

## EVALUATION OF CHROMATIC CHANGES OF A NANOCOMPOSITE RESIN USING THE NEW WHITNESS INDEX

DIANA CARLA MADA<sup>1</sup>, CRISTINA GASPARIK<sup>1</sup>,  
ALEXANDRA IULIA IRIMIE<sup>1</sup>, MARIUS DAN MADA<sup>1</sup>,  
DIANA DUDEA<sup>1</sup>, RADU SEPTIMIU CAMPAN<sup>2</sup>

<sup>1</sup>Dental Propedeutics and Esthetics Department, Faculty of Dental Medicine, Iuliu Hatieganu University of Medicine and Pharmacy, Cluj-Napoca, Romania

<sup>2</sup>Oral Rehabilitation Department, Faculty of Dental Medicine, Iuliu Hatieganu University of Medicine and Pharmacy, Cluj-Napoca, Romania

### Abstract

**Background and aims.** To evaluate the staining effects of two brands of coffee and the bleaching efficiency of two in-office bleaching methods, upon different opacities of a commercial nanocomposite.

**Methods.** Twenty four specimens of each opacity, A3 Dentin, A3 Body and A3 Enamel, were fabricated from Filtek Supreme (3MEspe). The specimens were further divided into two groups (n=12) and were immersed in two coffee solutions (Bio Organic Coffee Bellarom, 100% Arabica, and Iulius Meinel Coffee), for 24 hours. Between the staining sessions, the specimens were stored in sterile water, at 37°C. Each group was further divided into three (n=4), in order to be bleached, as follows: Group 1 - Beyond 35% in office, for 4 applications of 15 minutes each, Group 2 - Zoom Day White 6% in office, for 4 applications of 15 minutes each, Group 3 - Control Group, stored in sterile water. Color values were measured with a dental spectrophotometer Vita EasyShade 4.0 and five measurements were recorded for each sample at a time. Lightness L\*, color coordinates a\* and b\* were recorded, at baseline, after staining in coffee and after bleaching. Whiteness index (WID) of the three composite resins (A3D, A3B, A3E) in the three moments were calculated, as well as the color difference Delta E\* correspondent to the staining and bleaching process. Data were analyzed using one-way repeated measures ANOVA and the WID index was calculated WID (p<0.05). Univariate analysis of variance was performed for assessing the influence of staining solution upon composite resins, as well as for testing the effect of bleaching agents. The significance level was set at  $\alpha=0.05$  and pairwise comparisons were adjusted by the Least Significant Difference method.

**Results.** The pairwise comparisons showed no significant difference between the effects of the two bleaching agents upon the WID, meaning that they induce almost similar color changes. The results of the univariate ANOVA test indicated a significant effect of the composite resin and the staining solution upon the WID (p<0.05). However, no significant interaction effect was found between the composite resin and the staining solution (p=0.095). There was a significant difference in the staining effect of the two coffee solutions only for A3B and A3E composite resins (p<0.05).

**Conclusions.** The chromatic changes of the nanocomposite resin could be evaluated by the variation of the whiteness index. The staining effect induced by the two types of coffee was similar. The most effective protocol was the in-office bleaching method based on Beyond 35%.

**Keywords:** bleaching, staining, nanocomposite, color stability, whiteness index

## Background and aims

Composite resins are the most frequently used materials in dentistry, for tooth cavity restorations. They are usually composed of reinforcing fillers mixed in an acrylic monomer matrix that is polymerized to form a solid restoration [1-7]. There are numerous advantages for the use of dental resin composites, such as: no need to sacrifice healthy tooth tissue to create mechanical undercuts; adhesion to dental tissue, reduced risk of bacterial ingress; increasing of the fracture resistance of the restored tooth [8-11], ease of use, reduced number of treatment sessions, color in the range of natural dental shade, no mercury content. The research in the last decades improved the composites hardness, flexural resistance, translucency, coefficient of thermal expansion, chromatic stability [12-13].

Nowadays resin composites have replaced amalgam in almost every clinical situation, although they still possess inherent clinical disadvantages, linked mainly with their technical sensitivity and contraction, which explain marginal staining, secondary caries and microleakage; among other shortcomings are cited reduced wear, fracture resistance and requirement of a clinical procedure that is more time-consuming in comparison with amalgam fillings [13-14]. Despite all these factors, there continues to be an increasing use of composite resins. An important quality of composites resides in their versatility and possibility to be provided in different shades and opacities, to mimic the optical properties of natural dental structures.

The role of different opacities in direct restorations, is given by the complexity in the optical properties of the composite resins: the shade and the translucency/opacity [12]; it is believed that the major parameter to be considered in the selection of dental composites is the opacity or translucency [12].

Considering the unlimited possibilities of available shades and opacities used for reproducing the optical properties of the dental structures, the initial outcome of a direct restoration, may be excellent [12]. On the other hand one major disadvantage of composite resins is their coloristic instability and low resistance to staining, caused by intrinsic and extrinsic factors [12]. Extrinsic factors that can induce color changes are food colorants, beverages, UV radiation, temperature changes and water absorption [12].

A method of choice for removing the dental staining are different bleaching techniques, conducted either at home or as in-office procedures. Most of the bleaching methods are based on hydrogen peroxide (HP) or on carbamide peroxides (CP), which decompose and generate free radicals with bleaching effect.

The most rapid method of bleaching is the “in office bleaching”, based on 6%, 16%, 35%, 40%, 45% of either HP or CP, for 30 to 45 minutes, with or without light activation [15]. A lot of research has been carried out, to find out what are the effects of the tooth bleaching upon the tooth surface and upon the dental restorative materials.

The effects induced by bleaching upon the characteristics of composite resins, such as changes in color, further staining susceptibility, changes in surface roughness and in composites' hardness, microleakage and elution have been tested [15,16]. It was found by Li et al. [17] that significant changes occurred in the color of composite resins after bleaching with 15% CP, but the difference was especially noticeable when a high peroxide concentration (35%) was used on low-density resins [15-18].

When bleaching effect is evaluated, color differences are usually calculated using the CIEL\*<sub>a</sub>\*b\* formula [19], or more recently CIEDE 2000 formula [20]. Moreover, color difference thresholds have been introduced in dentistry, in order to quantify the clinical significance of the differences between two colored samples. Paravina et al. conducted a multicenter study upon visual thresholds in dentistry, and have reported that values smaller than  $\Delta E_{ab}^* = 1.2$  ( $\Delta E_{00} = 0.8$ ) are not perceptible, while values greater than  $\Delta E_{ab}^* = 2.7$  ( $\Delta E_{00} = 1.8$ ), are considered to be clinically unacceptable [21]. However, color difference formulas only show the amount of color difference, and not the direction of the color changes (increase or decrease in lightness, chroma, hue).

Whiteness is an important color attribute and there is a long history, even outside of the field of dentistry, of searching for a one-dimensional color index to quantify whiteness [22]. Currently, a number of whiteness formulae are in common use including the CIE whiteness index WIC.

Since the color of most human teeth corresponds to a small range of the color space from yellowish-white to light-brown and the degradation and aging of teeth is usually associated with yellowness, some researchers have proposed that changes in yellowness are important factors in the assessment of tooth whitening [23,24]. As a consequence, the quantification of whiteness has sometimes been expressed by a quantification of yellowness; lower yellowness values are considered as corresponding to higher whiteness. In a study to assess whether whiteness or yellowness indices can predict the perception of tooth whiteness, WIC gave the best result for measuring tooth whiteness when compared with visual assessments [24,25].

Recently, a new and customized whiteness index (WID) was designed specifically for dentistry [20]. It was developed based on correlations with visual perception of tooth shaped shade tabs and dental materials. The whiteness index (WID) is a simple linear formulation, obtained using the values of the three CIELAB chromatic coordinates. It is aiming to avoid the subjective visual factor in the dental color measurement, and has a clear interpretation: high positive values of the WID index indicate higher whiteness values of the specimen while low (even negative) values indicate lower values of whiteness of the specimen [20].

Our study aims to assess the staining effect of two brands of coffee as well as the bleaching efficiency of two in office methods, upon different opacities of a commercial

nanocomposite by calculating the variation of the new whiteness index.

The null hypothesis is that there was no significant change in whiteness index of composites after a. immersion in coffee and b. bleaching procedures.

**Methods**

**Specimen preparation, staining and bleaching procedures**

The composite resin used in the study was Filtek Supreme (3MTM ESPETM): A3 Dentin Shade, A3 Body Shade and A3 Enamel Shade.

Twenty four disk-shaped specimens of each opacity (n=24) were fabricated. The specimens were polished to a uniform thickness using abrasive sandpaper of 400, 600 and 1200 (2 mm thickness, 10 mm diameter).

Two groups of disks (n=12) were immersed in Bio Organic Coffee (Bellarom), 100% Arabica, and Iulius Meinel Coffee (Cafe Gourmet, Auslese, Italy) respectively, for 24 hours. The coffee machine used was Saeco Incanto Executive (Italy), with ceramic grinder for coffee beans (espresso mode, using 5g of grounded coffee for 100 ml water).

During the staining process, the specimens were stored in sterile water, at 37°C. Each group was further divided in three subgroups (n=3), in order to be bleached, as follows: Group 1 - Beyond 35% HP in office (980 nm), for 4 applications of 15 minutes each, Group 2 – Zoom Day White 6% HP in office (400-505 nm), for 4 applications of 15 minutes each, Group 3 – Control Group, stored in sterile water.

**Color measurements**

Data color values were measured using a dental spectrophotometer (VITA EasyShade Advance 4.0., VITA, Bad Sackingen, Germany) and the CIEL\*a\*b\* values were recorded: L\* refers to lightness; it's value ranges from 0 (black) to 100 (white), while a\* and b\* are measures of greenness and redness, respectively blueness or yellowness of an object. The a\* and b\* chromaticity coordinates reach 0 for neutral colors and increase in magnitude for saturated or intense colors [1]. Five measurements were taken for each sample at a time.

The color measurements were taken using a dental spectrophotometer before staining (baseline L<sub>0</sub>\*, a<sub>0</sub>\*, b<sub>0</sub>\*); after staining (L<sub>1</sub>\*, a<sub>1</sub>\*, b<sub>1</sub>\*), and at the end of bleaching

process (L<sub>2</sub>\*, a<sub>2</sub>\*, b<sub>2</sub>\*).

**Calculation of DE and WID**

The equation used for the calculation of Whiteness index WI<sub>D</sub> was [21]:

$$WI_D = 0.511L^* - 2.324a^* - 1.100b^*$$

WI<sub>D</sub> is a simple linear formulation, obtained using the values of the three CIELAB chromatic coordinates and has a clear interpretation: high positive values of the WI<sub>D</sub> index indicate higher whiteness values of the specimen, while low (even negative) values indicate lower values of whiteness of the specimen [22].

The formula for the calculation of the color difference ΔE<sub>00</sub> was [27]:

where:

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

$$\Delta L' = L_2^* - L_1^*;$$

$$\Delta C' = C_2' - C_1';$$

$$\Delta H' = 2\sqrt{(C_1' C_2')} \sin(\Delta h'/2);$$

$$S_L = 1 + [0.015(L' - 50)^2 / \sqrt{20 + (L' - 50)^2}];$$

$$S_C = 1 + 0.045C';$$

$$S_H = 1 + 0.015C'T;$$

$$R_T = -\sin(2\Delta\theta)R_C;$$

K<sub>L</sub>, K<sub>C</sub>, K<sub>H</sub> = parametric wheighting factors;

**Statistical analysis**

Univariate analysis of variance was performed for assessing the influence of staining solution upon composite resins, as well as for testing the effect of bleaching agents. The significance level was set at α=0.05 and pairwise comparisons were adjusted by the Least Significant Difference method.

**Results**

Mean values of color parameters and whiteness index (WID) of the three composite resins opacities (A3D, A3B, A3E) at baseline, after immersion in coffee, and after bleaching are presented in Table I, II and III:

Figures 1-3 show the variation of the WID after staining and bleaching, for the three composite resins.

**Table I.** Mean values of CIELAB color parameters and WID for A3D composite resin.

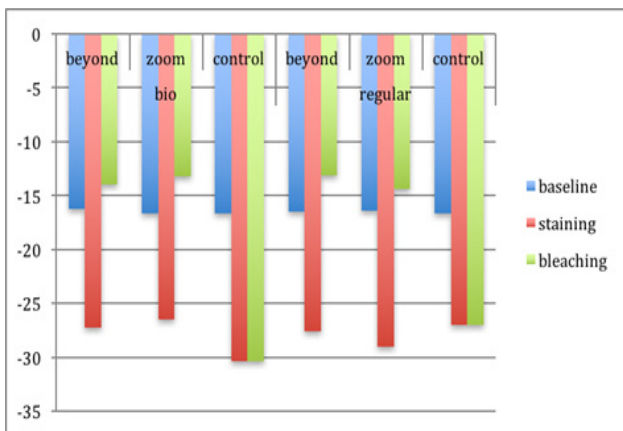
		baseline				After staining					After bleaching				
		L*	a*	b*	WID	L*	a*	b*	WID	ΔE00	L*	a*	b*	WID	ΔE00
bio	beyond	81.08	4.95	41.95	-16.22	78.85	6.85	46.95	-27.27	2.47	78.85	4.70	40.55	-13.95	2.97
	zoom	81.05	5.10	42.00	-16.64	78.60	6.63	46.63	-26.52	2.41	78.60	4.43	41.03	-13.24	3.50
	control	81.00	5.13	41.93	-16.64	77.35	7.50	47.68	-30.35	3.41	77.35	7.50	47.68	-30.35	0
regular	beyond	81.03	5.10	41.85	-16.48	77.60	6.85	46.68	-27.61	3.01	77.60	4.28	39.98	-13.12	3.08
	zoom	81.00	5.10	41.80	-16.44	77.08	7.13	47.15	-29.04	3.43	77.08	4.83	39.65	-14.38	3.12
	control	81.00	5.15	41.90	-16.67	77.73	6.78	46.38	-27.04	2.85	77.73	6.78	46.38	-27.04	0

**Table II.** Mean values of CIELAB color parameters and WID for A3B composite resin.

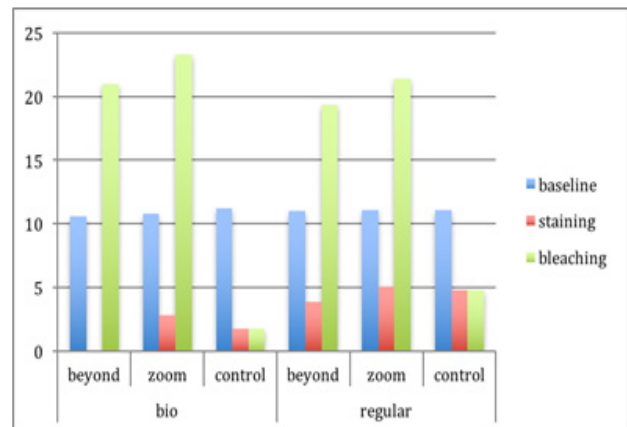
		baseline				After staining					After bleaching				
		L*	a*	b*	WID	L*	a*	b*	WID	$\Delta E_{00}$	L*	a*	b*	WID	$\Delta E_{00}$
<i>bio</i>	beyond	80.20	0.60	26.40	10.55	78.08	2.00	31.98	0.08	3.01	82.60	-1.05	21.50	21.00	6.26
	zoom	80.00	0.60	26.13	10.80	78.85	1.73	30.43	2.82	2.25	83.88	-1.80	21.58	23.31	6.22
	control	80.10	0.60	25.78	11.20	78.65	1.80	31.13	1.77	2.71	78.65	1.80	31.13	1.77	0
<i>regular</i>	beyond	80.10	0.60	25.93	11.03	79.35	1.65	29.85	3.88	1.99	80.75	-0.83	21.65	19.37	4.48
	zoom	80.20	0.60	25.90	11.09	79.45	1.25	29.68	5.05	1.81	81.00	-1.18	20.63	21.43	4.92
	control	80.10	0.60	25.93	11.06	79.53	1.33	29.78	4.81	1.85	79.53	1.33	29.78	4.81	0

**Table III.** Mean values of CIELAB color parameters and WID for A3E composite resin.

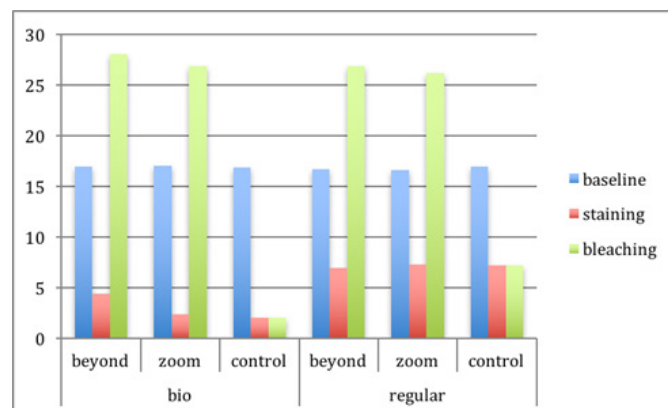
		baseline				After staining					After bleaching				
		L*	a*	b*	WID	L*	a*	b*	WID	$\Delta E_{00}$	L*	a*	b*	WID	$\Delta E_{00}$
<i>bio</i>	beyond	78.30	0.15	20.63	16.98	74.30	1.58	27.18	4.41	4.44	79.20	-2.03	15.60	28.02	7.87
	zoom	78.43	0.13	20.73	16.99	74.10	1.80	28.48	2.36	5.01	77.33	-2.20	16.18	26.83	7.76
	control	78.13	0.08	20.78	16.90	73.28	1.85	28.33	1.99	5.24	73.28	1.85	28.33	1.99	0
<i>regular</i>	beyond	78.03	0.25	20.55	16.68	76.15	1.13	26.70	6.93	3.35	77.13	-1.93	15.48	26.86	6.71
	zoom	78.18	0.10	21.05	16.56	76.10	1.10	26.43	7.26	3.11	76.83	-1.95	16.03	26.16	6.30
	control	78.50	0.08	20.93	16.92	76.15	1.20	26.30	7.19	3.25	76.15	1.20	26.30	7.19	0



**Figure 1.** WID variation for A3D composite resin.



**Figure 2.** WID variation for A3B composite resin.



**Figure 3.** WID variation for A3E composite resin.

The results of the univariate ANOVA test indicated a significant effect of the composite resin opacity and the staining solution upon the WID ( $p < 0.05$ ). However, no significant interaction effect was found between the composite resin and the staining solution ( $p = 0.095$ ).

Analyzing the pairwise comparisons, it can be observed that there was a significant difference in the staining effect of the two coffee solutions only for A3B and A3E opacities ( $p < 0.05$ ) (Table IV).

**Table IV.** Pairwise Comparisons of the staining solutions for A3D, A3B, and A3E composite resins.

<i>Dependent Variable: WID coffee</i>			
<i>comp_resin</i>	<i>(I) stain_sol</i>	<i>(J) stain_sol</i>	<i>Sig.<sup>*b</sup></i>
A3D	coffee_bio	coffee_regular	0.911
	coffee_regular	coffee_bio	0.911
A3B	coffee_bio	coffee_regular	<b>0.027</b>
	coffee_regular	coffee_bio	<b>0.027</b>
A3E	coffee_bio	coffee_regular	<b>0.002</b>
	coffee_regular	coffee_bio	<b>0.002</b>

\*. The mean difference is significant at the 0.05 level  
 b. Adjustment for multiple comparisons: Least Significant Difference

The analysis of variance also showed a significant effect of the bleaching agents upon the WID ( $p < 0.05$ ). Furthermore, a significant interaction effect was observed between the bleaching agents and the composite resins, and the bleaching agents and the staining solutions ( $p < 0.05$ ).

The pairwise comparisons showed no significant difference between the effects of the two bleaching agents upon the WID, meaning that they induce almost similar color changes (Table V).

**Table V.** Pairwise Comparisons of the bleaching effect for A3D, A3B, and A3E composite resins.

<i>Dependent Variable: WID after bleaching</i>					
			<i>Sig.<sup>*b</sup></i>		
			A3D	A3B	A3E
<i>coffee_bio</i>	beyond	zoom	0.706	0.218	0.526
		control	0.000	0.000	0.000
	zoom	beyond	0.706	0.218	0.526
		control	0.000	0.000	0.000
	control	beyond	0.000	0.000	0.000
		zoom	0.000	0.000	0.000
<i>coffee_regular</i>	beyond	zoom	0.498	0.270	0.708
		control	0.000	0.000	0.000
	zoom	beyond	0.498	0.270	0.708
		control	0.000	0.000	0.000
	control	beyond	0.000	0.000	0.000
		zoom	0.000	0.000	0.000

\*. The mean difference is significant at the 0.05 level  
 b. Adjustment for multiple comparisons: Least Significant Difference

### Discussion

The null hypothesis was rejected since significant changes in whiteness index of the composite resin were found after coffee immersion and bleaching procedures.

The most important staining effect was shown for A3E when immersed in regular coffee, while the lowest staining effect was shown for A3B when immersed in bio coffee.

The two categories of coffee induced different staining effects upon enamel and dentine opacities; this effect is probably due to differences in the absorption and interaction of staining solution with the organic matrix of the composite resins.

On the contrary, no difference has been found between the two bleaching methods. The difference between these methods consisted in the concentration of the bleaching gel (6% and 35% HP), and the brand (Beyond and Zoom), while the protocol was the same.

The structure of composite resin and characteristics of the particles have a direct impact on the surface smoothness and susceptibility to extrinsic staining [26].

The resin system - Filtek Ultimate - that we used in the study is slightly modified from the original Filtek™ Z250 Universal Restorative and Filtek™ Supreme Universal Restorative resin. The resin contains bis-GMA, UDMA, TEGDMA, and bis-EMA resins. To moderate the shrinkage, PEGDMA has been substituted for a portion of the TEGDMA resin in Filtek Supreme XT restorative. The fillers are a combination of non-agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler. The Dentin, Enamel and Body shades have an average cluster particle size of 0.6 to 10 microns. The Translucent shades have an average cluster particle size of 0.6 to 20 microns. The inorganic filler loading is about 72.5% by weight (55.6% by volume) for the Translucent shades and 78.5% by weight (63.3% by volume) for all other shades.

The high staining susceptibility of hybrid composite resin could be attributed primarily to its high resin contents and the related water contents. It has been demonstrated that diffusion coefficient of water absorption in hybrid composite is within the range of reported for conventional composite resin [27].

Recently, manufacturers are producing composites with smaller filler particles in an attempt to produce materials with similar surface smoothness as that of dental enamel. An increase in filler content produces smoother surfaces because of the lower particle size and better distribution within the resin matrix [28]. A different study showed that coffee altered the color of the tested composites, depending on the characteristics of the materials [29].

Color change of composite restorations in different color media during time is a common problem in esthetic dentistry, and, as shown in the study of Malekipour MR et

al, the staining solution and immersion time are significant factors that affect color stability of composite resins [30].

From the FTIR (Fourier Transform InfraRed – vibrational method) spectroscopy data, as shown in the study of Al Kheraif A.A., we know that the degree of conversion for Nano composites is higher than the values obtained for Microhybrid resin composites (70% vs. 56%), but no correlation has been found between the degree of conversion and color stability [31].

In our study, the most important bleaching effect was shown for A3E when bleached with Beyond 35% while the lowest effect was shown for A3B when bleached with Zoom Day White 6%, but with the mean difference not significant, statistically.

No significant interaction effect was found between the composite resin and the staining solution ( $p=0.095$ ), while the results of the univariate ANOVA test indicated a significant effect of the composite resin opacity and the staining solution upon the WID ( $p<0.05$ ). There also was a significant difference in the staining effect of the two coffee solutions only for A3B and A3E opacities ( $p<0.05$ ).

The pairwise comparisons showed no significant difference between the effects of the two bleaching agents upon the WID, meaning that they induce almost similar color changes. The analysis of variance also showed a significant effect of the bleaching agents upon the WID ( $p<0.05$ ). Furthermore, a significant interaction effect was observed between the bleaching agents and the composite resins, and the bleaching agents and the staining solutions ( $p<0.05$ ).

### Conclusions

Within the limitations of the study, the following conclusions may be drawn:

1. The chromatic changes of the nanocomposite resin could be evaluated by the variation of the whiteness index;
2. The results of the univariate ANOVA test indicated a significant effect of the composite resin and the staining solution upon the WID ( $p<0.05$ ).
3. The opacities of the nanocomposite resin showed different chromatic changes;
4. Analyzing the pairwise comparisons, it can be observed that there was a significant difference in the staining effect of the two coffee solutions only for A3B and A3E composite resins ( $p<0.05$ ).
5. The staining effect induced by the two types of coffee was similar.

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