

Color Stability of Restorative Resins Under Accelerated Aging*

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The color stability of seven commercial composite resins, an unfilled resin, and three glazes was studied under conditions of accelerated aging by reflection spectrophotometry and visually with Munsell color tabs. After aging for 900 hours, most of the resins had lower values of luminous reflectance and excitation purity and higher values of dominant wavelength and contrast ratio compared to values at baseline.

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Introduction.

Tooth-colored restorative resins have characteristically been unable to retain the shade developed at the time of insertion.^{1,2} Stain accumulation, dehydration, water sorption, and the chemical breakdown of unreacted components are factors contributing to color instability. Studies have been conducted to simulate clinical stain accumulation using organic dyes and foodstuffs^{3,4} and have related discoloration to surface texture and finishing technique. Resin materials accelerated by ultraviolet (uv) radiation have been shown to be less color stable than amine-accelerated systems during the early period after polymerization.⁵ The American Dental Association Specification No. 27 for Direct Filling Resins includes a test for color stability in which one-half of a disk is exposed to radiation from a light source (S-1 bulb) in a dry environment and com-

pared to the unexposed half for visually perceptible color change.⁶ It is the intent of this study to simulate clinical shade deterioration using controlled weathering conditions and monitor color changes with frequent visual and spectrophotometric evaluations.

Materials and methods.

The color of seven commercial composite resins, three glazes, and an unfilled restorative resin was evaluated under conditions of accelerated aging. The code, shade, batch number, and manufacturer are listed in Table 1 for each material studied.

Five disks (36 mm in diameter and 1.3 mm in thickness) were prepared for each material according to conditions specified in a previous study.⁷ All samples were exposed to conditions of accelerated aging for a total of 900 hours in a weathering chamber* at 43°C and 90 percent relative humidity. One surface of each sample was subjected continuously to the radiation of a 2500 watt xenon light source† with an intermittent water spray for 18 minutes every 102 minutes. Color evaluations were made before weathering (baseline) and after exposure of 10, 20, 50, 100, 300, and 900 hours.

Munsell value, chroma, and hue were determined by two evaluators making visual comparisons with color tabs (glossy finish)‡ for each resin at each time. Color difference (I) between each observation and the consensus color or between consensus colors at baseline and a subsequent time was

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* Weather-Ometer 25-WR, Atlas Electric Devices Co., Chicago, Illinois 60613.

† 12-2881 Xenon Burner, Atlas Electric Devices Co., Chicago, Illinois 60613.

‡ Munsell Book of Color, Munsell Color, Baltimore, Maryland 21218.

TABLE 1
CODE, PRODUCT NAME AND SHADE, BATCH NUMBERS, AND MANUFACTURERS OF
COMPOSITE AND UNFILLED RESINS AND GLAZES.

Code	Product name (shade)	Batch numbers	Manufacturer
Composite resins:			
A	Adaptic (Universal)	base - SF101 catalyst - SF101	Johnson & Johnson Dental Products Division East Windsor, NJ 08520
AR	Adaptic Radi- opaque (Universal)	base - 1126D03 catalyst - 1126D03	Johnson & Johnson
C	Concise (Universal)	base - 6159L13 catalyst - 6159L13	3M Company St. Paul, MN 55101
NF	Nuva Fil (Light)	base - 7414, 7426 initiator - 7661	L. D. Caulk Co. Div. of Dentsply Inter- national, Inc. Milford, DE 19963
P	Prestige (Universal)	base - HPR0114 catalyst - HPR0115	Lee Pharmaceuticals South El Monte, CA 91733
S	Simulate (Universal)	base - 1066 catalyst - 1160	Kerr Manufacturing Co. Div. of Sybron Corp. Romulus, MI 48174
V	Vytol (Light)	base - 042976 catalyst - 042976	L. D. Caulk Co.
Unfilled resin:			
SV	Sevriton (Ss- Light Yellow)	powder - PA14PE liquid - PH6PK	Amalgamated Dental Trade Dist., Ltd. London, England
Glazes:			
F	Finite	base - 041076 catalyst - G0025	Lee Pharmaceuticals
G	Adaptic Glaze	base - 0815D04 catalyst - 0815D04	Johnson & Johnson
NS	Nuva Seal	base - 75104 initiator - 7661	L. D. Caulk Co.

determined by the equation⁸, $I = (C/5)(2\Delta H) + 6\Delta V + 3\Delta C$, where C is the average chroma and ΔH , ΔV and ΔC are the positive differences in hue, value, and chroma, respectively.

Curves of percent reflectance versus wavelength (λ) were obtained for five samples of each resin between 405 and 700 nm with a double-beam, ultraviolet-visible spectrophotometer[§] and integrating sphere.[¶]

Luminous reflectance (Y), dominant wavelength (DW), and excitation purity (EP) were obtained for each sample backed by both a black[∞] and a white** standard.⁷ An estimate of the opacity of each resin was obtained by calculation of the contrast ratio (CR),⁹ Y black standard/Y white standard.

The spectrophotometric parameters (Y, DW and EP) were studied by a two-way

§ ACTA C III UV-Visible Spectrophotometer, Beckman Instruments, Inc., Irvine, California 92664.

¶ ASPH-U Integrating Sphere, Beckman Instruments, Inc., Irvine, California 92664.

[∞] Part No. 375287, Beckman Instruments, Inc., Irvine, California 92664.

**Part No. 104384, Beckman Instruments, Inc., Irvine, California 92664.

TABLE 2
SPECTROPHOTOMETRIC AND MUNSELL COLOR DATA FOR COMPOSITE RESINS AT BASELINE AND AFTER AGING

Code	Property*	Time, hours						
		0	10	20	50	100	300	900
A								
Y	59.4 (1.3)	57.8 (0.7)	58.6 (0.9)	58.9 (0.8)	59.1 (0.6)	58.3 (1.3)	62.8 (1.8)	
DW	578.90 (0.07)	580.39 (0.18)	580.18 (0.18)	579.81 (0.07)	579.72 (0.15)	578.96 (0.13)	579.50 (0.11)	
EP	0.270 (0.004)	0.222 (0.008)	0.212 (0.010)	0.215 (0.012)	0.220 (0.012)	0.252 (0.020)	0.182 (0.025)	
CR	0.675 (0.012)	0.698 (0.021)	0.703 (0.018)	0.707 (0.020)	0.709 (0.026)	0.730 (0.035)	0.827 (0.025)	
Hue Value/Chroma	2.5Y 7.5/3	10YR 7.5/2	10YR 7.5/2	10YR 7.5/3	10YR 7.5/2	2.5Y 7.5/3	10YR 7.5/2	
AR								
Y	61.0 (1.0)	58.2 (1.0)	58.1 (1.2)	57.4 (1.5)	58.6 (1.5)	57.4 (1.1)	62.3 (1.5)	
DW	579.57 (0.18)	580.70 (0.17)	580.44 (0.23)	580.07 (0.16)	579.85 (0.16)	579.26 (0.14)	581.2 (1.9)	
EP	0.265 (0.006)	0.225 (0.009)	0.224 (0.010)	0.226 (0.011)	0.236 (0.012)	0.278 (0.024)	0.180 (0.017)	
CR	0.694 (0.012)	0.721 (0.025)	0.720 (0.028)	0.718 (0.032)	0.721 (0.032)	0.729 (0.039)	0.849 (0.024)	
Hue Value/Chroma	2.5Y 7.5/3	10YR 7.5/2	10YR 7.5/2	10YR 7.5/2	10YR 7.5/2	2.5Y 7.5/4	10YR 8.0/2	
C								
Y	66.1 (1.4)	60.0 (1.5)	59.7 (1.2)	58.4 (1.1)	57.9 (1.0)	56.7 (0.3)	60.8 (2.3)	
DW	578.53 (0.05)	578.99 (0.13)	578.92 (0.11)	578.88 (0.04)	578.97 (0.09)	579.17 (0.24)	581.10 (1.68)	
EP	0.307 (0.007)	0.309 (0.014)	0.317 (0.014)	0.337 (0.016)	0.350 (0.016)	0.365 (0.035)	0.224 (0.017)	
CR	0.658 (0.013)	0.698 (0.029)	0.702 (0.026)	0.703 (0.027)	0.710 (0.025)	0.725 (0.038)	0.840 (0.028)	
Hue Value/Chroma	2.5Y 8.0/4	2.5Y 7.5/4	2.5Y 7.5/4	2.5Y 7.5/4	2.5Y 7.5/4	2.5Y 7.5/5	10YR 8.0/3	
NF								
Y	78.9 (1.5)	62.1 (0.7)	63.7 (1.0)	65.8 (1.1)	70.4 (1.4)	71.7 (1.6)	74.7 (0.7)	
DW	576.11 (0.11)	577.20 (0.32)	576.50 (0.21)	575.59 (0.12)	575.34 (0.08)	575.86 (0.06)	576.47 (0.14)	
EP	0.160 (0.001)	0.334 (0.008)	0.334 (0.006)	0.333 (0.007)	0.310 (0.009)	0.260 (0.015)	0.179 (0.013)	
CR	0.587 (0.010)	0.638 (0.020)	0.635 (0.014)	0.621 (0.014)	0.617 (0.012)	0.615 (0.016)	0.706 (0.017)	
Hue Value/Chroma	5.0Y 8.5/2	2.5Y 7.5/4	5.0Y 8.0/4	5.0Y 8.0/4	5.0Y 8.0/4	5.0Y 8.0/3	5.0Y 8.5/2	

*Obtained for a white background (condition b).

analysis of variance¹⁰ to determine the effects of products and times for the composite resins, unfilled resin, and for the glazes. Data for the contrast ratio were studied by a one-way analysis of variance.¹⁰ Tukey's intervals at the 95 percent level of confidence were calculated¹¹ for comparisons among means. Comparisons among colors determined visually were made by use of an estimate of the critical color dif-

ference⁷ (I_c) necessary to show a difference between two colors. I_c was calculated at the 95 percent level of confidence.

Results.

Mean values and standard deviations of luminous reflectance (Y), dominant wavelength (DW), excitation purity (EP), and

TABLE 3
SPECTROPHOTOMETRIC AND MUNSELL COLOR DATA FOR COMPOSITE AND UNFILLED RESINS AT BASELINE AND AFTER AGING

Code	Property*	Time, hours						
		0	10	20	50	100	300	900
Composite Resins:								
P								
Y	51.6 (1.2)	49.2 (1.1)	49.4 (0.9)	49.5 (0.8)	50.2 (0.8)	49.1 (0.6)	64.4 (1.5)	
DW	578.50 (0.09)	579.12 (0.11)	579.04 (0.11)	579.00 (0.10)	578.89 (0.26)	578.90 (0.03)	583.8 (4.5)	
EP	0.289 (0.005)	0.289 (0.005)	0.288 (0.008)	0.292 (0.008)	0.292 (0.011)	0.311 (0.012)	0.065 (0.010)	
CR	0.742 (0.012)	0.748 (0.016)	0.747 (0.017)	0.744 (0.012)	0.747 (0.015)	0.757 (0.022)	0.933 (0.014)	
Hue Value/Chroma	2.5Y 7.0/3	2.5Y 7.0/3	2.5Y 7.0/3	2.5Y 6.5/3	2.5Y 7.0/3	2.5Y 7.0/3	N8.5	
S								
Y	66.3 (1.6)	62.6 (1.2)	62.5 (1.1)	61.6 (1.3)	61.3 (1.3)	59.3 (1.4)	64.5 (1.5)	
DW	578.63 (0.14)	578.97 (0.11)	578.88 (0.06)	578.83 (0.06)	578.83 (0.05)	578.86 (0.09)	580.37 (0.43)	
EP	0.300 (0.007)	0.307 (0.006)	0.309 (0.005)	0.317 (0.004)	0.324 (0.005)	0.356 (0.008)	0.198 (0.017)	
CR	0.676 (0.012)	0.687 (0.013)	0.684 (0.011)	0.684 (0.012)	0.693 (0.011)	0.694 (0.011)	0.833 (0.004)	
Hue Value/Chroma	2.5Y 8.0/4	2.5Y 7.5/4	2.5Y 7.5/4	2.5Y 7.5/4	2.5Y 7.5/4	2.5Y 7.5/4	10YR 8.0/2	
V								
Y	72.2 (0.4)	61.4 (0.6)	60.0 (1.0)	57.5 (0.5)	58.4 (0.7)	55.8 (0.2)	66.0 (1.2)	
DW	575.91 (0.04)	575.66 (0.10)	575.61 (0.08)	575.63 (0.06)	575.80 (0.03)	576.43 (0.06)	580.1 (2.2)	
EP	0.207 (0.003)	0.213 (0.005)	0.221 (0.003)	0.239 (0.006)	0.254 (0.004)	0.299 (0.006)	0.116 (0.016)	
CR	0.690 (0.009)	0.723 (0.012)	0.730 (0.010)	0.732 (0.007)	0.738 (0.009)	0.743 (0.010)	0.900 (0.013)	
Hue Value/Chroma	5.0Y 8.0/2	7.5Y 7.5/2	7.5Y 7.5/2	7.5Y 7.5/2	5.0Y 7.5/3	5.0Y 7.5/3	5.0Y 8.5/1	
Unfilled Resin:								
SV								
Y	64.7 (1.5)	62.0 (1.6)	61.4 (1.7)	59.6 (1.5)	62.0 (1.7)	61.1 (2.0)	63.6 (3.2)	
DW	576.28 (0.14)	576.28 (0.18)	576.33 (0.10)	576.58 (0.11)	576.72 (0.15)	576.97 (0.10)	576.91 (0.52)	
EP	0.232 (0.020)	0.216 (0.020)	0.214 (0.018)	0.208 (0.018)	0.213 (0.018)	0.201 (0.021)	0.162 (0.046)	
CR	0.796 (0.055)	0.804 (0.052)	0.805 (0.055)	0.813 (0.053)	0.812 (0.052)	0.829 (0.047)	0.876 (0.040)	
Hue Value/Chroma	7.5Y 8.0/2	7.5Y 7.5/2	7.5Y 7.5/2	7.5Y 7.5/2	5.0Y 7.5/2	5.0Y 7.5/2	5.0Y 8.0/2	

*Obtained for a white background (condition b).

TABLE 4
SPECTROPHOTOMETRIC AND MUNSELL COLOR DATA FOR GLAZES AT BASELINE AND AFTER AGING

Code	Property*	Time, hours						
		0	10	20	50	100	300	900
F	Y	67.9 (1.1)	55.4 (0.9)	55.4 (0.7)	56.2 (0.7)	57.2 (0.6)	53.6 (0.8)	43.3 (0.9)
	DW	575.78 (0.19)	577.73 (0.11)	577.82 (0.12)	577.89 (0.05)	577.79 (0.08)	578.47 (0.13)	580.50 (0.19)
	EP	0.294 (0.013)	0.359 (0.006)	0.365 (0.004)	0.393 (0.005)	0.411 (0.007)	0.474 (0.008)	0.577 (0.008)
	CR	0.317 (0.010)	0.325 (0.010)	0.324 (0.009)	0.321 (0.007)	0.331 (0.009)	0.342 (0.010)	0.424 (0.018)
	Hue Value/Chroma	2.5Y 7.5/5	2.5Y 7.5/5	2.5Y 7.0/5	2.5Y 7.5/6	2.5Y 7.5/6.5	2.5Y 7.0/7	10YR 6.0/7
G	Y	80.4 (0.7)	77.5 (0.8)	77.6 (0.6)	78.2 (0.6)	79.9 (0.5)	78.4 (0.6)	72.0 (1.0)
	DW	573.48 (0.26)	574.53 (0.11)	574.71 (0.12)	574.62 (0.11)	574.21 (0.12)	574.29 (0.08)	574.99 (0.11)
	EP	0.154 (0.010)	0.137 (0.010)	0.133 (0.004)	0.130 (0.004)	0.127 (0.005)	0.157 (0.006)	0.238 (0.009)
	CR	0.116 (0.006)	0.107 (0.002)	0.106 (0.004)	0.103 (0.004)	0.107 (0.004)	0.107 (0.001)	0.109 (0.004)
	Hue Value/Chroma	5.0Y 9.0/3	7.5Y 9.0/2	7.5Y 9.0/2	5.0Y 9.0/2	5.0Y 9.0/2	5.0Y 9.0/2	2.5Y 8.5/3
NS	Y	87.7 (1.0)	82.8 (1.5)	82.3 (1.6)	80.6 (1.5)	83.0 (0.9)	81.4 (0.9)	76.9 (1.1)
	DW	571.07 (0.14)	571.09 (0.18)	571.71 (0.13)	572.61 (0.20)	573.29 (0.88)	573.49 (0.08)	574.12 (0.12)
	EP	0.035 (0.006)	0.172 (0.035)	0.136 (0.033)	0.115 (0.022)	0.103 (0.016)	0.134 (0.011)	0.209 (0.012)
	CR	0.102 (0.004)	0.105 (0.004)	0.102 (0.004)	0.102 (0.009)	0.109 (0.007)	0.103 (0.004)	0.096 (0.006)
	Hue Value/Chroma	10Y 9.5/1	10Y 9.0/4	10Y 9.0/3	10Y 9.0/2	7.5Y 9.0/2	7.5Y 9.0/2	5.0Y 8.5/3

*Obtained for a white background (condition b).

contrast ratio (CR) are listed in Tables 2 and 3 for the composite resins and the unfilled resin, and in Table 4 for the glazes at baseline and after aging. Data obtained for a white standard are presented. The consensus values of Munsell color determined against a white background are also listed in Table 2 - 4.

Differences among materials and among times and their interaction were significant at the 95 percent level of confidence for the three spectrophotometric parameters, and the contrast ratio for the composite and unfilled resins. Analysis of variance for the glazes was made and similar results were found. Tukey's intervals at the 95 percent level for comparisons of means among products and among times are listed in Table 5 for the composite and unfilled resins and for the glazes.

Mean values of luminous reflectance (Y) versus time of aging are plotted in Figure 1 for five resins. The curve shown for A is representative of the behavior of Ar, C, S, and SV. Among resins listed in Tables 2 and 3 the maximum change in Y varied from 4.9 for AR to 9.4 for C. The behavior of NF, P, and V was unique even though the maximum change in Y only varied from 15.3 to 16.8. After aging for 900 hours, five resins (C, NF, S, SV, and V) were darker, and three resins (A, AR, and P) were lighter than at baseline. The luminous reflectance of the glazes decreased from zero to 900 hours, but the change in Y was more dramatic for F than for NS or G.

Mean values of dominant wavelength for the resins and glazes increased after 900 hours of aging by 4.2 to 5.3 nm for V, F, and P; from 2.6 to 3.0 nm for C and NS;

TABLE 5
TUKEY'S INTERVALS FOR COMPARISONS AMONG MEAN VALUES OF SPECTROPHOTOMETRIC DATA

Comparisons	Tukey's Intervals			
	Luminous Reflectance (Y)	Dominant Wavelength, nm	Excitation Purity	Contrast Ratio
Among composite and unfilled resins	0.9	0.11	0.010	0.021
Among times for composite and unfilled resins	0.8	0.09	0.008	0.017
Among glazes	0.6	0.15	0.009	0.004
Among times for glazes	1.0	0.26	0.016	0.007

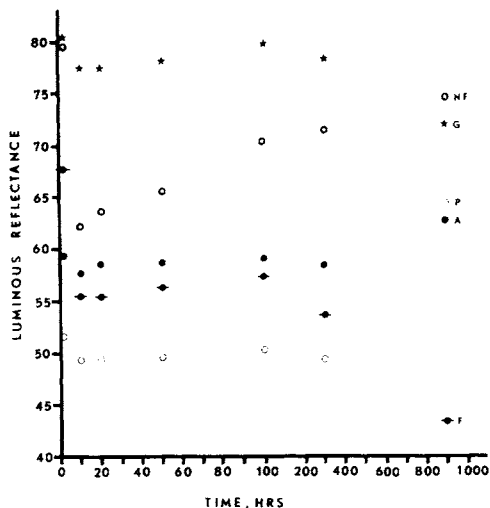


Fig. 1 - Luminous reflection of selected restorative resins versus time of aging. Note that the 900 hour data points also serve as the key.

from 1.5 to 1.7 nm for G, AR, and S; and by less than 0.6 nm for A, NF, and SV as compared to baseline.

Mean values of excitation purity (EP) versus time of aging are plotted in Figure 2 for five resins. The curve shown for A is representative of the behavior of AR and SV, whereas that for P is representative of C, S and V. The behavior of NF was unique. After aging for 900 hours, the change in EP was from -0.070 to -0.102 for SV, C, AR, A, V, and S, respectively, -0.224 for P and +0.019 for NF. Among the glazes tested, the

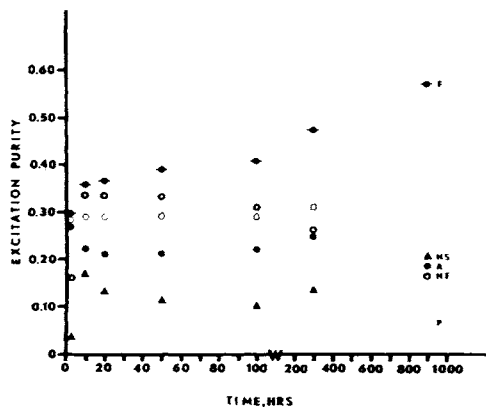


Fig. 2 - Excitation purity of selected restorative resins versus time of aging. Note that the 900 hour data points also serve as the key.

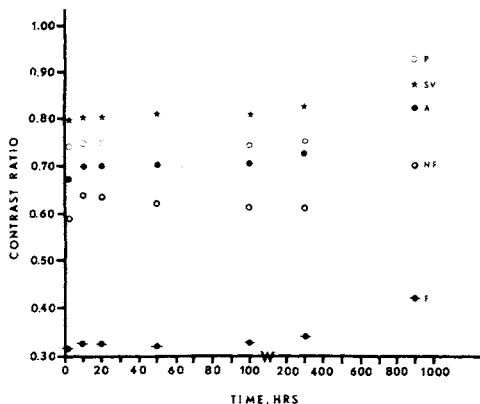


Fig. 3 - Contrast ratio of selected restorative resins versus time of aging. Note that the 900 hour data points also serve as the key.

behavior of F was unique, whereas the curves of NS and G were similar from 10 to 900 hours.

Mean values of the contrast ratio (CR) versus time of aging are plotted in Figure 3 for five resins. The curve shown for A is representative of the behavior of AR and S for which CR increased by about 0.15 after 900 hours of aging. The curve shown for P is representative of C and V for which CR increased by 0.18 for C to 0.21 for V. The resins NF and SV showed increases of 0.12 and 0.08, respectively, after 900 hours. Among the glazes, F showed an increase of 0.11, whereas both G and NS showed a decrease in CR of less than 0.01 after 900 hours.

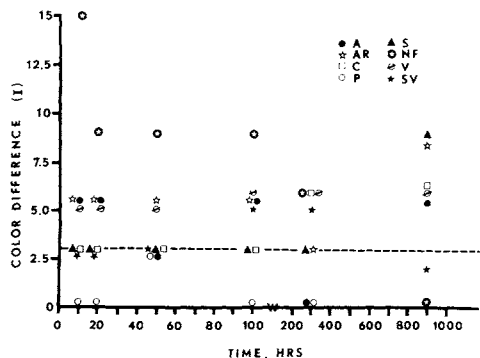


Fig. 4 - Color difference between baseline and aged samples for composite and unfilled resins. Dashed line indicates value of I_C . Value for I of P at 900 hours could not be calculated.

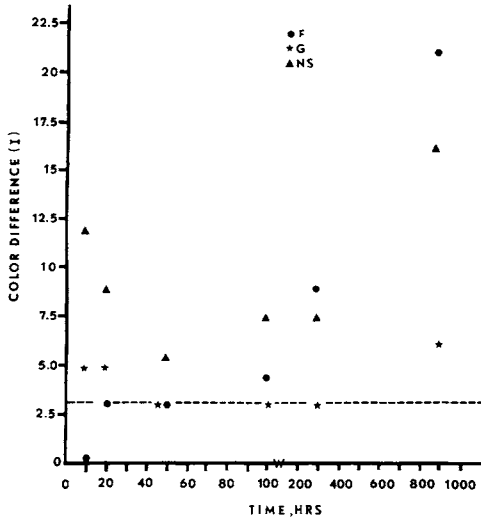


Fig. 5 - Color differences between baseline and aged samples for glazes. Dashed line indicates value of I_c .

The color differences (I) between each time of aging and baseline determined from the consensus values of Munsell color are plotted in Figure 4 for the composite and unfilled resins, and in Figure 5 for the glazes. The critical color difference (I_c) was 3.1 as indicated by a dashed line in each figure. After 10 hours of aging, significant changes in color were observed in four composite resins (A, AR, NF, and V), and in glazes G and NS. After 900 hours of aging, however, only two restorative resins (NF and SV) had values of I less than I_c . The value of I for P after 900 hours could not be calculated because P was achromatic.

Discussion.

The xenon light source of the weathering instrument is a long arc, water-cooled type which has an energy distribution curve that approximates that of noon summer sunlight at Chicago, Illinois.¹² The borosilicate glass filter used with the light source has a low cutoff at 275 nm so that the radiant energy is very close to natural unfiltered sunlight. The manufacturer of the weathering instrument estimates that 300 hours of aging are equivalent to one year of clinical service.

The ability to predict long-term changes

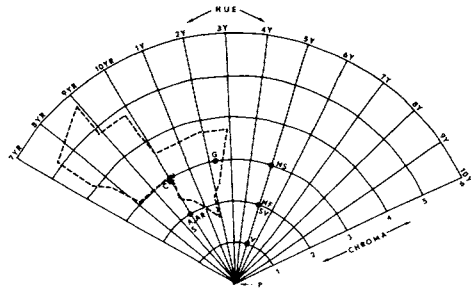


Fig. 6 - A comparison of the color distribution for hue and chroma between restorative resins after aging for 900 hours and human teeth.¹³ F is off scale with a hue of 10YR and a chroma of 7.

in the color of restorative resins based on short-term accelerated aging is desirable, but may not be achievable. For aging up to 300 hours, the spectrophotometric parameters of the resins were more or less equal to values obtained after 10 hours of aging, except for F and NF. Between 300 and 900 hours of accelerated aging, however, dramatic changes in color occurred for most of the resins that would not have been anticipated from earlier behavior.

Among the resins tested at baseline, only A, AR, and P were within the range for hue and chroma of human teeth, whereas A, AR, P, SV, and V were within the range for value and chroma of human teeth as reported previously.⁷ After aging for 900 hours, only G is within the range of hue and chroma for human teeth (see Figure 6) as reported by Sproull.¹³ A, AR, C, and S possess too little and F too much chroma, whereas NF, NS, P, SV, and V are outside the range for a natural hue. AR, S, and SV fall within the range for value and chroma of human teeth¹³ after aging for 900 hours (see Figure 7). The materials C, G, NF, and NS after aging are too light in value for the amount of chroma displayed, whereas A, P, and V show insufficient chroma and F shows too much chroma compared to the range for human teeth. The failure of most resins to fall within the range of color for human teeth is exaggerated by the dramatic increase in opacity of the resins (except G and NS) that would result in less effective transmission of the color of the supporting tooth in a clinical situation.

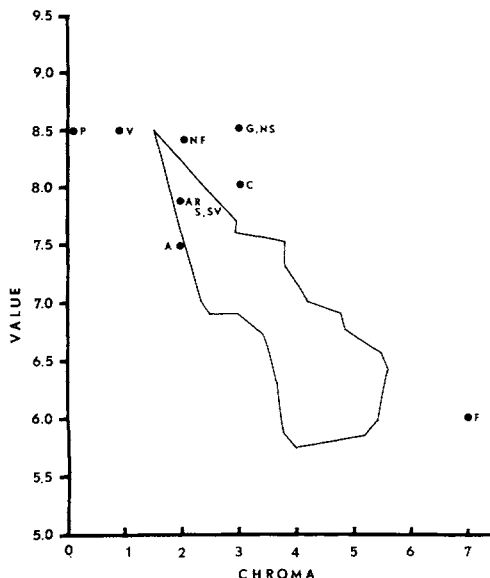


Fig. 7 — A comparison of the color distribution for value and chroma between restorative resins after aging for 900 hours and human teeth.¹³

Conclusions.

The color stability of seven commercial composite resins, an unfilled resin, and three glazes was studied under conditions of accelerated aging by reflection spectrophotometry and visually with Munsell color tabs. After aging for 900 hours, most of the resins had lower values of luminous reflectance and excitation purity and higher values of dominant wavelength and contrast ratio compared to values at baseline. After 10 hours of aging, significant visual changes in color were observed in four composite resins and in two glazes. After 900 hours, all but one composite and an unfilled resin showed visual changes in color that were different statistically from baseline. For some of the resins, changes in color that were observed at 900 hours could not have

been predicted from data obtained over a shorter period of aging.

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