

EFFECT OF ENERGY DENSITY ON COLOR STABILITY IN DENTAL RESIN COMPOSITES UNDER ACCELERATED AGING

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ABSTRACT

The effects of the energy density that is used for polymerization on properties of dental resin composites are well known. However, few studies relate color stability to this factor. The aim of this study was to assess color changes (ΔE^*), *in vitro*, in terms of accelerated aging under UV exposure of specimens prepared with different energy densities. Four commercial dental resin composites were included in the study. Thirty six specimens were prepared for each one of them, following the procedure established by ISO 4049 Standard, and assigned to three groups: A (3.75 J/cm²), B (9 J/cm²), C (24 J/cm²). Each group was further subdivided into four subgroups: 1 (no aging), 2 (500 hours aging), 3 (1000 hours aging) and 4 (1500 hours aging). The results were analyzed by means of ANOVA and Tukey's test ($\alpha = 0.05$) to

determine the effect of the factors. Correlation was performed in order to determine the possible relationship among variables.

Energy density is not a significant factor in color stability. However, aging is directly proportional to color changes. ΔE^* depends on filler size; hybrid material presented ΔE^* of 2.1(0.5), 2.4(0.6) and 3.3(0.3) at 500, 1000 and 1500 hours of accelerated aging respectively, and nanofilled material showed ΔE^* of 3.0(0.6), 4.5(1.2) and 5.9(0.6) at the same times respectively. It can be concluded that ΔE^* does not depend on energy density; however other factors are involved in color change. Further studies in this area are warranted.

Key words: dental composite, color stability, accelerated aging, energy density, UV.

EFFECTO DE LA ENERGÍA DE POLIMERIZACIÓN SOBRE LA ESTABILIDAD DE COLOR EN COMPOSITOS DENTALES SOMETIDOS A ENVEJECIMIENTO ACELERADO

RESUMEN

Los efectos de la energía de polimerización sobre las propiedades físicas y mecánicas de las resinas compuestas utilizadas en odontología han sido ampliamente analizados. Sin embargo, existen pocos trabajos donde se relaciona la estabilidad de color con la energía aplicada al sistema al momento de iniciar la polimerización. El objetivo de este estudio fue la valoración, *in vitro*, del cambio de color (ΔE^*) de resinas compuestas, en función del tiempo sometido a envejecimiento acelerado por UV, utilizando probetas elaboradas con diferentes cantidades de energía luminica. Cuatro marcas comerciales de composites fueron incluidas en el estudio. Con cada una de ellas, se elaboraron 36 especímenes siguiendo los procedimientos indicados por la Norma ISO 4049/7.12. Se dividieron en tres grupos de 12 unidades cada uno, de acuerdo con la energía de polimerización: "A" (3.75 J/cm²), "B" (9 J/cm²) y "C" (24 J/cm²). A su vez, se subdividieron en: "1" (sin envejecimiento), "2" (500 horas de envejecimiento), "3" (1000 horas de envejecimiento) y "4" (1500 horas de envejecimiento). Los resultados se analizaron por medio de ANOVA y prueba de Tukey ($\alpha = 0.05$) para determinar el

efecto de los factores considerados. Un estudio de correlación fue realizado para determinar la posible relación entre las variables.

La energía de polimerización no es un factor significativo en la estabilidad del color. Sin embargo, el tiempo de envejecimiento es directamente proporcional al cambio de color. De acuerdo con los resultados obtenidos, el tamaño de partícula del relleno inorgánico es un factor que influye significativamente en los valores de ΔE^* . Los materiales híbridos presentaron, bajo envejecimiento acelerado, valores de ΔE^* de 2.1 (0.5), 2.4 (0.6) y 3.3(0.3) a las 500, 1000 y 1500 horas, respectivamente, mientras que las resinas con nanorelleno mostraron un ΔE^* de 3.0(0.6), 4.5(1.2) y 5.9(0.6) en los mismos tiempos y bajo las mismas condiciones. Puede concluirse que la variación total de color no depende de la energía de polimerización, pero otros factores inherentes al material pueden estar involucrados para acentuar estos cambios. Es importante el desarrollo de más estudios tomando en consideración estos aspectos.

Palabras clave: composite dental, energía de polimerización, estabilidad de color, envejecimiento acelerado, UV.

INTRODUCTION

One of the main development points in restorative dental materials has been the necessity to create highly aesthetic dental restorations; color stability and curing energy density are important factors for achieving this goal.

Since dental resin composites appeared in 1956¹, the great advantage over dental amalgam, is the "tooth color" they give the restorations. For this reason, color stability has been extensively studied²⁻⁴. The technological development of the CIELa*b* system allows for quantitative assessment of this factor^{5,6}.

Buchalla et al.⁷ reported that color difference (ΔE^*) in resin discs, increased after storage for one month under artificial day light and that the difference increased under the influence of water storage.

Erosion of the organic matrix and exposure of the inorganic filler, are the possible causes for color changes, once the specimens have been subjected to aging in weathering chambers. Powers et al.⁸⁻¹⁰ found that the material becomes darker and more opaque as the time under weathering conditions progresses. This fact was corroborated by the finding that the values of L* and a* diminish, while b* increases, when resin composite specimens are submitted to the action of UV and humidity¹¹.

Energy density, in light-cured resin composites, is a factor related to physical, chemical and mechanical

characteristics; it has been proven that the superficial hardness, flexural strength, sorption, solubility and degree of conversion of these materials, depend on this energy¹²⁻²¹.

Hosoya²² assessed in vitro color in dental composites as related to energy density in a five year study, concluding that the color change modes of specimens differed for the different color shades and light curing times. However, few studies have addressed the issues of color stability and curing energy density. The purpose of this study was to relate energy density to color stability of dental resin composites in terms of exposure time to accelerated aging by UV.

MATERIALS AND METHODS

Two hybrid composites (Filtek Z250 and Tetric Ceram) and two nanofilled composites (Filtek Supreme and Tetric EvoCeram), were selected as representative restorative materials. Manufacturing information is provided in Table 1.

Thirty six specimens for each material were made, using a stainless steel mold of 15mm-diameter x 1mm-depth. The specimens were divided in three groups of twelve (according to the energy output): "A" (3.75 J/cm²), "B" (9 J/cm²) and "C" (24 J/cm²), and each one of them, was further subdivided into four groups: "1" (no aging), "2" (500 hours aging), "3" (1000 hours aging) and "4" (1500 hours aging).

TABLE 1. Products used in this study

Material	Shade	Organic Matrix	Filler			Manufacturer	Batch
			Type	% (vol)	Size		
F Z250	A3	Bis-GMA UDMA Bis-EMA	Zirconia/ Silica	60%	0.001-3.5 μ m	3M ESPE	4LK
F. Supreme	Body A3	Bis-GMA TEGDMA UDMA Bis-EMA	Zirconia/ Silica	60%	5 – 20 nm	3M ESPE	5GK
Tetric Ceram	A3	Bis-GMA UDMA TEGDMA	Yterbium trifluoride Ba- Al- fluorsilicate glass Barium glass silica	60%	0.04 – 3 μ m	Ivoclar Vivadent	G133D1
Tetric EvoCeram	A3	Bis-GMA UDMA TEGDMA	Yterbium trifluoride Barium glass	55%	40 – 3000 nm	Ivoclar Vivadent	G169D7

TABLE 2. Reference values

Material	Energy [J/cm ²]	L*	a*	b*
Tetric Ceram	9	67,2	-2,5	15,8
F. Z-250	9	59,7	-1,1	9,9
F. Supreme	9	55,5	0,4	10,8
Tetric EvoCeram	9	61,9	-1,1	10,9

After the mold was filled with the resin composite paste, a glass slide covered with a polyethylene sheet was applied to remove excess. This sheet remained on the material while light-polymerization was performed, using a commercial halogen unit (Spectrum 800 Denstply Caulk, Milford, DE, USA) at a current voltage kept constant by means of an automatic stabilizer. Before curing each specimen, the intensity of light irradiation was verified with a digital radiometer (Cure Rite Model # 800 EFOS Incorporation Williamsville USA). The cure was performed according to ISO 4049/7.12 and all the discs were prepared by a single operator.

Composite discs were placed in a weathering chamber (Accelerated Weathering Tester, model QUV/Basic, Q-Panel Lab. Products Cleveland, Ohio USA), using fluorescent tubes UVB 313 with a maximum peak of 313 nm and to 100% of relative humidity, applying a cycle of 4 hours of ultraviolet radiation at 60°C and 4 hours of vapor condensation at 40°C. Each subgroup was exposed for the previously described times. After that, specimens were polished initially with abrasive paper 600 and finally with abrasive paper 1000.

Color was evaluated on a white background, using a spectrophotometer (Color-Eye 7000, Gretag-Macbeth LLC, New Windsor, NY, USA). Spectral reflectance values were recorded in a range of 360-750 nm, with increases of 10 nm, and converted to CIELa*b* values. The total color difference (ΔE^*) was calculated as follows:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

Where:

ΔE^* = Total color difference.

ΔL^* = Difference of L* values between reference and study specimen

Δa^* = Difference of a* values between reference and study specimen

Δb^* = Difference of b* values between reference and study specimen

TABLE 3. Mean values and SD of ΔE^*

Material	Aging [h]	Energy [J/cm ²]	E*	S.D.	N
Tetric Ceram	500	3,75	2,3	0,8	3
		9	1,9	0,8	3
	1000	24	2,7	0,6	3
		3,75	2,9	0,3	3
	1500	9	3,1	0,2	3
		24	3,0	0,4	3
Z-250	500	3,75	2,0	0,5	3
		9	1,7	0,2	3
	1000	24	2,4	0,3	3
		3,75	1,6	0,3	3
	1500	9	2,2	0,4	3
		24	2,1	0,4	3
F. Supreme	500	3,75	2,8	0,3	3
		9	2,5	0,1	3
	1000	24	2,6	0,5	3
		3,75	3,6	0,2	3
	1500	9	3,2	0,1	3
		24	3,4	0,2	3
T. EvoCeram	500	3,75	4,0	0,1	3
		9	3,7	0,5	3
	1000	24	2,9	0,4	3
		3,75	6,1	0,3	3
	1500	9	5,0	0,5	3
		24	6,0	0,3	3

For each material the average of subgroup B1 (9 J/cm² and no aging) was taken as the reference value, as described in Table 2.

Analysis of variance was used to evaluate the effect of the experimental variables (energy density, time of aging and material) on ΔE^* (P<.05). Correlation and lineal regression were performed to determine the possible relationship among the variables. The subgroups "A1" and "C1" were eliminated for the statistical analysis, because the color stability was analyzed as a function of aging time.

Factor	DF	Mean Square	F	P
MATERIAL	3	39,7	267,7	0,00
AGING	2	38,7	260,9	0,00
ENERGY	2	0,5	3,1	0,05
MAT*AGI	6	3,7	24,7	0,00
MAT*ENE	6	0,3	2,0	0,08
AGI*ENE	4	0,1	0,8	0,53
MAT*AGI*ENE	12	0,6	4,0	0,00
Error	72	0,1		
Total	108			

Material	N	Correlation coefficient	R ²	p
Tetric Ceram	27	0,71	0,50	< 0,05
F. Z-250	27	0,72	0,52	< 0,05
F. Supreme	27	0,93	0,87	< 0,05
Tetric EvoCeram	27	0,86	0,74	< 0,05

RESULTS

Table 3 lists arithmetic means and standard deviations for ΔE^* results. No statistical significance ($P=0.05$) was found for the effect of energy density, but significant differences ($P<0.01$) were found among materials and aging times (Table 4).

Non significant interaction between material and energy density allowed us to include all materials in Tukey's test. Statistically significant differences were observed among the groups analyzed (Table 5). The correlation analysis (Table 6) showed that the aging time influenced ΔE^* significantly for each material. Fig. 1 reflects the behavior of ΔE^* through-out aging time, taking the filler size as a factor.

DISCUSSION

The reference values obtained in this study can be compared with the values observed at first sight. Tetric Ceram is the clearest material while Filtek Supreme is the darkest with L^* values of 67.2 and 55.5 respectively. Other studies^{7,23} that used a similar methodology to obtain ΔE^* do not exhibit significant differences with the values obtained in this study.

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Aging [h]	N	1	2	3
500	36	2,6		
1000	36		3,5	
1500	36			4,7
Significance		1	1	1
Tukey test $\alpha=0.05$				

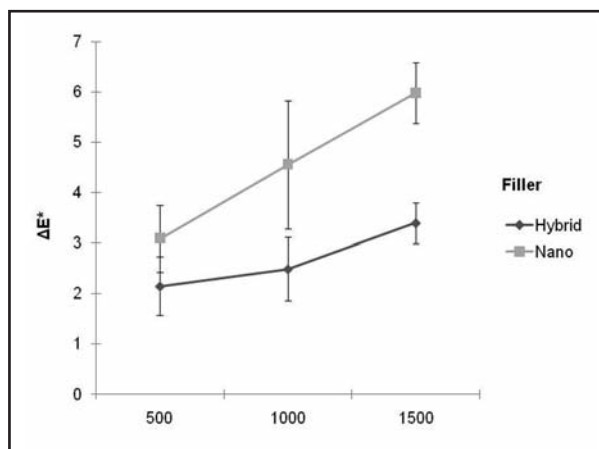


Fig. 1: Behavior of ΔE^* including filler size as a factor.

Color stability was similar in all the materials used in the present study before degradation by UV, in agreement with other authors who reported that the aging time is directly proportional to ΔE^* ^{6,9,11}. However, the fact that no statistically significant differences were found between different energy density groups suggests that the hypothesis should be rejected. Thus, under the conditions of this study, the energy density is not a decisive factor in color stability.

Paravina et. al.¹¹ determined a ΔE^* value of 3.3, as a perceptible change in color. Employing this parameter, and in agreement with the data presented in graph 1, nanofilled materials would change more quickly than hybrid materials. It is known that color changes under UV are the result of degradation products in the organic matrix^{9,10}. The pre-polymers included in nanofilled materials might facilitate the early appearance of these products. For this reason, it is important to carry out further investigation on this aspect.

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