Effect of different beverages on the color stability and degree of conversion of nano and microhybrid composites

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This study sought to evaluate the effects of different staining solutions on the colour stability of nanocomposites compared with micro hybrid resin and to evaluate the degree of conversion of these two materials. Two different shades of two different composites were cured in polytetrafluoroethylene disk rings. Coffee, tea and cola drinks were used as staining solutions, and distilled water was used as a control. Data were statistically analysed using a paired T-test with a significance level of 5%. Nano composite showed the highest degree of conversion (DC) values based on calculation of the bonded and free carbonyl peak intensities in the spectrum. The colour analysis showed that nanohybrid had the highest ΔE values when exposed to coffee solutions; they showed less color stability despite having a higher degree of conversion. Nano resin composite showed significantly higher discoloration than microhybrid composite.

Keywords: Color change, Degree of conversion, FTIR spectroscopy, Microhybrid composites, Nanocomposites

INTRODUCTION

Photo-activated resin composites are commonly used as restorative materials in dentistry for both anterior and posterior restorations and can be bonded to dentin or enamel tissues. The degree and depth of conversion of composite resins that cure under visible light depends on the components of the material (organic matrix, type and volume of the organic filler and the size of filler particles) and on the intensity and duration of exposure to blue light. The degree of conversion (DC) is a measure of the carbon double bonds converted into single bonds¹.

Several methods have been reported for studying the DC of composite resins. These include evaluation of hardness², optical microscopy³ and vibrational methods, *i.e.* infrared spectroscopy (Fourier Transform Infra Red)⁴ and Raman spectroscopy (Raman)⁵. Adequate polymerisation is necessary to achieve optimal mechanical and optical properties⁶.

Discoloration is a significant aesthetic problem for direct tooth-colored restorations. The shades of both chemical- and light-cured composite resins have been reported to change over time due to extrinsic or intrinsic discoloration⁷. Changes in color can be attributed to intrinsic discoloration due to the restoration and extrinsic discoloration due to accumulation of plaque and stains⁸.

In the light-cured composite resins that are currently used, changes in color are usually related to extrinsic factors and depend on several factors, such as the staining agent⁹, the surface roughness¹⁰, contact

time with or immersion in coloring environments¹¹ and the type of composite resin used⁷. Previous studies of color stability have shown that beverages, such as coffee, tea, red wine, and cola, as well as mouth rinses, can cause staining of chemical and light-cured composite resins to varying degrees. The staining potential of these beverages and solutions varies according to their composition and properties¹².

Nanotechnology-based composites are now being used to provide a higher DC compared to Microhybrid resin composites. Recently, nanofill composite materials have been developed. These materials use sub-micrometre particles (nanofillers) to further enhance the optical and physical properties of the resins¹³.

The aim of this study was to evaluate the effect of different staining solutions on the color stability of Nanocomposites compared with Microhybrid resin and to evaluate their degree of conversion. Two null hypotheses were evaluated: (1) Nano composites with a higher degree of conversion have better color stability compared to Microhybrid composites; and (2) Nanohybrid resin composites (Grandio Nano) are more color stable than Microhybrid (Arabesk Top).

MATERIALS AND METHODS

Materials

Two types of light-cured resin composite restorative materials and two different shades of each (A₂ and A_{3.5}) were used in this study (Table 1). The two materials investigated were a microhybrid composite (Arabesk Top, Lot No. 0925121, Voco, Germany; AT) and nanocomposite (Grandio Nano, Lot No.1010431, Voco, Germany; GN).

Color figures can be viewed in the online issue, which is available at J-STAGE.

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Table 1 Types of restorative materials used in the study

Materials	Shade	Description	Composition	Monomer
Grandio Nano	A2, A3.5	Universal NanoHybrid	87 wt% inorganic filler 71.4 vol % Nano-hybrid composite SiO ₂ (20–60 nm) Glass ceramic	Bis-GMA, UDMA, TEGDMA Initiator: Camphoroquinone
Arabesk top	A2, A3.5	Glass ceramic, Microhybrid resin	77 wt% (56 Vol% microfiller ca. 0.05 µm and very small particles with mean particle size 0.7 µm Bariumaluminumsilicate glass Lithiumaluminumsilicate glass ceramic Highly dispersed silicon dioxide	Bis-GMA, UDMA, TEGDMA

Bis-GMA: bisphenol-A-glycidyl methacrylate, UDMA: urethane dimethacrylate, TEGDMA: triethyleneglycol dimethacrylate

Degree of conversion

A sample of the uncured composite was mixed with potassium bromide (KBr) powder to make a disc shaped specimen. Absorbance peaks were collected before curing from 10 scans at a resolution of 4 cm⁻¹, over a range of 4,000–300 cm⁻¹, acquired with a Fourier Transform Infra Red Spectrophotometer (Shimadzu, Kyoto, Japan) operating in absorbance mode. The microhybrid and nanohybrid composites were light cured for 40 s with a light emitting diode (LED) in a polytetrafluoroethylene mould (Diameter 4 mm, thickness 3 mm) covered with a mylar strip on one side and glass slide on other. They were then pulverised into fine powder by a mortar and pestle immediately after curing. The time delay between light polymerization and FTIR measurement was 2–3 min. Forty micrograms of powder were mixed with 4 mg of potassium bromide powder, similar disc-shapes specimens were placed on a support and analysed to record the absorbance peaks. The amounts of double carbon bonds which are converted in single bonds provide the DC%. The percentage of the unreacted carbon-carbon double bonds (%C=C) was determined from the ratio of absorbance intensities of aliphatic C=C stretching vibrations at 1,637 cm⁻¹ and aromatic C-C stretching vibrations at 1,608 cm⁻¹ to an internal standard before and after curing of the specimen. As the absorption of aromatic carbon-carbon stretching band remains static during curing it serves as an internal standard. The two-frequency technique is used for calculation of the monomer conversion. The degree of conversion was calculated according to equation (1) used by Lohbauer *et al.* (2005)¹⁴.

$$\text{Degree of conversion (\%)} = 100 \times \left\{ 1 - \frac{A_m(\text{C-C})}{A_p(\text{C=C})} \right\} \quad \text{Eq. (1)}$$

Where $A_m(\text{C-C})$ (1,608 cm⁻¹) are aromatic stretching vibrations before curing, and $A_p(\text{C-C})$ are aromatic vibrations after curing, $A_p(\text{C=C})$ are similar aliphatic stretching vibrations for cured material and $A_m(\text{C=C})$ (1,637 cm⁻¹) are stretching vibrations for aliphatic carbon-carbon double bonds for uncured materials.

Color stability

Twenty disk shaped specimens of each shade (Diameter

of 11±0.1 mm, thickness of 3±0.1 mm) were prepared in accordance with the manufacturer's instructions and by using a polytetrafluoroethylene ring. These specimens were then cured with the Elipar free light curing unit (3M ESPE, Dental Products, Germany) for the required number of seconds (40 s). The specimens were stored in a dark environment at 37°C±1°C in an incubator before testing for 24 h. No polishing procedure was performed. To prepare the coffee solution, 15 g of coffee powder (Nescafe Classic, Nestle Hellas, Athens, Greece) was poured into 500 mL of boiling distilled water. After 10 min of stirring, the solution was filtered through filter paper. The tea solution was prepared by immersing 5 pre-packaged doses of tea powder (Golden Sun, Eswaran Brothers, Colombo, Sri Lanka) into 500 mL of boiling distilled water for 10 min. Fresh solutions were made after each sampling. The two staining solutions were stored in separate vials and maintained at the temperature of the oral cavity of (37°C±1°C). Additionally, a new can of Cola Drink (Pepsi Co, Saudi Arabia) was used. Once the samples were placed in the tubes, the lids were tightly sealed.

The color measurements of each specimen were made before immersion (baseline) using a spectrophotometer (Color-Eye 7000A, Gretag Macbeth, New Windsor, NY, USA). The purpose of this instrument is to detect the amount of light illuminated by an object and the amount of light that is reflected from it. Calibration of the spectrophotometer was done using a black and white ceramic tile provided by the manufacturer. After conducting baseline measurements, 5 randomly selected specimens of each shade ($A_{3.5}$, A_2) were submerged in one of the three staining solutions or distilled water (control solution) at 37°C±1°C for 90 days. Color measurements were recorded again after 1, 2, 7, 15, 30, 60 and 90 days. The specimens were gently washed with distilled water for 5 min and then dried with tissue paper before each measurement was conducted. The color change of each specimen was then recorded. The CIE LAB (Commission Internationale de l'éclairage) color system was used for recording the values. This system is comprised of an approximately uniform color space with color coordinates for white-black (L^*), redness-greenness (a^*) and yellowness-blueness (b^*). The L^* value refers to the lightness of an object and its value ranges from zero

(black) to 100 (white). The a^* and b^* value are a measure of redness or greenness and yellowness or blueness of an object. The a^* and b^* chromaticity co-ordinates reach zero for neutral colors and increase in magnitude for saturated or intense colors. These L^* , a^* and b^* values were recorded at least 3 times by the spectrophotometer for each experimental time period. The mean values of L^* , a^* and b^* were calculated where L_0 , a_0 , b_0 were the values of the stained specimen before bleaching and L_1 , a_1 , b_1 were recorded after bleaching. The color difference (ΔE^*) was calculated from the mean ΔL^* , Δa^* and Δb^* values using the following formula:

$$\Delta E = \{(L^*_1 - L^*_0)^2 + (a^*_1 - a^*_0)^2 + (b^*_1 - b^*_0)^2\}^{1/2} \quad \text{Eq. (2)}$$

For each shade of the Grandio Nanohybrid and Arabesk Microhybrid materials, the ΔE values were subjected to statistical analysis using a Paired t -test. The SPSS Version 18 statistical software for Windows was used for the analysis. The interpretation of these results was based on the fact that ΔE values greater than 3.3 are considered to produce a visually or clinically unacceptable discoloration of the specimen. Origin Pro 7.5 (Microsal Software, Northampton, US) was used to make regression curves.

RESULTS

Degree of conversion

Figure 1 shows representative spectra before and after polymerization of microhybrid resin composites in which aliphatic C=C is seen at $1,637.56 \text{ cm}^{-1}$ and C=C aromatic stretching vibrations are seen at $1,608 \text{ cm}^{-1}$, since the absorption of the aromatic carbon-carbon stretching bands remain static during polymerization it serves as an internal standard. The black line in Fig. 1 shows the spectrum before polymerization and red line marks the spectrum after polymerization for shade A_{3.5}. The aliphatic peak tends to decrease due to absorption or consumption of C=C bonds. The %DC of Nano Hybrid composites have been calculated to be 70%; this is of the same order of magnitude as similar resins that have been described in the literature. The microhybrid composite AT showed a conversion rate of around 56%. Hence, the nanocomposite exhibited a higher monomer-polymer conversion rate compared with the microhybrid resin composite. The results of the mean (SD) values for degree of conversion are listed in Table 3.

Color stability

Table 2 gives the means and standard deviations (SD) of the color change values (ΔE^*) of GB and AT (A_{3.5} and A₂). The most severe discoloration was apparent with coffee and tea solutions¹⁵. The discoloration in these cases was significant when compared to the neutral samples stored in distilled water^{16,17}. Figures 2 and 3 show the scatter plots and curves adjusted for regression equations between ΔE and immersion time for each material, GN and AT. Each curve is well fitted a regression curve of $\Delta E = at^b$, where a and b are constant and t is time. Though

Fig. 1 FTIR spectra before and after light polymerization showing typical C-C and C=C peaks.

it is difficult to explain a physical meaning of “ b ” value, change in ΔE with time are experimentally defined by those equations. The regression curves have shown a direct relationship of ΔE with time. The samples stored in coffee displayed an increase in the value of ΔE value over time. The distilled water and cola caused the least discoloration.

With the GN (A_{3.5} and A₂), the highest ΔE value was recorded for specimens exposed to coffee solutions ($\Delta E=9.863$), while the least affected were specimens exposed to cola ($\Delta E=3.49$). The specimens immersed in distilled water (control) showed the least color change throughout the experiment period. Unacceptable color change was noted in all samples immersed in coffee or tea for 2–90 days. Distilled water seemed to produce significant discoloration, but it was within the acceptable range ($\Delta E < 3.3$).

After 90 days of immersion, AT showed less color change than GN in all solutions. This difference was statistically significant.

DISCUSSION

The physical, mechanical and biological properties of a photo-activated composite restorative resin are strongly influenced by the DC achieved by the organic matrix^{18,19}. Ideally, a resin material would have all of its monomer converted to polymer during polymerisation^{14,20}. In the present study, the DC evaluation using FTIR spectroscopy showed that the DC values of the microhybrid composite resin were (56%) and nanocomposite resin were (70%).

A high degree of polymer conversion and efficient polymerization can affect color stability because residual monomer left in the polymeric chain can lead to the formation of colorimetric degradation products that facilitate the penetration of solvents from the oral environment into the polymeric network. This phenomenon promotes hydrolytic degradation of the newly formed chain¹⁸. However, this was not observed in the present study; therefore, the first hypothesis can

Table 2 Mean (Standard Deviation) of the color change value ΔE

Resin Composite	Shade	Immersion Period	Staining Solution			
			Distilled water	Coffee	Tea	Pepsi Cola
Grandio NanoHybrid	A3.5	1 day	0.546(0.01) ^a	2.134(0.51) ^a	1.812(0.10) ^a	0.938(0.04) ^a
		2 days	0.808(0.05)	3.802(0.22) [*]	2.776(0.30)	1.11(0.11)
		7 days	1.144(0.09) ^b	5.936(0.32) ^{*b}	3.637(0.43) ^{*b}	1.445(0.03) ^b
		15 days	1.383(0.12)	6.661(0.30)	4.471(0.42) [*]	2.095(0.23)
		30 days	1.638(0.16)	6.475(0.21) [*]	6.354(0.41) [*]	2.436(0.51)
		60 days	2.27(0.22)	7.748(0.70) [*]	7.636(0.20) [*]	2.89(0.31)
		90 days	3.352(0.43) ^{*c}	8.848(1.17) ^{*c}	8.676(1.01) ^{*c}	3.697(0.40) ^{*c}
Arabesk light-cured MicroHybrid	A 3.5	1 day	0.816(0.05) ^a	3.114(0.12) ^a	2.449(0.31) ^a	1.095(0.21) ^a
		2 days	1.243(0.21)	3.383(0.46) [*]	3.012(0.51)	1.538(0.10)
		7 days	1.849(0.04) ^b	4.109(0.25) ^{*b}	3.743(0.13) ^{*b}	2.019(0.32) ^b
		15 days	2.149(0.31)	5.019(0.26) [*]	4.646(0.20) [*]	2.669(0.22)
		30 days	2.544(0.12)	5.668(0.81) [*]	4.854(1.04) [*]	3.067(0.52)
		60 days	3.082(0.31)	5.931(0.32) [*]	5.206(0.26) [*]	3.211(0.51)
		90 days	3.659(0.51) [*]	6.78(0.51) ^{*c}	5.659(0.81) ^{*c}	3.632(0.40) [*]
Grandio NanoHybrid	A2	1 day	0.354(0.01) ^a	3.232(0.13) ^a	2.443(0.70) ^a	0.973(0.02) ^a
		2 days	0.754(0.05)	3.518(0.31) [*]	3.866(0.12) [*]	1.072(0.42)
		7 days	1.525(0.10) ^b	6.11(0.41) ^{*b}	4.604(0.50) ^{*b}	1.408(0.10) ^b
		15 days	2.052(0.32)	6.96(1.04) [*]	5.246(0.23) [*]	2.085(0.05)
		30 days	2.367(0.13)	7.546(0.23) [*]	6.732(0.41) [*]	2.685(0.70)
		60 days	2.701(0.51)	8.97(0.26) [*]	8.608(0.63) [*]	3.171(0.31)
		90 days	3.168(0.43) ^c	9.863(2.12) ^{*c}	9.404(1.41) ^{*c}	3.498(0.40) ^{*c}
Arabesk light-cured MicroHybrid	A 2	1 day	0.925(0.12) ^a	4.163(0.20) [*]	2.734(0.13) ^a	1.548(0.10) ^a
		2 days	1.229(0.09)	5.389(0.35) [*]	3.21(0.23)	1.937(0.05)
		7 days	2.042(0.23) ^b	5.865(0.81) [*]	3.993(0.43) ^{*b}	2.397(0.70) ^b
		15 days	2.528(0.70)	6.748(0.20) [*]	4.387(1.12) [*]	2.721(0.11)
		30 days	2.957(0.11)	7.885(1.04) [*]	5.405(0.32) [*]	3.204(0.120)
		60 days	3.122(0.51)	8.851(0.41) [*]	6.086(0.52) [*]	3.804(0.43) [*]
		90 days	3.593(0.12) [*]	9.273(2.12) ^{*c}	6.282(0.35) ^{*c}	4.204(1.04) [*]

*Indicates clinically unacceptable values ($\Delta E \geq 3.3$).

Similar superscripted letters in the same column indicate statistical significance ($p < 0.05$).

Table 3 Percent Mean (SD) of the degree of conversion values

Resin Composite	A _{3.5}	A ₂
Grandio Nano	69.6 (1.81)	69 (2.92)
Arabesk top	55.8 (1.92)	56.7 (2.91)

be rejected. An increase in degree of conversion does not improve the optical properties of the material, as the Nano hybrid composite resin showed more staining than the microhybrid²¹. For clinically successful restorative dentistry a minimum DC has still not been precisely established²². According to Kournetas, the degradation of the matrix results in hydrolysis of the bond with the silane coupling agent. Consequently it causes detachment of the filler, which accounts for most of the color change²³. The increase in color change of resin composites stored in distilled water with time may occur due to presence

of water which tends to soften the polymer by causing swelling of the network and reducing the frictional forces between polymer chain^{20,21}. In this study the use of the glass plate provided smoother surface for all materials tested. In agreement with our study it was found that Mylar strip formed the smoothest surface²³. In addition to the finishing and polishing procedures may result in different surface textural changes and sub surface defects. In general the regression curves for GN and AT yielded a strong correlation between ΔE and time. This correlation could be due to the difference in the filler size and load, resin type and photoinitiator present in the material. The coffee samples of AT for shade A_{3.5} showed the highest values for color change with time²⁴.

Villalta *et al.*²⁴ showed that a nanocomposite (Filtek Supreme) underwent a more significant color change than a microhybrid composite (Esthet-X) as a result of staining with coffee or red wine solutions. Yazici *et al.* also reported that the nanocomposites had a higher color change than the microhybrid composites after staining¹⁵. These results are in agreement with our study, which

Fig. 2 Regression curve analysis of color change (ΔE) vs time (days) for Grandio (A_{3.5}, A₂).

Fig. 3 Regression curve analysis of color change (ΔE) vs time (days) for Arabesk top (A_{3.5}, A₂).

found that the microhybrid composite resin (AT), which had a greater inorganic volume (77.4%), showed the lowest ΔE values for all tested solutions when compared to the nano composite resin (GN), but this difference was not statistically significant (Table 2).

AT is a glass ceramic microhybrid composite with almost the same matrix formulations as GN with different filler size and distribution. It has around 77 wt% microfiller and very fine particles that are approximately 0.05 μm in size. This glass ceramic structure should provide optimal stability^{25,26}. Therefore, the staining susceptibility of AT may be related to the type of filler, the type of resin and the type of staining agent²⁷. This result is in agreement with other studies that have concluded that nanocomposites undergo a greater color change than microhybrid composites after staining. It has been reported that increased particle size results in less color change due to a decrease in the proportion of organic filler matrix²⁸. Therefore, the second hypothesis is also rejected, as the Microhybrid resin composite showed more color stability than Nanohybrid resin composite which is in agreement with Ertas *et al.*²⁸ and demonstrated that nanohybrids did not exhibit superior stain resistance against these beverages.

Studies on color stability have reported that values of ΔE higher than 3.3 can be considered clinically unacceptable²⁸. The different composite resins tested in this study showed color change after 1 day of immersion in coffee and tea solution, but these changes were considered clinically acceptable ($1 < \Delta E < 3.3$). In this study, the color changes of GN exceeded a ΔE value of 3.5 and 3.8 for shades A_{3.5} and A₂, respectively after 2 days of immersion in coffee solution. AT specimens demonstrated unacceptable color change after 1 day of immersion in coffee and after 7 days of immersion in tea.

Among the possible reasons for the increased staining caused by coffee and tea are the susceptibility of the

polymer to water sorption and the hydrophilic nature of its matrix^{27,29,30}. This water sorption is important because if the composite resin can absorb water, it is also capable of absorbing other fluids, such as coffee or tea, which would result in discoloration of the resin⁸. Imazato *et al.*³¹ studied the effect of degree of conversion on the internal discoloration of composites and concluded that the correlation of these two factors was significant. They found out that both the unreacted C=C and amines are linked with composite discoloration^{19,20,22,29,30}. Another factor responsible was the type of monomers used, resins having higher concentration of triethyleneglycol dimethacrylate (TEGDMA) were more prone to discolor than composites containing higher concentrations of urethane dimethacrylate (UDMA)³⁰⁻³³. These monomers make the resin matrix hydrophilic in nature which enhances water and other fluid sorption, causing color change within the material^{9,30,34}.

Ruyter *et al.*²¹ have reported that tea and coffee have yellow colorants, which have different polarities. The constituents of tea that have high polarity are released first followed by those with lower polarity in coffee. The discoloration caused by tea is due to the adsorption of these highly polar colorants onto the surface of the material, and the discoloration can be reversed by using a tooth brush. Um and Ruyter revealed that discoloration by coffee was due to both adsorption and absorption of colorants. This absorption and penetration of colorants into the organic phase of the materials were probably due to compatibility of the organic phase with the yellow colorants of the coffee¹⁵.

In the present study, color change values for all resin composite restorative materials in cola were less than 3.3 after 60 days. This may be due to the absence of yellow colorant, which is abundant in coffee and tea⁹. The findings of Al-Kheraif and Ertas *et al.* also lent support to the present study in that coffee and tea produced more discoloration than cola¹⁴.

Further studies should be done to better understand the relationship between the physicochemical properties of composites and the degree of conversion.

CONCLUSIONS

From the FTIR spectroscopy data, we determined that the degree of conversion for Nano composites was higher than the values obtained for Microhybrid resin composites (70% vs 56%). The discoloration of GN was significantly higher after 90 days of immersion in coffee and tea ($\Delta E > 3.3$), and the discoloration was considered clinically acceptable after immersion in cola ($1 < \Delta E < 3.3$) for periods shorter than 90 days. No correlation was found between the degree of conversion and color stability.

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