

COLOR AND OPACITY VARIATIONS IN THREE DIFFERENT RESIN-BASED COMPOSITE PRODUCTS AFTER UV AGING

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Abstract

Objective: The aim of this study was to evaluate color and opacity variations of all the Vita shades simultaneously available for three structurally different resin-based products after exposure to a UV light source.

Methods and Materials: The six shades all present in the following restorative systems were selected for the trial: Spectrum TPH (De Trey), Tetric Ceram (Ivoclar-Vivadent), and Z100 (3M ESPE), i.e. A2, A3, A3.5, A4, B2, B3. Composite resin discs measuring 1.5 mm in diameter and 1 mm in thickness were prepared and their color measured with a laboratory spectrophotometer (PSD1000, OceanOptics). The specimens were stored in a UV-aging chamber for 24h according to the ISO 7491-1985 standard. A second measurement was then taken to assess the color and opacity variations.

Results: Although all the materials showed a certain degree of discoloration due to UV light exposure, the majority (94,5%) of the color values was within the range of clinical acceptability ($\Delta E < 3,3$). Only Tetric Ceram underwent color changes that could be perceived clinically. The opacity of all the materials increased after UV aging, with the exception of Tetric Ceram A4 and B3, which had no variation.

Conclusion: The results of this study indicate that composite resin exposure to UV light for 24h causes changes in CIE color space coordinates of the materials involved in the study. When compared with similar previous research dealing with the same shades and materials, UV light showed less effect on color stability than water.

Clinical Significance: **UV irradiation can affect color stability of resin composites in different degrees which could relate** to different resin matrix properties.

Short Title: Color variation in composite after UV aging

Key words: Color, Composite resin, UV aging.

Introduction

Composite resin materials are progressively going to substitute amalgam restorations. As aesthetics is one of the main reasons for replacement, it is really important that the aesthetic characteristic of the materials be able to mimic the natural tissue, and that the results remain stable over the time of clinical service. It is known that dental composites are susceptible to discoloration because of several factors, such as water hydrolysis^{1,2}, ultraviolet light exposure^{3,6}, and staining substances^{7,9}. The rapid turn-over of the products driven by research progress and commercial motivations, limit the possibility to collect reliable clinical long-term data. For this

reason, accelerated tests to simulate oral environment aging have been proposed. As a matter of fact, ISO¹⁰ and ADA¹¹ specifications include color stability as one of the requirements for clinical acceptability of a material. Studies have already been conducted on this topic^{2,6}. However, data are difficult to compare, as manufacturer often adopt different shade guides and different names for the colors of restorative materials. It was only recently that some manufacturers agreed to use shade guides correlated to the Vita shade guide. The purpose of this study was to evaluate color and opacity variations resulting from exposure to a UV light source of all the Vita shades simultaneously available for three structurally different restorative composite resins.

Materials and Methods

The restorative systems selected for the study were: Spectrum TPH (De Trey, Konstanz, Germany), Tetric Ceram (Ivoclar-Vivadent, Schaan, Liechtenstein) and Z100 (3M ESPE, St Paul, MN, USA).

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Figure 1: The mold used for fabricating the samples.

For each material, the availability of Vita shades was checked, and the shades that were included in all the three systems were chosen for the trial.

Thus, six colors were selected: A2, A3, A3.5, A4, B2, B3.

For each restorative material specimens of 1.5 mm in diameter and 1 mm in thickness were prepared using a steel mold (Figure 1). The material was placed into the mold. Then, a mylar strip was positioned over its surface and covered by a glass plate. The glass plate was manually pressed until it reaches the steel mold surface. The resin composite was light-cured for 10 sec through the glass plate. Then, the latter was removed and the material was directly irradiated for 50 sec with the Visilux 2 light curing unit (3M ESPE, St Paul, MN, USA,) for all the specimens. The light intensity of the curing unit was 500 mW², as measured with Demetron 100 radiometer (Demetron Research Co, Danbury, CT, USA).

The greatest possible care was taken to avoid any bubble formation within the material. The thickness of each disc was precisely measured with an electronic digital caliper (1651 DGT, Beta, Milano, Italia) with a 10µm resolution.

Only the specimens that did not show any difference in thickness were included in the study, having a previous study shown that the material thickness affects color assessment¹².

For each shade three discs were prepared, giving a total of 54 specimens per shade.

Specimens were stored at room temperature for 24 hours in order to allow for some increase in the polymerization degree.

For color measurements, a spectrophotometer (PSD1000, Ocean Optics, FL, USA) equipped with an integrating sphere (ISP-REF, Ocean Optics, FL, USA) with a 10 mm opening was used. The spectrophotometer was connected with a computer running OOILab 1.0 (Ocean Optics, FL, USA) in CIE Lab* color

system as color measurement software. CIE (Commission Internationale de l'Eclairage) Lab* system was established in 1976 and 1978 as International standard for color measurement. It consist of a three-dimensional color coordinate system by which every color may be identified.

Of the three variables, L* is defined as the attribute by which a perceived color is judged to be equivalent to one of a series of grays ranging from black (0) to white (100). a* is defined as the difference in "a" between a specimen and a standard reference color.; if "a" is positive, there is more redness than greenness, if "a" is negative, there is more greenness than redness. b* is defined as the difference in "b" between a specimen and a standard reference color; if "b" is positive, there is more yellowness than blueness; if "b" is negative, there is more blueness than yellowness. D65 illumination and 10° standard observation angle were selected. For color measurement, a 50% gray card (Kodak Co, Rochester, USA) was used as a neutral background. For the evaluation of the opacity, as it is defined as the ratio of the reflectance of a specimen disk when backed by a black standard to that when backed by a white standard¹³, a black and a white cardboard were taken from color separation scale Q-14 (Kodak Co, Rochester, USA).

The software was programmed to take 10 measurements for each specimen and calculate their mean.

ΔE was calculated with the formula $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$ ¹⁴ and used to compare the materials samples.

As the ability of the human eye to discern differences in color differs from subject to subject, depending on both eye characteristics and individual skill, values of ΔE lower than 1 are regarded as undetectable by the human eye. ΔE values between 1 and 3.3 are perceived also by an untrained, but careful

Table I. Lab* color values and calculated ΔE.

Material	Shade	L*	a*	b*	Delta E
Spectrum TPH	A2	68.59	0.25	12.66	
Spectrum TPH	A2	68.59	-0.33	13.20	0.8
Spectrum TPH	A3	65.21	0.34	13.89	
Spectrum TPH	A3	65.64	-0.09	15.03	1.3
Spectrum TPH	A3.5	65.59	1.43	18.30	
Spectrum TPH	A3.5	65.81	1.10	18.48	0.4
Spectrum TPH	A4	61.22	1.81	21.12	
Spectrum TPH	A4	60.02	1.35	18.37	1.5
Spectrum TPH	B2	66.84	-0.57	15.08	
Spectrum TPH	B2	66.85	-0.89	14.99	0.3
Spectrum TPH	B3	63.58	0.98	18.15	
Spectrum TPH	B3	63.74	0.77	18.58	0.5
Tetric Ceram	A2	67.81	-1.41	14.11	
Tetric Ceram	A2	68.82	-1.26	12.41	2.0
Tetric Ceram	A3	68.78	-1.13	17.07	
Tetric Ceram	A3	69.49	-0.86	14.32	2.8
Tetric Ceram	A3.5	66.23	0.15	16.03	
Tetric Ceram	A3.5	66.86	0.19	15.31	1.0
Tetric Ceram	A4	61.22	1.81	21.12	
Tetric Ceram	A4	64.29	1.02	19.87	3.4
Tetric Ceram	B2	69.02	-1.82	11.60	
Tetric Ceram	B2	69.22	-1.63	10.15	1.5
Tetric Ceram	B3	66.53	-1.15	19.66	
Tetric Ceram	B3	66.76	-0.98	17.78	1.9
Z100	A2	67.62	-1.51	12.35	
Z100	A2	67.00	-1.14	13.15	1.1
Z100	A3	69.16	-0.61	14.46	
Z100	A3	68.49	0.02	15.36	1.3
Z100	A3.5	65.17	-1.51	16.22	
Z100	A3.5	64.51	-0.57	16.98	1.4
Z100	A4	65.17	-0.81	16.56	
Z100	A4	64.60	-0.20	17.18	1.0
Z100	B2	70.42	-1.04	11.98	
Z100	B2	69.84	-0.76	13.55	1.7
Z100	B3	68.33	-0.72	17.40	
Z100	B3	67.75	-0.25	18.41	1.3

Before treatment
After treatment

examiner, and can be considered clinically acceptable. Conversely, values of ΔE greater than 3.3 are detected also by inexperienced observers and are therefore considered clinically unacceptable¹⁴.

After the color measurement, specimens were placed in an accelerated UV-aging chamber (Suntest Accelerated Exposure Table Unit, Hanau-Hereaus GmbH, Germany) for 24 hours following the ISO 7491-1985 specification¹⁰.

In order to test the color and opacity variations after UV-

aging, a second color measurement was performed after having removed the specimens from the UV chamber.

The level of significance was set at p=0,05. The Kolmogorov-Smirnov test was used for checking for the normality of data distribution. The one-way analysis of variance was applied with ΔE as dependent variable and type of resin composite as factor in the evaluation of color changing after UV-aging; with ΔOpacity as dependent variable and type of resin composite

as factor in the evaluation of opacity changing after UV-aging. The Tuckey HSD test was applied for post-hoc comparison. The statistical analysis was processed by the SPSS 12.0 software (SPSS, Inc., Chicago, IL, USA).

Results

Color

All the materials showed a certain degree of discoloration due to UV light exposure (Table 1). If the value $\Delta E > 3.3$ is taken as the limit for clinical appreciation of color variation, the great majority (94,5%) of the values were within this range for the tested materials.

However, the one-way analysis of variance showed a statistically significant different behaviour ($p < 0.05$) between Spectrum TPH, Z100 and Tetric Ceram. Post-hoc comparison showed that between Spectrum TPH and Z100 no significant difference could be demonstrated ($p > 0.05$), although the overall results were slightly better for Spectrum TPH. Otherwise there is a statistically significant difference between Spectrum TPH and Tetric Ceram ($p < 0.05$), with Spectrum TPH showing a lower discoloration if compared to Tetric Ceram. The L^* value of Spectrum TPH and Tetric Ceram varied without a specific trend, while in Z100 the parameter had a quite constant decrease, which means that the resin composite became progressively darker. The Δa^* value were low for all the materials ($0.0 < \Delta a^* < 0.9$). The b^* value variations were higher than a^* variations. Z100 (mean 0.94) and Spectrum TPH (mean 0.86) had lower magnification in comparison with Tetric Ceram (mean 1.65). This higher variation of b^* values for Tetric Ceram is probably responsible for the worst result of Tetric Ceram in ΔE calculation. Spectrum TPH and Tetric Ceram shifted without a sharp pattern, while Z100 had always an increase in b^* value (yellowish). There was not a clear pattern of color shifts between clearer and darker colors, and no surely appreciable differences were noted between A and B based colors.

Opacity

The opacity of all tested materials increased after UV aging, except for Tetric Ceram A4 (0.0) and B3 (+0.9) that had no variation (Table 2). As these values are very low, it may be due to the tolerance of measuring devices.

The one-way ANOVA statistical analysis revealed that there were no statistically significant difference ($p > 0.05$) between the

three materials tested. Also for the opacity, as for color shifts, there was no evidence of consistently different behaviour for clearer and darker colors and for A and B based colors.

Discussion

The results of this study indicate that UV light exposure of composites for 24h causes changes in color of the selected materials. Such change has been previously reported in the literature^{3, 4, 5, 6, 15, 16}. The problem in the evaluation of the results from this type of test is the limit in the ability to detect differences. In fact, there is not a total agreement as for the limit of the human eye to discern the differences that are instrumentally calculated, being obvious that a discoloration that is not detectable by the human eye is clinically meaningless. Some authors^{9, 16, 17} reported this limit to be $\Delta E = 2$, while other authors reported $\Delta E = 3.3$ as the limit¹⁸⁻²² for clinical acceptability of color differences. This variation is probably connected with the variability in human eye appreciation of color differences and in experimental conditions of comparison between instrumental and visual evaluation of color differences. Moreover, Seghi et al.¹⁷, in a laboratory study on dental ceramics reported that under ideal conditions of illumination and viewing, a value of $\Delta E < 1$ was seldom distinguished by the majority of the observers. Based on this data, we divided the limit of the visual correlation of the results in three categories. Values of $\Delta E < 1$ were considered as only instrumentally detectable. Values of ΔE between 1 and 3.3 were considered visually detectable from challenged operators but still clinically acceptable. Conversely, values of $\Delta E > 3.3$ were considered to be detected also by untrained observers, laypeople such as patients, and should therefore be regarded as clinically unacceptable. This classification is based on literature references and seems to allow a balanced evaluation of the results of color measurement tests²³. Following these criteria, all the measured discoloration except one (Tetric Ceram A4: $\Delta E = 3.4$) fell within the limits of clinical acceptability. However, the behaviour of the three selected materials was clearly different, with Tetric Ceram appearing more sensitive to discoloration due to UV irradiation to a statistically significant degree. In order to understand the reason for these differences, it is necessary to take into account the different nature of the matrix, as the amount of resin matrix could not be directly associated with the degree of

color shift (filler load by volume is 57% for Spectrum TPH, 60% for Tetric Ceram and 66% for Z100). The color shifts may be related to the nature of the resin matrix (Bis-GMA, Bis-EMA and TEGDMA for Spectrum TPH, Bis-GMA, UDMA and TEGDMA for Tetric Ceram and Bis-GMA and TEGDMA for Z100). It may be supposed, even if a confirmation for this hypothesis is not available in literature, that UDMA could be more sensitive to UV radiation than TEGDMA. The fact that the matrix nature may be the responsible for color shift may be supported by the observation that no significant differences were found in the shifting pattern between darker and lighter shades, and between A and B groups for any of the tested material. Based on these data it may also be speculated that nature and amount of pigments do not interfere with the shifting process.

If these results are compared with those obtained in another test on the same composite resins and shades²⁴, it seems that, under the study's experimental conditions, water leads to a greater discoloration effect, clinically relevant, while the effect of UV aging seems to be clinically negligible. This finding is in agreement with another study on recent formulation composites²⁵. Moreover, it is important to underline that the statistical analysis revealed a different behaviour of the materials following water and UV aging. As a matter of fact, Spectrum TPH was the material mostly affected by water aging, whereas UV aging had the most effective influence on Tetric Ceram. Z100 exhibited overall good color stability, even if it never had the best results both in water and UV discoloration.

Conclusion

The study showed that UV irradiation acted as a discoloring agent to different degrees for all the tested materials. Taking the value $\Delta E=3.3$ as the limit for clinical acceptability in discoloration, only one of the materials tested (Tetric Ceram A4) exhibited a discoloration that could be detected clinically. Nevertheless, none of the three materials selected for this study performed statistically significantly worse than the other two. This may be explained by the different nature of the resin matrix. Also opacity resulted to be influenced by UV aging, even if a clinical limit for visual acceptability of opacity shift has not been proposed in the literature yet.

Further studies are necessary in order to better understand the process of discoloration of resin-based composites,

evaluating the various parameters that affect clinical discoloration and differentiating between surface and internal discoloration.

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Table II. X, Y, Z color values and calculated ΔOpacity

Material	Shade	Back	X	Y	Z	Opacity	X	Y	Z	Opacity	Delta Opacity
Spectrum TPH	A2	White	54,36	56,28	39,97		54,11	56,32	39,86		
Spectrum TPH	A2	Black	34,69	36,56	30,61	65,0	34,75	36,80	30,33	65,3	-0,3
Spectrum TPH	A3	White	48,07	49,55	33,33		48,07	49,77	32,81		
Spectrum TPH	A3	Black	30,85	32,49	26,16	65,6	31,35	33,15	25,84	66,6	-1
Spectrum TPH	A3.5	White	49,56	50,36	29,64		49,74	50,67	30,17		
Spectrum TPH	A3.5	Black	31,71	33,11	24,10	65,7	31,92	33,45	24,35	66,0	-0,3
Spectrum TPH	A4	White	39,54	40,23	22,82		39,14	39,86	23,34		
Spectrum TPH	A4	Black	25,06	26,15	18,96	65,0	25,37	26,49	18,62	66,5	-1,5
Spectrum TPH	B2	White	50,96	53,07	34,55		50,77	53,02	35,00		
Spectrum TPH	B2	Black	32,57	34,56	27,26	65,1	32,51	34,58	27,31	65,2	-0,1
Spectrum TPH	B3	White	44,86	45,78	26,88		44,98	46,04	27,06		
Spectrum TPH	B3	Black	29,15	30,55	22,06	66,7	29,29	30,77	22,00	66,8	-0,1
Tetric Ceram	A2	White	56,22	58,67	37,57		58,26	60,69	41,22		
Tetric Ceram	A2	Black	32,83	35,15	28,68	59,9	33,97	36,30	30,67	59,8	0,1
Tetric Ceram	A3	White	59,03	61,28	36,16		60,07	62,17	40,20		
Tetric Ceram	A3	Black	34,07	36,38	27,94	59,4	34,94	37,23	30,34	59,9	-0,5
Tetric Ceram	A3.5	White	54,76	56,01	33,67		55,36	56,52	35,30		
Tetric Ceram	A3.5	Black	31,35	33,17	25,66	59,2	32,39	34,20	27,43	60,5	-1,3
Tetric Ceram	A4	White	46,61	47,14	23,65		47,09	47,57	26,09		
Tetric Ceram	A4	Black	29,29	30,79	20,36	65,3	29,61	31,07	21,71	65,3	0
Tetric Ceram	B2	White	59,30	62,31	43,22		59,83	62,73	46,03		
Tetric Ceram	B2	Black	34,06	36,54	31,67	58,6	34,43	36,88	32,93	58,8	-0,2
Tetric Ceram	B3	White	51,59	53,52	29,64		52,68	54,59	32,16		
Tetric Ceram	B3	Black	31,74	33,91	24,08	63,4	31,96	34,10	25,29	62,5	0.9
Z100	A2	White	53,24	55,98	39,21		52,03	54,42	37,24		
Z100	A2	Black	32,87	35,15	29,68	62,8	32,26	34,42	28,42	63,2	-0,4
Z100	A3	White	53,63	55,68	37,09		52,15	53,76	34,97		
Z100	A3	Black	35,29	37,47	30,29	67,3	34,69	36,65	28,89	68,2	-0,9
Z100	A3.5	White	48,72	50,97	31,41		47,61	49,29	29,75		
Z100	A3.5	Black	29,88	32,00	24,57	62,8	29,42	31,28	23,50	63,5	-0,7
Z100	A4	White	47,31	49,18	30,27		46,38	47,85	28,92		
Z100	A4	Black	30,28	32,23	24,51	65,5	29,88	31,63	23,63	66,1	-0,6
Z100	B2	White	56,72	59,32	42,57		55,44	57,73	39,87		
Z100	B2	Black	36,82	39,20	33,66	66,1	36,16	38,43	31,74	66,6	-0,5
Z100	B3	White	52,47	54,46	33,13		51,65	53,31	31,63		
Z100	B3	Black	34,14	36,32	27,49	66,7	33,68	35,68	26,21	66,9	-0,2

Before treatment
After treatment