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# Color Stability of Expired Restorative Dental Composite Resins

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**Abstract:** *Although expired dental **c**omposites should not be used in practice, limited information on their properties is available. The purpose of this study was to evaluate their color stability. 3 composite resins (ice/SDI, rok/SDI, spectrum/Dentsply) from 15 to 146 months after their expiration date and the same materials 23 to 45 months before their expiration date were used to make round specimens. All specimens were finished and polished and after their initial storage in water of 37$^o$C for 48h, they were immersed in coffee baths simulating 30,60,90,180,360 days of coffee's staining effect. A mean of three measurements of color parameters in the CIELAB system were taken with a portable colorimeter (ShadeEye/Shiofu) at all periods, and data were analyzed statistically by 2way repeated measures ANOVA at a=0.05. Color difference values ($\Delta E^*$) between base line and staining periods for the investigated materials were 17.7 to 47.6 for the new materials and 13.3 to 38.2 for the expired materials. Statistical analysis indicated significant differences between staining periods ($p<0.05$) with all materials, but no significant differences between expired and unexpired materials ($p>0.05$). Although investigated materials had expired for long, their staining behavior within coffee was similar to unexpired. Differences found among materials and immersion time.*
***Keywords :*** *color stability, dental composites, expiration date, staining solution.*

## I. Introduction

The period during which a material fulfils its intended use is defined as shelf life [1], and the end of this shelf life is named expiration date. Composite resins like most of dental materials require a shelf life [2] to assure that their original physical and mechanical properties remain the same at least until its expiration date. A number of parameters are usually considered by the manufacturer to assign an expiration date to a product [3].

A shelf life of a medical device is dependent on the actual device constituents, the presence of preservatives, and storage conditions like temperature, light and humidity since all constituents may influence the functional properties of the material [4]. Performance of composite resins is related to the rate of constituents' degradation over time [5] as a result of oxidative chain scission effects, oxidation hydrolysis, changes in crystallinity or environmentally dependent factors [6]. Polymer matrix and silane coupling agents are those at a greater risk for degradation. Manufacturers determining material's shelf-life may assign a product with a shorter shelf life in order to assure its performance with greater safety. FDA reported that many prescription drugs may retain efficacy past their expiration dates [7]. A shelf life that is usually given to composite resins is about 3-5 years but Hondrum and Femandez [8] showed that light-cured composites retained physical properties for up to seven years regardless storage condition.

Resin restorations with a more natural appearance require the use of composites with a higher variety of shades [9, 10]. As a result, a number of resin materials of specific shade reach their expiration date with a significant amount of material still in the package. Expired composite resins, as most of the medical devices, are neither recommended nor is ethically acceptable to be used in patients. For this reason, if still polymerizable, they are used for diagnostic purposes, for direct mock-ups, for temporary crowns, for repairing margins or faces of temporaries, to stabilize matrix bands etc. Even so, the question of how strongly or which of the properties are affected by the time past their expiration date, still remains.

Only, a few studies investigated the properties of composites past their expiration date. Hardness or microhardness is the property most frequently used as a measure of the expired material deterioration, but the results are not in complete agreement to each other. Penugonda and Penugonda [11] and Boroujeni and Mohamati [12] indicated that composites past their expiration date were still functional with hardness or microhardness not significantly different to unexpired composite. Heiderscheidt et al [13] also showed no significant changes in microhardness, flexural strength and volumetric shrinkage between expired and unexpired composites. However, Tirapelli et al [14] showed a significantly lower microhardness in one of the expired composites compared to not-expired. The change was explained by the incomplete cure of the polymer matrix due to degradation, over time, of the components involved in its polymerization, especially of its initiator [15]. Garcia et al [16] evaluated microhardness as well as surface roughness and degree of conversion of composite resins 180 days after their expiration date, and found inferior properties than those of unexpired materials. This was explained by the increase of remaining monomer in expired products and the creation of polymer without a typical form, both leading to material degradation [16]. D'Alpino et al [17] studied new, aged and expired composites and found that these states influence their crystallinity and magnetic properties. D'Alpino et al [18] also showed that aging and expiration date increases glass transition temperature, and flexural strength but most of the kinetic parameters are not influenced by expiration date [19]. Cytotoxicity of expired composites was also investigated since it is expected that a certain amount of monomers is leaching out with time. Bal et al [20] showed that expiration date of composite resins greatly affects cell's viability.

Material deterioration is not easily detected by the clinician. An effective initiator which is responsible for a normal polymerization propagation rate and eventually the physical/mechanical properties of the composite is easily detected in chemically cured systems. In light cured systems, where polymerization rate is very short, an ineffective initiator is difficult to detect and the material may have fewer free radicals, contain more active groups of decreased mobility, more unreacted monomers and may present premature polymerization of its ingredients. Many mechanical tests are designed to indicate differences among composites in specific properties. Diametral tensile test was suggested as more appropriate than compressive or microhardness test for measuring deterioration of composite materials [21] as it affects the internal coherence of the material. However, it was noted that changes in mechanical properties of composites over 3 to 5 years may not be necessarily clinically noticeable, although their impact on the longevity of the restorations is possible [22]. Color stability is definitely one of the most important properties of an esthetic material, directly related to clinician's and patients' satisfaction. An undetected deteriorated composite may present a higher affiliation to staining molecules when polymerized. Measuring the behavior of expired composites in staining solutions is an important knowledge and might be a more reasonable, simpler and faster indication of material's deterioration.

Research on color stability of expired dental composite resins is poor. Garcia et al [23] investigated color and opacity of expired composites and found that high viscous materials presented no significant differences between expired and unexpired composites, but low viscous materials showed significant differences. For this reasons, the purpose of this study was to test the differences in color changes between expired and unexpired composite resins, using a widely used staining solutions and the effect of time on these changes. The null hypotheses tested were that no differences exist between expired and unexpired composites, among materials or immersion cycles in the staining solution.

## II. Materials And Methods

For the purpose of this study three different composite resins (expired and unexpired) were chosen (ice/SDI Ltd, Victoria, Australia - rok/ SDI Ltd, Victoria, Australia- Spectrum / Dentsply-DeTrey GmbH, Konstanz, Germany). Details of these materials are shown in Table 1. Time past their expiration date for the expired composites ranged from 15 to 146months ( ice-15months, rok-89months and spectrum-146 months) while time range before expiration date for the unexpired composites was 23 to 53 months (ice-45months, rok-53months and Spectrum-23months). Expired composites were stored in a cool place for all their life after expiration date and used only for the preparation of specimens. Unexpired composites were kept in a refrigerator at 6-8$^{o}$C temperature and were removed only for the preparation of specimens after their adjustment to room temperature.

From each material, 10 discoid specimens of 6.5 mm in diameter and 2mm in thickness were made. Specimens were finished with SiC wetordry paper 400 grit, 800grit and 1000 grit, polished on a velvet microcloth (DP-Nap/Struers A/S, Ballerup, Denmark) with 5 and 1 micron $Al_2O_3$ paste, and immersed in a water bath of 37$^{o}$C for 48 hours (baseline). After this, specimens were placed in a coffee bath, within an oven of 37±1$^{o}$C, for 2, 4, 6, 12 and 24 hours, simulating 30, 60, 90, 180 and 360 days of continuing staining action (two daily drinks of coffee equals 4minutes action per day). Coffee baths were prepared from filtered coffee (Jacobs, Douwe Egberts, Amsterdam, The Netherlands) according to manufacturer's instructions (three teaspoons in 250 ml water). After the first 48hours in a water bath (baseline) and at each subsequent coffee baths (experimental time intervals), specimens were removed from the baths, placed and agitated in a fresh water bath for 30sec, dried carefully using soft absorbent paper, measured for their color, and reentered a fresh staining solution prepared as above.

Primary (L*,a*,b*) and secondary (ΔE*, ΔL*, Δa* και Δb*) color parameters in the CIELAB system, for each material and time intervals were measured, using a portable contact colorimeter ShadeEye / Shofu (Shofu Dental Corporation, Japan ). The average of three consequent measurements in the center of each specimen, after repositioning of the device, was the final value for each specimen. Data were analyzed statistically by 2way repeated measures ANOVAs for differences between means at a=0.05, while coefficient of determination ($R^2$) for the association of storage time and color change was also estimated at a= 0.05. Statistical analyses were performed using SPSS statistical package v.15 (IBM Corporation, New York, USA).

## III. Results

Color difference values from the baseline color (ΔE*) at all predetermined cycles intervals are presented in Table 2. ΔE* values are over 2.7 for all groups, even from the 1st interval of immersion in coffee and continued to increase with the number of cycles. Changes of expired and new (unexpired) composites are best illustrated in Figures 1-2. Statistical analysis with 2way repeated measures ANOVA showed sphericity value E=0.720. Tests of within-subjects effects indicated significant differences among intervals (cycles) (F=200.01, P<0.001) but also significant interaction for cycles X materials X exp. groups (F=12.91, P<0.001). Tests of between subjects effects indicated no significant differences between expired and new materials (F=0.570, P=0.453) but a significant interaction of materials X exp. groups (F=14.92, P=0.009). Due to interactions, pair-wise multiple comparisons with Bonferroni correction between cells of the same interval, are shown in Table 2.

The above changes in ΔE* were the result of a decrease in L* and an increase in a* and b* primary color parameters (Table 3). It is evident from 95% CI of the means that differences exist between initial and final values of all new or expired composites, for all primary parameters. Differences between new and expired composites also exist but only in respect to a* and b* parameter.

Results of the analysis of the regression with cycles of all color parameters are shown in Table 4. Coefficient of determination ($R^2$) for most of the color parameters is above 0.6 with small differences between new and expired composites. Regression lines slope present some differences between new and expired materials or among color parameters. However, 95% confidence intervals do not support a significant difference (Table 4).

## IV. Discussion

The results of our study accepted the null hypothesis that there is no difference in color changes between expired and not expired composites and rejected the hypothesis of no difference among materials or cycles of their immersion in coffee used as a staining solution.

The study showed that coffee has a strong staining action on all three materials (new or expired) and a relation of its staining action with the number of immersion cycles. For all materials, ΔE* was increased in the 1st month over the clinical perceptible level of ΔE*>2.7 units and close to the clinically unaccepted level of 5.5 units [24]. The change became very intense in the 6th month, and reached the value of 52.6 ΔE* units at the end of the experimental period. Coffee's strong staining action is due to the absorption of water-soluble colorants [25], enhanced by the acidic nature of the stains, since many of coffee's ingredients are water-soluble and acid in nature substances (caffeine, citric acid, chlorogenic acid, acetaldehyde etc.). The fact that materials are heavily stained in coffee is not new and many studies have indicated this action [26, 27].

Staining was different for the different materials. Spectrum- showed the highest color difference of all materials (38.2-47.6 ΔE* units) and rok- the lowest (13.3 – 17.7 ΔE* units), not different, however, from ice-. Differences in the composition among materials explain their differences in staining. Organic matrix of "rok" and ice contains a higher amount of fillers and less volume of matrix than Spectrum. Spectrum contains a smaller amount of fillers than the rest, and its higher volume fraction of resin matrix may therefore absorb more stains in the staining solution. Resin matrix of Spectrum is also responsible for its higher staining since Bis-GMA molecule which is more prone to water absorption than TEGDMA [28], is the main part of its matrix, in contrast to the TEGDMA contained in the other composites.

New and expired materials showed no significant differences in ΔE*. Garsia et al [16] who studied the properties of expired and unexpired composites, suggested that expiration date affects degree of conversion, surface roughness and microhardness in certain materials. Oxidation of unsaturated methacrylate groups arising from the increased number of free monomer in expired composites is possibly the reason for their chromatic instability [29, 30]. However this cannot explain the finding of this study of equal staining behavior between expired and unexpired composite of different brand. The extend of expiration time does not seem to play a significant role, since Spectrum with the longest expiration time (146 months) still has the same staining behavior with the new composite. Since manufacturers are continually trying to improve their products by altering the composition of the monomers, concentration of activators and more importantly the type and quantity of fillers [31], newer materials are expected to be more stain resistant than older batches. However this is not proved in our study and for this reason it is logical to assume that structural changes in the material due to time past their expiration date do not affect its basic mechanical behavior and leaching of monomers may contribute in the remodeling of the material, given that polymerization process is not affected.

## V. Conclusion

The results of our study showed the following:

**a)** Staining solution (coffee) had a significant effect on the color stability of all materials, and color change of composites was proportional to their immersion time in the staining solution.

b) Some materials were affected more and others, but there was no difference between new and expired composites in color stability.

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**Figures and Tables**

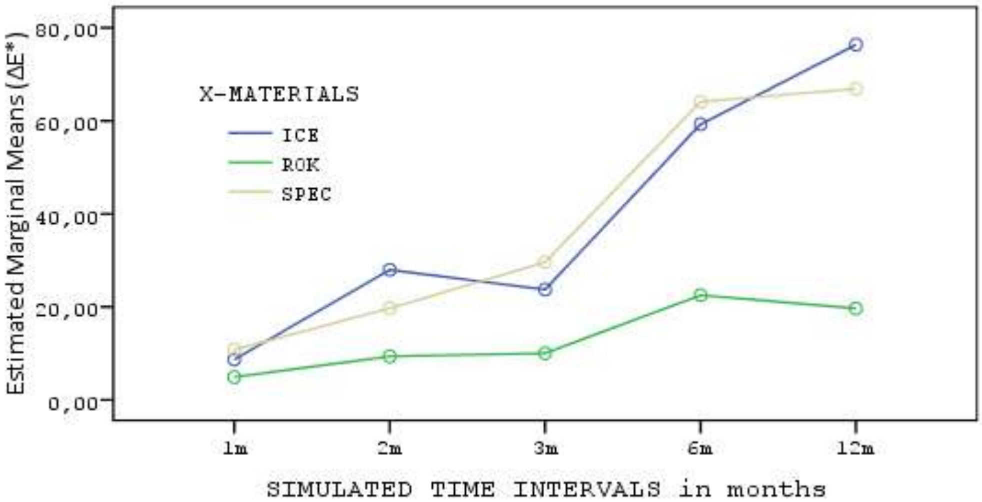


**Figure 1.** Estimated marginal means of ΔE* values for expired materials at all intervals.

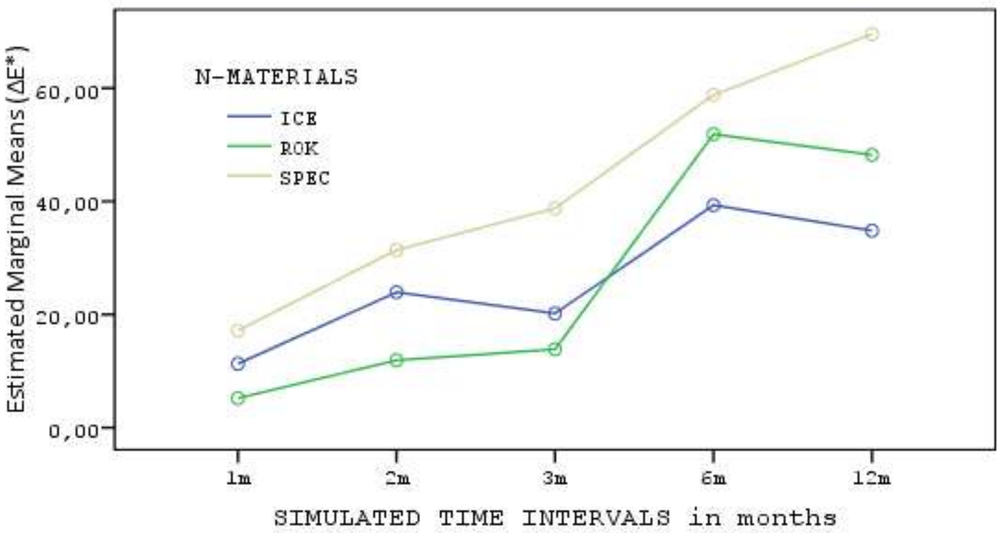


**Figure 2.** Estimated marginal means of ΔE* values for new materials at all intervals.

**Table 1.** Details of materials used in the study (m=months)

| Name / Company | Type of Material | Composition | Shade | Batch no | Expiration date | Time before expir. date | Time after expir. date |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
| Ice / SDI | Nano-hybrid | - Methacrylic ester 39%vol<br>- Fillers 61% (40nm-1.5m) | A2<br>A2 | 080806T/exp<br>130837T/new | 2013/08<br>2018/08 | --------<br>45 m | 15 m<br>-------- |
| Rok / SDI | Hybrid | - Methacrylic ester 32.3%vol<br>- Fillers 67.7% (40nm-2.5m) | A2<br>A2 | 0406179/exp<br>130472/new | 2007/06<br>2018/04 | --------<br>53 m | 89 m<br>-------- |
| Spectrum TPH3 / Dentsply DeTrey | Submicron-Hybrid | -Bis-GMA<br>-Bis-EMA<br>-TEGDMA<br>-SiO2 10-20nm<br>-BrAlBoSi <1m<br>-BrFlAlBoSi <1m | A2<br>A2 | 01754 /exp<br>0244 /new | 2002/09<br>2016/10 | --------<br>23 m | 146 m<br>-------- |
| | | | | | | | |

**Table 2.** Average color difference (ΔE*), standard deviation (±) and 95% confidence interval of marginal means of composites, at simulated months of immersion in staining solution.

| Material | 1month | 2months | 3months | 6months | 12months | Total | 95% CI |
|---|---|---|---|---|---|---|---|
| Ice-New | 11,3±4,8$^{a}$ | 23,9±8,7$^{b}$ | 20,2±4,6$^{b}$ | 39,3±8,9$^{c}$ | 34,8±6,8$^{c}$ | 25,9 | 20,9-30,9 |
| Ice-Eχpired | 8,7±5,4$^{a}$ | 27.9±5,8$^{b}$ | 23,7±6,9$^{b}$ | 59,3±12,3$^{c}$ | 76,4±21,3$^{c}$ | 27,3 | 19,5-35,2 |
| Rok-New | 5,2±3,7$^{a}$ | 11,9±4,9$^{a}$ | 13,8±4$^{a}$ | 51,9±15,1$^{b}$ | 48,2±20,8$^{b}$ | 17,7 | 10,1-25,3 |
| Rok-Eχpired | 4,9±1,3$^{a}$ | 9,4±7,3$^{ab}$ | 10±2,8$^{ab}$ | 22,5±10,4$^{ab}$ | 19,6±8,6$^{ab}$ | 13,3 | 9,4-17,1 |
| Spectrum-New | 17,2±6,7$^{a}$ | 31,4±19,8$^{b}$ | 38,8±24,2$^{b}$ | 58,8±21,4$^{c}$ | 69,6±29,4$^{c}$ | 47,6 | 37,4-57,9 |
| Spectrum-Eχpired | 10,8±9,7$^{a}$ | 19,7±8,6$^{ab}$ | 29,6±8,1$^{b}$ | 64,1±13,3$^{c}$ | 66,9±16$^{c}$ | 38,2 | 27,6-48,9 |
| Total | 9,7 | 20,7 | 22,7 | 49,3 | 52,6 | | |
| 95% CI | 7,1-12,2 | 16,0-25,4 | 17,4-27,9 | 42,0-56,6 | 42,6-62,6 | | |

Note: Results of pair-wise multiple comparisons test with Bonferroni correction within columns, are shown as superscript letters. Same letter denotes no significant differences among cells at a=0.05.

**Table 3.** Means and their 95% CI of primary color parameters (L*, a* and b*) for new and expired composites, at 0 and 12 interval of immersion.

| | **Simulated** | **L*** | | **a*** | | **b*** | |
|---|---|---|---|---|---|---|---|
| **Material** | **Month** | **Mean** | **95% CI** | **Mean** | **95% CI** | **Mean** | **95% CI** |
| Ice-New | 0 | 76,06 | 75,4-76.7 | -0.70 | -0.85-0.55 | 16.90 | 16.4-17.4 |
| | 12 | 70,27 | 69,5-71.0 | 0.93 | 0.59-1.27 | 21.99 | 21.3-22.7 |
| Ice-Expired | 0 | 76,46 | 75.9-76.9 | -0.97 | -1.04—0.90 | 12.99 | 12.7-13.3 |
| | 12 | 69,19 | 68.6-69.8 | 0.69 | 0.54-0.80 | 21.11 | 20.2-22.1 |
| Rok-New | 0 | 74,43 | 73.9-4.9 | 0.19 | 0.05-0.34 | 19.51 | 18.9-20.1 |
| | 12 | 68,35 | 67.7-69.0 | 2.11 | 1.94-2.28 | 25.43 | 24.8-26.1 |
| Rok-Expired | 0 | 77.66 | 76.8-78.5 | -0.89 | 1.00—0.78 | 19.11 | 18.3-19.9 |
| | 12 | 70.35 | 69.4-71.3 | 0.89 | 0.66-1.13 | 21.93 | 21.2-22.7 |
| Spectrum-New | 0 | 71.43 | 73.9-74.9 | -0.95 | -1.03—0.87 | 11.13 | 10.7-11.5 |
| | 12 | 68.35 | 67.7-69.0 | 0.81 | 0.69-0.94 | 18.71 | 17.6-19.8 |
| Spectru-Expired | 0 | 75.89 | 75.4-76.4 | -0.40 | 0.51—0.33 | 9.48 | .4-9.6 |
| | 12 | 69.99 | 69.5-70.5 | 1.23 | 1.10-1.37 | 17.05 | 16.3-17.8 |

**Table 4.** Coefficient of determination ($R^2$) and slope of the trend lines for L*, a*, b* and ΔE*color parameters for up to 12 months immersion of composite resins in coffee.

| **Material** | | **Slope-New** | **$R^2$-New** | **95% CI** | **Slope-Expired** | **$R^2$-Expired** | **95% CI** |
|---|---|---|---|---|---|---|---|
| Ice/SDI | L* | -0.418 | 0.763 | .635-.891 | -0.456 | 0.797 | .701-.893 |
| | a* | 0.102 | 0.559 | .384-.733 | 0.114 | 0.716 | .588-.843 |
| | b* | 0.317 | 0.480 | .289-.671 | 0.598 | 0.766 | .657-.875 |
| | ΔE* | 1.925 | 0.573 | .401-.744 | 5.927 | 0.898 | .846-.949 |
| Rok/SDI | L* | -0.538 | 0.604 | .441-.767 | -0.499 | 0.735 | .614-.855 |
| | a* | 0.151 | 0.829 | .746-.911 | 0.122 | 0.640 | .487-.793 |
| | b* | 0.515 | 0.721 | .595-.847 | 0.243 | 0.638 | .485-.791 |
| | ΔE* | 4.204 | 0.718 | .591-.845 | 1.340 | 0.638 | .485-.791 |
| Spectrum | L* | -0.503 | 0.626 | .469-.783 | -0.430 | 0.715 | .587-.843 |
| Dentsply | a* | 0.148 | 0.803 | .709-.897 | 0.125 | 0.802 | .708-.896 |
| /DeTray | b* | 0.484 | 0.627 | .470-.784 | 0.563 | 0.721 | .595-.847 |
| | ΔE* | 4.423 | 0.868 | .803-.933 | 5.190 | 0.798 | .702-.894 |