions in shade matching the same tooth. Miller, *et al.*<sup>16</sup> have listed various parameters that can affect the apparent tooth color, the major ones being the time of the day, the light source used, and surrounding conditions. Therefore, to eliminate any subjectivity of color perception, a spectrophotometer was used in this study for color measurements, which made it possible to assess the color change.

Composite restorations, although being highly aesthetic, exhibit a disadvantage of discoloration over time. This discoloration may be due to intrinsic or extrinsic

factors.<sup>17</sup> The intrinsic factors mainly include the choice of the material used along with the matrix properties and the interface between the matrix and the fillers. These factors directly affect the color of the restoration and can only be dealt with by changing the entire restoration. The extrinsic factors, on the other hand, are the exogenous sources such as food colorants, tobacco, and drinks.<sup>18</sup> The amount of color imbibed in the composite resin depends on the amount of water sorption the composite exhibits.<sup>19</sup> Studies have also shown that different surface finishing and polishing also affect the stain resistance property of the composite resin restoration.<sup>20</sup> Hence to negate this bias in the method of finishing and polishing composite resin, a common protocol with sofex discs was utilized for all the study samples.<sup>21</sup>

Coffee was chosen as one of the staining solutions in the present study because it has shown a high capacity of staining composite resin restorations and natural teeth.<sup>22</sup> Immersion of composite discs in a coffee solution for 30 days simulates two years of coffee consumption. The other staining solution used was turmeric. In the present study, maximum discoloration was seen in the composite discs immersed in coffee. This result is as per other studies.<sup>23,24</sup> The discs immersed in turmeric solution were significantly stained when compared to the control. However, the amount of color change was much less than that shown in the study by Malhotra, *et al* and Usha, *et al.*<sup>1,25</sup> This could be because, in our study, the samples were taken out from the staining solution, rinsed and dried before storing in distilled water. This procedure was to simulate the in-vivo condition of oral hygiene maintenance.

The dental composite resins are available in various technologies depending upon their molecular structure, i.e., the filler particle size as well as the amount of fillers present. In the present study, all three composites tested had nano-fillers. However, the nano-filler technology used

was different. Filtek™ Z350 XT is a nano-filled composite with non-aggregated as well as aggregated nano-filler clusters. The other two composites tested are nano-hybrid composites, which have small particle filler particles along with nano-sized filler particles. (Figure 2). These changes in filler size can affect the filler loading as well as the polishability. Thus, in turn, can affect the susceptibility of the composites to stains.<sup>11,12</sup>

In this study, Filtek™ showed the least amount of color change because of the presence of nano-filled technology that contains silica nanofillers and loosely bound nanoclusters made of silica/zirconia. This is said to provide better filler content along with high polishability.<sup>22</sup> The high polishability is attributed to the lightly agglomerated nanoparticles in the nanocluster that is only partially removed from the resin when subjected to finishing and polishing procedures. This will result in smaller surface irregularities.<sup>12</sup> Additionally, TEGDMA, a rather hydrophilic monomer, has been replaced with a hydrophobic resin combination of UDMA and bis-EMA. This could also be a contributing factor to the color stability of the nano-filled composite tested.<sup>24,26</sup>

In the final  $\Delta E$  values, Ceram.X™ showed the highest mean color change in the turmeric solution ( $\Delta E = 3.63$ ) as well as in the coffee solution ( $\Delta E = 5.91$ ), both of which

were clinically unacceptable. This nanoceramic hybrid composite contains spherical nanoparticles that are organically modified along with micro-sized glass fillers. The filler load in Ceram.X is lower than the other two composites tested. Lesser the volume of fillers, more will be the resin matrix (hydrophilic resin) available to absorb the stains. This could be one of the reasons why it showed increased staining.<sup>27</sup> Furthermore, the larger filler particles in this nanohybrid composites could also result in more surface irregularities after polishing.<sup>27</sup> Rough surfaces will adsorb stains more than smooth surfaces.

Brilliant EverGlow™ resin consists of silica glass particles, which increases the porosity of the biomaterial and, therefore, produces a higher level of surface roughness.<sup>28</sup> The composite resins that contain large filler sizes are more prone to discoloration due to the stains because of increased water sorption by the complex polymer network of the material.<sup>12</sup> The submicro-hybrid composite as the name suggests will have a combination of both small and large filler particles although few voids will be filled, but after the complete finishing and polishing, a large number of voids are left on the composite surface which eventually leads to greater water sorption and hence more discoloration.<sup>29</sup>

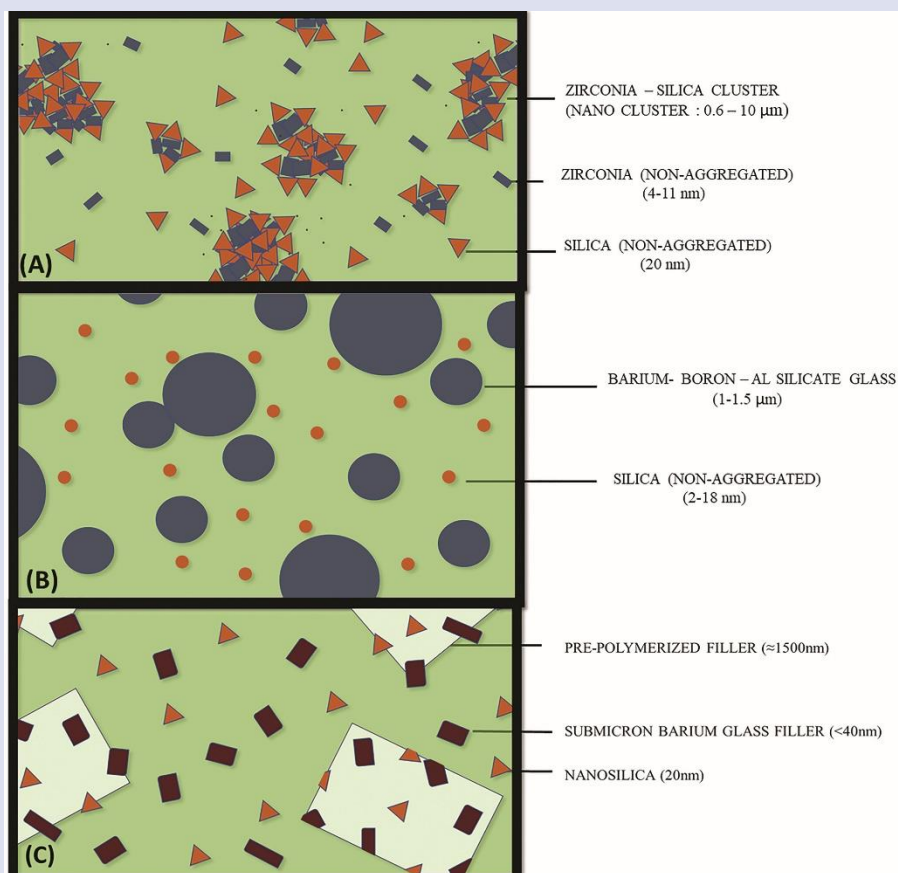


Figure 2. Filler particles and their distribution in the composites tested in this study; (A) Nano-filled composite with non-aggregated and aggregated filler Particles (B) Nano-hybrid composite with spherical filler particles (C) Nano-hybrid composite with pre-polymerized fillers and submicron filler particles

The filler load in Brilliant EverGlow is higher because it has incorporated pre-polymerized particles as filler as well. This could be the reason why this composite showed lower staining as compared to the other nano-hybrid composite. However, when compared to the nano-filled composite, the above-mentioned nano-hybrid composite showed more staining, though not statistically significant. This could be due to presence of more hydrophilic resins like TEGDMA.<sup>30</sup>

This study was done to identify the composite resin, which takes up the least amount of stain and hence can be used widely for a longer duration of time, providing better aesthetics. The results of this study give an insight into how different resin composites may behave when exposed to different dietary chromogens, thus affecting the clinician's choice of the material and the patients control of dietary habits. The present study only evaluated the in vitro effects of the different indigenous stains on the composite resins; however, in-vivo studies are required, which would take into consideration the effects of saliva and change of the pH of the oral cavity.

## Conclusions

Among the dental composite resins tested, nano-filled composite showed better color stability than the nano-hybrid composites.

All the dental nanocomposites showed clinically unacceptable discoloration when immersed in coffee solution ( $\Delta E > 3.3$ ). Whereas, turmeric solution caused clinically significant staining only in the nano-hybrid composite with ceramic technology. ( $p < 0.05$ ).

Thus, filler shape along with filler volume as well as resin composition could all have played a role in the variations seen among the color stability of the nanocomposite resin tested in the current study.

## Conflicts of Interest Statement

The authors have no conflicts of interests.

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