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Influence of Storage Media on Color Stability of Different Resin Composites as Determined by ΔE and ΔE_{00}

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Abstract

Objective: To evaluate the color stability of 3 different resin composites when exposed to storage in water, air or artificial saliva.

Materials and Methods: Initial color of 81 specimens was assessed by a calibrated reflectance spectrophotometer over a black as well as a white background. Specifically, 9 disc shaped specimens made out of 3 resin composite materials were kept in distilled water, air and artificial saliva (Glandosane) at 37°C. After a storage period of 4 weeks, spectrophotometric measurements were repeated and the color changes calculated by means of ΔE and ΔE00.

Results: When analysed over a white background, median ΔE values varied from 0.6 (Filtek Supreme Dentin/water) to 7.1 (Filtek Supreme Enamel/Glandosane). When analysed over a black background median ΔE values varied from 0.4 (Filtek Supreme Dentin/water) to 5.0 (Filtek Supreme Enamel/Glandosane). When analysed over a white background, median ΔE00 values varied from 0.4 (Filtek Supreme Dentin/water) to 4.6 (Filtek Supreme Enamel/Glandosane). When analysed over a black background median ΔE_{00} values varied from 0.4 (Filtek Supreme Dentin/water) to 4.6 (Filtek Supreme Enamel/Glandosane). Statistical analysis performed by means of ANOVA and Fisher's LSD post hoc tests showed differences between groups. The correlation between ΔE and ΔE00 was 99.09% and 99.65% over black and white background, respectively.

Conclusion: Within the limits of the present study, storage media significantly influenced color stability of resin composites.

Introduction

Restorative composite resins are commonly used materials for esthetic direct restorations. Knowing that these materials are not inert and undergo changes over time especially in respect to their color, it is interesting to assess the influence of different storage media regarding this parameter. Artificial saliva may be considered the gold standard that simulates oral environment. However, other alternatives may be considered such as distilled water or simply air. So far, few articles in literature have investigated the influence of storage media on color stability of resin composites [1,2] and no consensus exists on this topic.

Discoloration can be evaluated objectively with various instruments or subjectively by means of human eyes. Since instrumental measurements eliminate the subjective interpretation of visual-color comparison, spectrophotometers and colorimeters have been used to quantify color changes in dental materials [3,4]. One of the most commonly used methods to assess composites' color changes is the reflectance spectrophotometry with the CIE (Comission Internationale de l'Eclairage) L*a*b* color system which has been widely used in the literature [5,6]. The color coordinates of the CIELAB system are L*, representing the value and ranging from 0 (black) to 100 (white). a* and b* are chromaticity coordinates along the red-green (positive value indicates red, negative indicates green) and yellow-blue axes (positive value indicates yellow; negative indicates blue), respectively.

The working hypothesis of this study was therefore to test the color stability of different composite brands under the influence of different storage media by using a spectrophotometer and by determining their ΔE as well as their ΔE_{00}. The null hypotheses

were that storage media have no significant effect on color stability and that ΔE and ΔE_{00} are highly correlated.

Materials and Methods

Eighty-one disc-shaped specimens with a diameter of 10 mm and 1.0 +/- 0.05 mm thickness as verified by means of a digital caliper (digit-cal capaμsystem, S/N 8R565806, TESA, Renens, Switzerland) were made of three different composite materials (Table 1) by gently pressing a defined amount of material between two glass slides [7].

Materials	Manufacturer	Shade	Batch Nbr.	Matrix composition*	Fillers composition*
Filtek™ Supreme XTE Enamel	3M-ESPE, St.Paul, MN, USA	Enamel A2	N483889	Bis-GMA, UDMA, Bis-EMA, TEGDMA, PEGDMA	Silica, aggregated zirconia (63 vol%, 78 wt%
Filtek™ Supreme XTE Dentin	3M-ESPE, St.Paul, MN, USA	Dentin A2	N502353	Bis-GMA, UDMA, Bis-EMA, TEGDMA, PEGDMA	Silica, aggregated zirconia (63 vol%, 78 wt%
Tetric EvoCeram® Bulk Fill	Ivoclar Vivadent, Schaan, Liechtenstein	IVA	S21120	Bis-GMA, Bis-EMA, UDMA (21% of the mass)	Barium aluminium silicat glass, yttermium fluoride, mixed oxide (61 vol% and 17% isofillers, 77wt%)
* reported by the manufacturers Bis-EMA: ethoxylated bisphenol-A-glycidyldimethacrylate, Bis-GMA: bisphenol-A-glycidyldimethacrylate, PEGDMA: poly(ethylene glycol) dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate					

Table 1: List of materials evaluated.

The composite resins specimens were light cured for 20 s using a 1100mW/cm2 LED light curing unit (Bluephase, S/N 1523817, Ivoclar Vivadent, Schaan, Liechtenstein). Specimens' color measurements L*a*b* were assessed by quantitative numerical measurement approach, using a calibrated reflectance spectrophotometer (Spectro Shade Handy Dental Type 713000, S/N HDL2891, Medical High Technologies, Arbizzano di Negar, Italy).

This device has a build-in aiming routine that enables a reproducible positioning perpendicular to the sample's surface to ensure equal measurement conditions for all the evaluated specimens. Specifically, this device is equipped with a D65 light source (6500°K) that is converted into monochromatic light by means of a grating. This light is splinted in order to have each specimen illuminated at the same time from two sides at 45° angle. The reflected light is directed at 0° on both the system's two detector areas (both 18 x 13 mm2). One detector is a color CCD chip that generates the color video image. The other, black and white CCD detector records the spectrophotometric data. Polarization filters allow eliminating surface gloss. The data is stored in a proprietary image file format which is used to create detailed CIE L*a*b* data.

In this experiment CIE L*a*b* measurements of each specimen were performed over both white (WB) (L*=92.6, a*=-1.2, b*=2.9) and black (BB) (L*=1.6mm, a*= 1.2, b*=-1.0) background made of plasticized paper [8].

After recording the initial spectrophotometric measurement of the samples (t_0), they were randomly divided in 3 groups (27 samples per group) and stored as follows: dry storage (Group 1), artificial saliva (Glandosane) (Group 2) and distilled water (H_2O) (Group 3) (Table 2). The specimens were left for a period of 4 weeks at constant temperature of 37°C in an incubator (INP-500, Memmert GmbH & Co.KG, Schwabach, Germany) in the absence of light. Glandosane and H_2O were changed every 7th day to avoid bacteria or yeast growth [9].

Actives ingredients	potassium chloride, sodium chloride, magnesium chloride, calcium chloride, potassium diphosphate, sodium carboxymethylcellulosa, sorbitol.
Excipients: Conserv	sorbic acid (E 200), sodium benzoate (E 211), Aqua ad solutionem et Propellentia ad Aerosolum pro 1 g

Table 2: Composition of Glandosane (Helvepharm AG, Frauenfeld, Switzerland).

After storage, spectrophotometric measurements were performed (t_4) and ΔE (t_0-t_4) and ΔE_{00} (t_0-t_4) were calculated according to the classic formulas:

$$\Delta E^* = \sqrt{(L_1 - L_2)^2 + (a_1 - a_2)^2 + (b_1 - b_2)^2}$$

$$\Delta E^*_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \frac{\Delta C'}{k_C S_C} \frac{\Delta H'}{k_H S_H}}$$

For ΔE and ΔE_{00} two-way ANOVA tests were run to check on significant color differences. Fisher's LSD post hoc test was than run to check on differences between groups.

Results

The ΔE and ΔE_{00} values between t_0 and t_4 varied depending on the composite brand and the storage media (Figure 1,2). Filtek Supreme Dentin in water showed the lowest median color changes (ΔE = 0.6 (WB) / 0.4 (BB); ΔE_{00} = 0.4 (WB) / 0.4 (BB)) while Filtek Supreme Enamel in Glandosane showed the highest median color changes (ΔE = 7.1 (WB) / 5.0 (BB); ΔE_{00} = 4.6 (WB) / 4.6 (BB)).

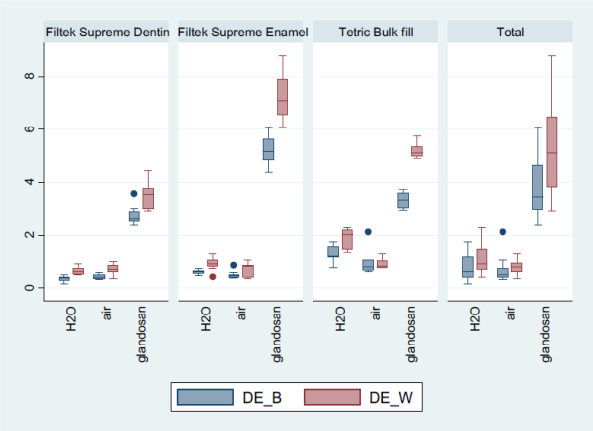

Figure 1: ΔE values box-plot by composites.

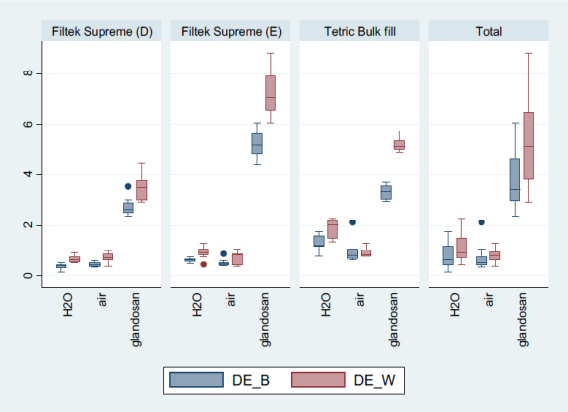

Figure 2: ΔE_{00} box-plot by composites.

Two-way ANOVA test confirmed that all storage media played a significant role (p-values < 0.001) in color change. The interaction between composite and storage media was significant too. In particular, artificial saliva (Glandosane) was by far the liquid that generated the highest color variations (Fisher LSD post-hoc tests are illustrated in Tables 3 and 4 for ΔE and ΔE_{00}). No significant difference was found between air and H_2O for all composites except for Tetric Bulk Fill due to a higher color change in H_2O especially with a white background. The correlation coefficient for ΔE and ΔE_{00} was 0.9909 and 0.9965 over black and white background, respectively (Figure 3,4). Both ΔE and ΔE_{00} led to the same statistical conclusions.

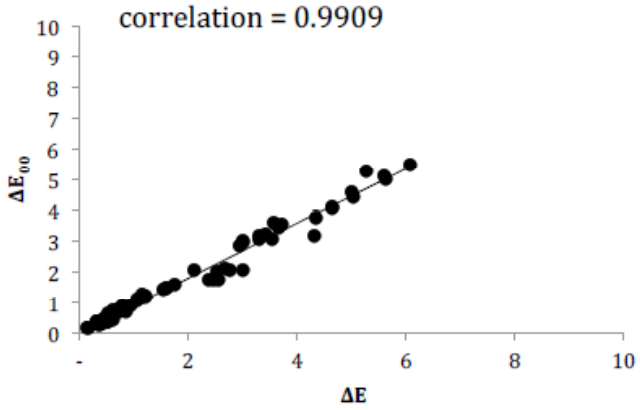

Figure 3: ΔE vs ΔE_{00} (Black background).

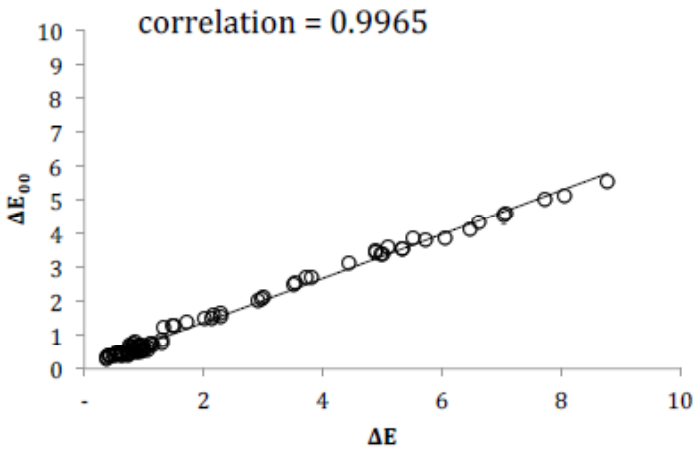

Figure 4: ΔE vs ΔE_{00} (White background).

White Background			Black Background		
Filtek Supreme Dentin			Filtek Supreme Dentin		
	AIR	H_20		AIR	H_20
H_20	0.999	-	H_20	0.524	-
Glandosane	<0.001	<0.001	Glandos-ane	<0.001	<0.001

Filtek Supreme Enamel			Filtek Supreme Enamel		
	AIR	H_20		AIR	H_20
H_20	0.963	-	H_20	0.456	-
Glandosane	<0.001	<0.001	Glandos-ane	<0.001	<0.001
Tetric Bulk Filled			Tetric Bulk Filled		
	AIR	H_20		AIR	H_20
H_20	<0.001	-	H_20	0.018	-
Glandosane	<0.001	<0.001	Glandos-ane	<0.001	<0.001

Table 3: ΔE Fisher's LSD post-hoc test p-values.

White Background			Black Background		
Filtek Supreme Dentin			Filtek Supreme Dentin		
	AIR	H_20		AIR	H_20
H_20	0.191	-	H_20	0.254	-
Glandosane	<0.001	<0.001	Glandos-ane	<0.001	<0.001
Filtek Supreme Enamel			Filtek Supreme Enamel		
	AIR	H_20		AIR	H_20
H_20	0.811	-	H_20	0.716	-
Glandosane	<0.001	<0.001	Glandos-ane	<0.001	<0.001
Tetric Bulk Filled			Tetric Bulk Filled		
	AIR	H_20		AIR	H_20
H_20	<0.001	-	H_20	0.004	-
Glandosane	<0.001	<0.001	Glandos-ane	<0.001	<0.001

Table 4: ΔE_{00} Fisher's LSD post-hoc test p-values.

Discussion

The evaluation of color differences can be performed by means of two techniques: the subjective visual method or the objective instrumental method. Instruments such as colorimeters or spectrophotometers have been developed to detect small differences that might be imperceptible to the human eye [10]. In this study, a spectrophotometer was used to avoid subjective bias [7,11].

Nowadays several color notation systems are available but the most used one for dental restorative materials have been in the last decades the CIELAB [12] that is why we decided to adopt this system in our experiment. Due to the imperfections of the CIELAB system, several advanced color-difference formulae were developed. The most recent one is the CIE 2000 or CIEDE2000, officially recommended as the new CIE color difference equation for dental purposes [13]. The new CIEDE2000 formula includes three weighting functions: lightness (new formula), chroma (adopted from CIE94) and hue (new formula), with the arithmetic mean of a pair of specimens used for calculating all weighting functions instead of the geometric mean [14]. In order to see if significant differences could be detected depending on the CIE system both delta E (ΔE and ΔE_{00}) were calculated and statistically analyzed, giving the same conclusions. This was witnessed by the high correlation coefficient obtained in this study for the two delta E both on black and white background. These findings are in accordance with previous studies [15,16].

Concerning the preparation method, we decided not to polish our samples. This was done in order to standardize and to mimic the worst-case clinical situation where a composite is polymerized through a mylar strip without any finishing/polishing as might be the case in Class III or Class IV restorations. It was described in literature that covering the composite surface with a celluloid strip [17] leads to a smooth uppermost resin-rich layer with less filler content, thus more prone to coloration. The relatively high resin content and its vulnerability to water uptake have been indicated as possible reasons for higher discoloration [18,19] as water absorbed by the polymer matrix may cause matrix-filler debonding or even hydrolytic degradation of the filler itself [20]. Polishing eliminates the resin-rich surface layer [21], but it can also introduce variables that are difficult to control as shown in a recent study where surface roughness of esthetic restorative materials influenced the color coordinates [22]. To avoid bias through polishing and to simulate the most extreme but clinically relevant situation, no polishing of the surface was performed. The decision of 20 seconds polymerization was made in order to provide the same energy to all the specimens.

Concerning color stability of resin composites, literature suggests that they are not chemically inert, interacting with the storage media [10,23]. Furthermore, even storage time has been shown to have a significant influence on color stability [1]. Commonly in composite staining studies, artificial saliva, water or dry storage are the negative control [1,3,5,7,16,23], that is why we decided to experience if any statistical difference could be found in between the three-storage media. When exposed to their natural environment in the oral cavity, composite resin restorations are in constant contact with moisture. The present study tested the relationship between storage medium and the composite itself. The same behavior pattern was found independently of the composite used or the background. All composite materials of the dry storage group experienced a slight color change, likely due to post-polymerization of the material. On the other hand, artificial saliva is the medium inducing the higher color change.

The pH of Glandosane (pH 5.75) is in the range of normal saliva pH but in the lower portion. This choice has been made in order to mimic the clinical conditions of carious patients who usually have a lower saliva pH [24, 25]. Furthermore, Batra et al. [26] have reported that acidic media are responsible of surface degradation and thus induce ΔE changes.

Furthermore, artificial saliva contains conservators that might induce a difference comparing with distilled water. Human saliva contains mucins and enzymes. Several studies have shown that Cholesterol Esterase (CE) and Pseudocholinesterase (PCE) could degrade composite resins [27-29]. Under this light, it might be expected that results of color-stability tests for resin composites performed in vitro with artificial saliva could show some discrepancy with respect to the in vivo situation where values could be worsened. However, to perform in vitro studies, artificial saliva might be used as control because it closer simulates oral environment and causes statistical color variation when compared to distilled water and air.

It can be of interest even to analyze the amount of color change and to check if it is clinically relevant or just noticeable only by mechanical devices such as spectrophotometer or colorimeters.

On this purpose Ghinea [30] and colleagues attempted to determine the magnitude of color change that can be considered acceptable for 50% of observers (50:50%acceptability threshold) for color difference formulas. The authors used TSK Fuzzy Approximation for determining acceptability thresholds and reported 2.23 for the CIEDE2000 formula as the key value. In our study these values are obtained by all resin composite tested only when put in contact with Glandosane. On the other hand, all the other storage media induced changes which were always below the so called "acceptable level".

Concerning the behavior of the different materials tested several general considerations related to their compositions can be done. Tetric Evo Ceram Bulk Fill when stored in water, showed the highest color change. A possible explanation could be due to a higher conversion rate than Filtek Supreme. Tetric Bulk fill, in fact, contains not only camphorquinone but also even Ivocerin that increases conversion rate. Moreover, his higher degree of translucency could influence positively the light transmission and further enhance the degree of polymerization.

In Filtek Supreme, camphorquinone is used as the main photoinitiator. Its color is yellow and it becomes colorless when completely polymerized. On the other hand, aging process of resin composite cause a yellow shift of the material. Therefore, a non-complete polymerization of Filtek could have act as bias of the final result obtained. Furthermore, the slightly higher translucency of Filtek Enamel (30%) compared to Filtek Dentin (17%) could have allowed a better conversion rate of Filtek Supreme Enamel causing higher color change when compared with the results obtain with Filtek Supreme Dentin. This fact could be the explanation of the higher color difference obtained for Filtek supreme Enamel if compared to the dentin shade of the same manufacturer.

Further interpretations of the obtained results are not possible because manufacturers do not reveal the exact composition of their resin composite materials. Further study made out of resins where all components and their concentrations are known are suitable in order to better understand resin composite behaviors.

Conclusion

Within the limits of the present study, storage media significantly influenced color stability of the composite resins tested. ΔE and ΔE_{00} were highly correlated and obviously led to the same statistical conclusions. The null hypothesis that different conservation media have no effect on the color stability of composite resins at different times was rejected. More studies are needed to confirm these findings.

Declaration of Interest: none

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