# An accelerated test for color stability of restorative resins

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The clinical relevance of previous tests for color stability of restorative resins has never been established. The present study has demonstrated that the color change produced in 15 proprietary composite resins by storing for one month in water at 60°C is well correlated with the color change obtained after storing for 12 months in water at 37°C. Thus, the former test may be assumed to be a clinically relevant accelerated test for color stability of restorative resins.

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It is well known that restorative resins tend to discolor during service in the oral cavity (2, 3). The discoloration may arise for a number of reasons, including an internal color change of the resinous materials (5). For screening purposes and in research an accelerated test for color stability is desirable. Irradiation with UV-light has been advocated as such a test (8, 9). The introduction of UV-light absorbers into the proprietary resins has reduced the color change provoked by UV-light, but the resins continue to discolor in clinical tests. Further, it has been demonstrated that the ranking of the materials with respect to color stability depends on the source of UV-light (6). Thus, tests by means of UV-light may seem to be of restricted validity.

Dry heating at 60°C for 23 hours constitutes another test for color stability (8) and still other methods have been employed (4). However, the relevance of the various methods has never been established.

It was the aim of the present study to search for an accelerated test for color stability that can be assumed clinically relevant. The underlying assumption of the study was that the color change produced by long-lasting storage in water at 37°C is a reliable measure of the discoloring potential in the clinical situation. An accelerated laboratory test that correlates well with the above test would then presumably be of clinical relevance.

## MATERIALS AND METHODS

Table 1 shows the brands of composite resin that were included in the investigation.

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Table 1. *List of brands used in the investigation*

| Code | Name | Batch No. | Manufacturer |
|---|---|---|---|
| A | Adaptic | 7H 030 | Johnson & Johnson<br>New Jersey, USA |
| B | Concise | cat.: 7270J28<br>uni.: 7300N13 | 3M Company<br>Minnesota, USA |
| C | Concise<br>Cap-C-Rynge | 82891 | 3M Company<br>Minnesota, USA |
| D | Cosmic | cat.: WL 6 YA<br>base: WL 8 YA | Amalgamated Dental<br>London, England |
| E | Durafill | AUG. 79/12 | Kulzer & Co.<br>Bad Homburg, W. Germany |
| F | Estic | cat.: 40906<br>base: 41015 | Kulzer & Co.<br>Bad Homburg, W. Germany |
| G | Estic<br>Microfill | cat.: MAJ 79/48<br>base: APRIL 79/26 | Kulzer & Co.<br>Bad Homburg, W. Germany |
| H | Fotofil | 8 G 818 | Johnson & Johnson<br>New Jersey, USA |
| I | Isopast | / x7 / | Vivadent<br>Schaan, Liechtenstein |
| K | Isopast<br>Variant | 121/078 | Vivadent<br>Schaan, Liechtenstein |
| L | Nimetic | cat.: C 053<br>base: C 050 | Espe<br>Seefeld, W. Germany |
| M | Nuva-Fil | 08 11 78 | L. D. Caulk Co.<br>Delaware, USA |
| N | Prestige | cat.: HPR 0126<br>uni.: HPR 0125 | Lee Pharmaceuticals<br>California, USA |
| O | Silar | cat.: 8 B 2<br>uni.: 8 B 1 | 3M Company<br>Minnesota, USA |
| P | Uvio-Fil | E 071 | Espe<br>Seefeld, W. Germany |

The materials were polymerized in cylindrical brass molds (h = 2 mm, d = 15 mm), covered by an air-impervious matrix. The chemically activated materials were mixed in accordance with the manufacturers' instructions, except that the hand-mixed materials were mixed for 45 seconds. The light activated materials received several exposure doses from both sides through a transparent matrix band. The specimens were produced at room temperature and allowed to cure at 37°C for 24 hours before removal from the molds. The specimens were then polished on both sides with carborundum paper No. 1000 to a final thickness of 1.80–1.85 mm. After a further cure of 6 days at 37°C the specimens were placed on a white backing and their color measured by means of a Hunterlab D25–A colorimeter (Hunterlab, Fairfax, Virginia, USA). The colors were expressed in terms of the Hunter values L, a and b, defined by

Table 2. *Color change of the investigated brands in relation to temperature and storage time*

| Code | 37°C | 50°C | | 60°C | | 70°C | |
|---|---|---|---|---|---|---|---|
| | 12 mo | 1 mo | 2 mo | 1 mo | 2 mo | 1 mo | 2 mo |
| A | 1.2 | 1.1 | 1.9 | 2.2 | 4.7 | 7.4 | 10.8 |
| B | 2.2 | 2.3 | 3.0 | 3.7 | 4.7 | 4.5 | 6.0 |
| C | 3.6 | 4.5 | 4.3 | 4.0 | 4.1 | 5.1 | 8.1 |
| D | 5.7 | 4.6 | 6.2 | 7.4 | 10.3 | 14.0 | 17.8 |
| E | 2.1 | 1.8 | 2.2 | 2.4 | 3.1 | - | - |
| F | 1.4 | 2.9 | 3.1 | 2.9 | 2.4 | 3.3 | 4.7 |
| G | 4.3 | 3.8 | 5.5 | 5.9 | 7.8 | 10.9 | 14.4 |
| H | 5.7 | 5.0 | 5.2 | 5.5 | 6.6 | 7.2 | 10.6 |
| I | 7.1 | - | - | 7.2 | 9.9 | - | - |
| K | 12.5 | 8.5 | 12.9 | 12.5 | 17.9 | 20.1 | 8.4 |
| L | 1.0 | 0.6 | 1.3 | 2.6 | 3.9 | 4.7 | 6.4 |
| M | 2.3 | 1.6 | 1.8 | 2.4 | 2.8 | 1.8 | 2.5 |
| N | 12.8 | 5.7 | 9.3 | 9.1 | 13.1 | 12.0 | 15.0 |
| O | 6.1 | 2.5 | 4.9 | 6.8 | 10.7 | 13.2 | 20.7 |
| P | 0.8 | 0.8 | 0.7 | 1.0 | 1.2 | 1.5 | 1.7 |

$$L = 100\,(Y/Yo)^{1/2}$$

$$a = \frac{175\ (X/Xo - Y/Yo)}{(Y/Yo)^{1/2}}$$

and

$$b = \frac{70\ (Y/Yo - Z/Zo}{(Y/Yo)^{1/2}}$$

In these expressions X, Y and Z are the CIE tristimulus values and Xo, Yo and Zo the tristimulus values of the CIE standard illuminant C (7).

The specimens were then placed in demineralized water at 37, 50, 60 or 70°C, all temperatures being constant within ± 1°C. As the formation of color is a process of oxidation, free access of air to the water baths was secured by means of a hole in the stopper of the glass containers. The specimens were not exposed to daylight or other light sources during storage.

Every month for one year the specimens were removed from the storage water and brushed with a soft tooth brush to remove any bacteria or fungoid growth. The specimens were then wiped dry and the color measured.

For each specimen the color change, ΔE, was calculated according to the formula:

$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$, in which ΔL, Δa and Δb represent the change in the L, a and b values, respectively.

For each brand and temperature three specimens were examined.

## RESULTS AND DISCUSSION

As a rule the specimens became darker, more red and more yellow by the water storage. Table 2 gives the total color changes obtained after 12 months at 37°C, and after 1 and 2 months at 50, 60 and 70°C. Each number represents the mean of the change in color of three

specimens. The mean of the standard deviations for each set of three specimens was 0.4 units of color change.

It appears that color change depends on brand as well as on time and temperature of the storage bath.

For the 15 brands the color change obtained after 12 months at 37°C was correlated with the color change obtained after 1 or 2 months at 50, 60 or 70°C. The coefficients of correlation are presented in Table 3. The first five correlation coefficients are different from zero at the $p = 0.001$ level of statistical significance. The last coefficient differs from zero at the $p = 0.05$ level of significance (1).

Although the first five coefficients are not statistically different from each other ($p > 0.10$) (1) the highest coefficients of correlation were obtained when correlating with the values for 2 months at 50°C and 1 month at 60°C. However, a 1-month test is preferable to a 2-month test; the relationship between color change after 12 months at 37°C and after 1 month at 60°C is shown in Fig. 1.

In conclusion, it was demonstrated that the color change produced in 15 proprietary composite resins by storing for 1 month in water at 60°C is well correlated with the color change obtained after storing for 12 months in water at 37°C. The former test may therefore be assumed to be a clinically relevant accelerated test for color stability of restorative resins.

Table 3. *Coefficients of correlation between color change after 12 months at 37°C and after 1 or 2 months at 50, 60 or 70° C*

| | 50°C | | 60°C | | 70°C | |
|---|---|---|---|---|---|---|
| | 1 mo | 2 mo | 1 mo | 2 mo | 1 mo | 2 mo |
| 37°C 12 mo | 0.87 | 0.95 | 0.95 | 0.93 | 0.82 | 0.49 |

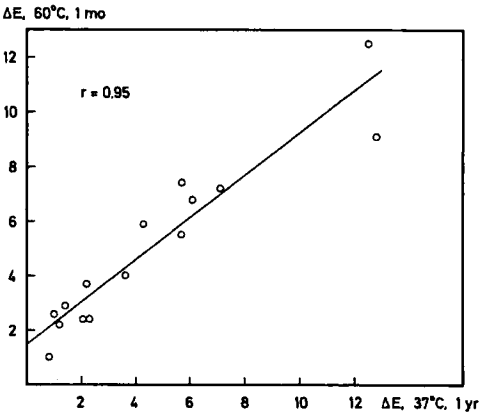


Fig. 1. Relationship between color change produced by storing for 12 months in water at 37°C (abscissa) and for 1 month in water at 60°C (ordinate).

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