In Situ Investigation on Color change of Resin Composite Restoratives Cured by Two Different Curing Units

Dalia Mostafa Abu ElMagd, PhD[1,2;] Ahmed Adel AbdelAziz, MSc[3]; Ola Ibrahim Fahmy, PhD[4]; Hebat-Allah Mohamed Taher, PhD[5]

[1]Professor, Restorative Department,King Abdulaziz University,College of Dentistry,Jedda,Saudi Arabia
[2] Professor, Restorative Department, Faculty of Oral and Dental Medicine, Cairo University, Egypt.
[3] Technical Specialist, 3M Unitek (Health Care Division), Egypt.
[4] Professor and head, Restorative department, Faculty of Oral and Dental Medicine, Misr International University (MIU), Egypt.
5 Lecturer, Restorative Department, Faculty of Oral and Dental Medicine, Cairo University, Egypt.
Corresponding Author: daliamagd@gmail.com

Abstract: Purpose: To investigate and compare in situ the effect of halogen and high-performance light emitting diode light curing unit on the colour change of resin composite. **Methods:** 240 standardized disc-shaped resin composite specimens (4 x 1.5 mm) were prepared;120 using microhybrid composite and 120 using nanofilled composite (60 bleaching shade and 60 enamel shade for each group). Each subgroup of 60 specimens was divided into two subclasses (30 specimens each) one was polymerized using halogen and the other using HP LED. Twenty patients with partial or complete dentures participated after written consent. They were divided into two groups of ten patients. The first group received 80 specimens (10 from each subclass) investigated after one week, then received another 80 investigated after 6 months and the second group received 80 specimens investigated after one year. Colour assessment was performed by correlating data obtained using two methods: (1) A reflectance spectrophotometer. (2) A digital determination method. Data were analysed using ANOVA and Tukey's post-hoc test. **Results:** There was no statistically significant difference of mean color change for both curing lights. Good agreement was found between the contemporary spectrophotometer and the digital spectroradiometer at $p < 0.001$. **Conclusions:** Biodegradation of resin composite by the oral environmental factor has a direct effect on its optical properties. Discoloration properties of the resin composite are material, shade and time dependant. However, the curing unit has no effect on discoloration. **Clinical Significance:** Esthetics of resin composite restoration should be routinely re-examined since aging may cause significant influence on the color stability of the resin composites. From an esthetic point of view, it is recommended to use nanocomposites because it has proven to be more efficient with regards to color stability.

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Key Words: In situ, Resin Composite, Bleach Shade, Color Change, Light Curing Unit, Spectrophotometer, Spectroradiometer.

1. Introduction

Ideally, the colour of esthetic dental materials should not be affected by the polymerization mechanism or any aging procedures. However, significant colour changes have been reported in several studies on the colour changes of resin composites after polymerization and aging.

The magnitude of this change varied among the brands and composition of the resin composites, Celik et al.,(2011). In the oral cavity, the materials are exposed to a rather complex milieu that comprises different endogenous (proteins, enzymes, polysaccharides, bacteria) and exogenous substances (all different sorts of compounds coming from the diary intake diet). These components establish a complex and intricate interplay of interactions, which result along with an important mechanical action, in a general biodegradation phenomena towards the biomaterials present in the oral cavity. These processes may permanently alter the properties of the material and compromise its function, Bettencourt et al.,(2010).

Microhybrid composites are most widely used as they provide optimal mechanical and physical properties combined with good polishing properties. However, one of the most important advances of the last few years in the field of filler technology is the application of nanotechnology to dental composites. Nanofillers have been developed with the aim of combining the advantages of hybrid and microfilled composites in the same restorative material. They show favorable mechanical properties, which are at least equal to or may surpass those of hybrid materials. They exhibit a higher surface quality, a better polish and gloss, an increased retention as well as an increased wear resistance, Janus et al.,(2010).

In the era of esthetic composite restorations the demand for overall good color stability is increasing, Schulze et al.,(2003). Currently available composites provide satisfactory strength and high esthetic

appearance. However, despite recent improvements, color stability of light-cured composites after long-term intraoral exposure remains a concern. Various studies have shown that the color stability of composites can be influenced by several extrinsic factors, such as intensity and duration of the polymerization, exposure to environmental factors, including ambient and UV radiation, heat, water or food colorants. Moreover, color stability is also affected by intrinsic factors, such as the composition of the resin matrix, filler loading and particle size distribution, type of photoinitiator and percentage of remaining C = C bonds (%RDB), Sarafianou et al.,(2007). A high degree of conversion not only gives hardness and strength to a material, but also responsible for color stability, Arrais et aal.,(2007). Thus, a reduction in the remaining double bonds to the lowest possible level is normally considered a desirable feature of a polymerization system. The degree of conversion of a given resin composite is influenced by the energy density of the light curing unit, Al-Kheraif (2011).

The early products of light-cured resin composites were cured by UV light, later versions by visible light. A halogen lamp is routinely used as the dental light activation units. Halogen lamps produce light by incandescence, whereby a filament is heated and causes the excitation of atoms over a wide range of energy levels producing a very broad spectrum. Filters are therefore needed to restrict the emitted light to the blue region of the spectrum for the polymerization of resin composites. However, halogen based light-curing units (LCU) used to polymerize dental materials have several drawbacks. For example, halogen bulbs have a limited effective lifetime of approximately 100 hours. In addition, the LCU's bulb, reflector, and filter can degrade over time because of the high operating temperatures and the large quantity of heat, which is produced during operating cycles. This result in a reduction of LCU's curing effectiveness over time, insufficient physical properties, and an increased risk of premature failure of restoration, Al- Martin (1998);Kheraif(2011).

To overcome the problems inherent to halogen LCU, solid-state light emitting diode (LED) technology has been proposed for curing light-activated dental materials. Rather than a hot filament as used in halogen lamps, LED uses junctions of doped semiconductors (p–n junctions) for the generation of blue light. No filters are required in LED because the spectral output of gallium nitride blue LED falls conveniently within the absorption spectrum of the camphoroquinone (CQ) photoinitiator (400–500 nm) present in light-cured dental resin composites. Furthermore, LEDs have an expected lifetime of several thousand hours without significant degradation of light intensity over time, Pilo et al.,(1999).

Third generation LED curing lights have been recently introduced in the dental market by using several different types of LEDs within the light to deliver a broader spectral output compared to narrower spectral output of first generation curing lights. Moreover, this newer generation of curing lights has incorporated the latest advancements in high power LEDs so that they are capable of delivering a power density of about 1,000 mW/cm^2. Since all of the spectral output of the LEDs is concentrated in the blue wavelength range, more efficient curing should be possible with the high power LED lights, resulting in reduced curing time compared with the first generation LED lights and conventional halogen lamps. At the same time, they are lightweight and portable and have long life spans, Stahl et al.,(2000).

Numerous publications are available describing the influence of tungsten halogen light and LED light on the material properties as well as on the conversion rate. However, few publications that investigated the influence of tungsten halogen light and LED light on the color change insitu could be identified.

The aim of this study was to investigate and compare insitu the effect of halogen light curing unit and light emitting diode light curing unit on the color change of resin composite.

2. Materials and Methods

2.1. Materials:

2.1.1. The study sample:

A total of 240 standardized disc-shaped resin composite specimens (4 x 1.5 mm) were prepared for this study. The prepared resin composite specimens were divided into two main groups (120 specimens each) according to the type of composite used where:
R1 represents Artemis micro-hybrid resin composite[a],
R2 represents Filtek Supreme XT nano-filled resin composite[b].

Each group was further subdivided into two subgroups (60 specimens each) according to composite shade selection where S1 represents bleaching shade composite and S2 represents Enamel shade composite.

[a] *Ivoclar Vivadent AG, Schann/Liechtenstein*
[b] *3M ESPE, Minnesota, USA*

Each subgroup was further divided into two groups (30 specimens each) according to polymerization curing unit used where:
L1 represents specimen cured with halogen Light Curing Unit, Elipar 2500,

L2 represents specimen cured with HP LED (High-performance light emitting diode third generation), Elipar Free Light 2.

Time was taken as a variable in this study where 80 resin composite specimens were investigated after one week of polymerization (T1), another 80 after 6 months of polymerization (T2) and the other 80 after one year of polymerization (T3) (Table 1)

Table 1. Resin composite and light curing units according to the manufacturer's information

Material	Description
Artemis[a]	It is a microhybrid resin composite,the resin phase is Dimethacrylates, 76% of fillers (silanized Barium glass filler, Ytterbium trifluoride, silanized Mixed oxide, silanized Ba-Al-fluorosilicate glass, highly dispersed silicone dioxide) and other additives (Stabilizers and catalysts). The resin phase consists of: 7-9 % Bis-GMA 3-4 % TEGDMA 9-11 % Urethanedimethacrylate
Filtek™ Supreme XT Universal Restorative System[b]	It is a nanofilled resin composite, the resin consists of three major components: A blend of 5-15% UDMA (urethane dimethacrylate) and 5-15% Bis-EMA6 (Bisphenol Apolyetheylene glycol diether dimethacrylate) with <5% TEGDMA (tri[ethylene glycol] dimethacrylate) to adjust the viscosity. UDMA and Bis-EMA6 resins are of higher molecular weight and therefore have fewer double bonds per unit of weight. In addition to 1-10% Bis-GMA. They contain a combination of a non-agglomerated/non-aggregated, 20 nm nanosilica filler, and loosely bound agglomerated zirconia/silica nanocluster, consisting of agglomerates of primary zirconia/silica particles with size of 5-20 nm fillers. The cluster particle size range is 0.6 to 1.4 microns. The filler loading is 78.5% by weight. These shades are radiopaque.
Elipar™ 2500 Halogen Curing Light[b]	It produces high intensity visible blue light of 670 mW/cm2 in the 400 to 500 nm waveband. The Elipar 2500 light will cure all dental restoratives activated by light around the 470 nanometer wavelength. The radiation from the tungsten halogen bulb (75 watt) is selectively reflected and filtered within the light to reduce the ultraviolet, infrared radiation and unneeded visible light. Therefore, only the blue light band is available to be absorbed.
Elipar™ FreeLight 2 LED Curing Light[b]	It is a high-performance third generation light unit for intraoral polymerization of dental materials. The unit consists of a charger and a cordless handpiece powered by a rechargeable battery. The unit produces high intensity of approximately 1000 mW/cm2. The unit uses a high-performance Light Emitting Diode (LED) as the light source. In contrast to halogen light units, the unit emits light mainly in the wavelength range of 430 to 480 nm, e.g. the relevant range for camphorquinone-containing products.

2.1.2. Resin composite specimens:

Standardized disc-shaped resin composite specimens (4 x 1.5 mm) were prepared for this study. A split Teflon mold was constructed to help in preparation of the resin composite samples. The mold consisted of two parts with an outer metallic ring to assist reassembling of the two parts together. The internal diameter of the Teflon mold was 4 mm in diameter and 1.5 mm in thickness.

The resin composite material was applied inside the mold cavity over a transparent celluloid Mylar strip (14 mm in thickness) and another celluloid strip was applied over the composite material in the mold. A glass slab was placed under the mold, and another glass slab was placed over the mold in order to flatten the surface of the mold, and then removed. The composite material was light cured in the mold using two different light curing machines, one was a halogen light curing unit[b] (670 mW/cm2) and the other was a high-performance light emitting diode third generation light curing unit[b] (1000 mW/cm2), at a zero distance to the top of the mold, for 20 seconds. One surface of each specimen was marked using a number 0.5 round bur to be easily distinguished from the surface to be examined.

2.1.3. Subjects:

Twenty patients with partial or complete denture were selected for this study after written consent according to Hilsinki principles. They were divided into two groups, ten patients in each group. The specimens of the first ten patients were tested after a period of one week (T1), and then another group of specimens were tested after a period of six months (T2), whereas the other ten patients were tested after the period of one year (T3).

2.2. Methods:

2.2.1. Design of the work:

A pen was used to mark the shape and size of the resin composite disc specimens on the denture flange. Then, a cavity was made using an inverted cone bur

from the center of the marked area towards the marked line periphery inside the labial or lingual flanges of the denture. In each denture, eight cavities were made. The cavities were circular in shape with a diameter larger than 4 mm and a depth larger than 1.5 mm to accommodate the resin composite discs specimens. Each eight resin composite specimens of different categories were placed in each patient, in each different time period, in a sequential order so that it can be differentiated easily. The resin composite specimens were seated in the cavity made inside the denture and cemented in place using resin co+mposite cement, Multilink[a].

Color assessment was done by correlating data obtained using two methods:

(1) A reflectance spectrophotometer (UVPC Shimadzu 3101PC – Germany). The measurements were done in the visible range of wavelength (400-800 nm). The color change between the baseline and after the treatment was measured. Colorimetric values of the specimens were determined using the L* a* b* system of the Commission Internationale de l'Eclairage (CIE L* a* b* color scale).

(2) A digital determination method - Minolta CS1000A (Konica Minolta Business Technologies Inc., Tokyo, Japan). Then, the color difference ΔE* was calculated for each sample using the following equation:

$$\Delta E^* = \{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}^{1/2}$$

2.2.2.Statistical analysis:

Data was recorded, tabulated and statistically analyzed. Data were presented as mean and standard deviation (SD) values. Regression model using Multi Factorial Analysis of Variance (ANOVA) was used in testing significance for the effect of type of resin, shade, curing modes, time and their interactions on color, Tukey's post-hoc test was used for pair-wise comparison between the means when ANOVA test is significant. The significance level was set at $p \leq 0.05$.

3. Results

The color assessment was done using two methods - contemporary spectrophotometer method and digital method – and the tristimulus L*a*b color parameters were calculated and subtracted from the values obtained from the one week clinical study (baseline values).

3.1.Using Contemporary Spectrophotometer:

The mean ΔE recorded for Filtek Supreme XT (R2) was lower than that recorded for Artemis (R1) (5.5 ± 1.7 for R2, and 6.4 ± 1.4 for R1). The mean ΔE recorded for the bleaching shade (S1) was lower than that recorded for Enamel Shade (S2) (5.7 ± 1.6 for S1, and 6.3 ± 1.6 for S2). There was no statistically significantly difference of mean ΔE between the usage of the curing lights, Halogen light curing unit (L1) and high-performance light emitting diode (HP LED) curing unit (L2). The mean ΔE recorded for 6 months (T2) was lower than that recorded for 1 year (T3) (5.1 ± 1.4 for T2, and 6.9 ± 1.4 for T3).

There was a statistically significant difference between different materials R1 and R2 at an F-value of 14.109 and p-value < 0.001; between different shades S1 and S2 at an F-Value of 6.663 and p-value < 0.011; between different times T2 and T3 at an F-value of 55.429 and p-value < 0.001. There was no significant difference between different curing lights L1 and L2. When the four variables interacted together, the difference was significant at F-value of 8.386 and p-value < 0.001 (Table 2).

Tukey's test was used to rank and test the significance of differences between the mean ΔE of different subgroups. The lowest mean ΔE recorded was R2S1L1T2 (4.3 ± 1.5) and R2S1L2T2 (4.3 ± 1.1). While the highest mean ΔE recorded was R1S2L1T3 (8.0 ± 0.7) followed by R1S1L2T3 (7.4 ± 0.9) (Figure 1).

Figure 1. Bar chart representing means and SD for comparison between ΔE values using spectrophotometer with different interactions

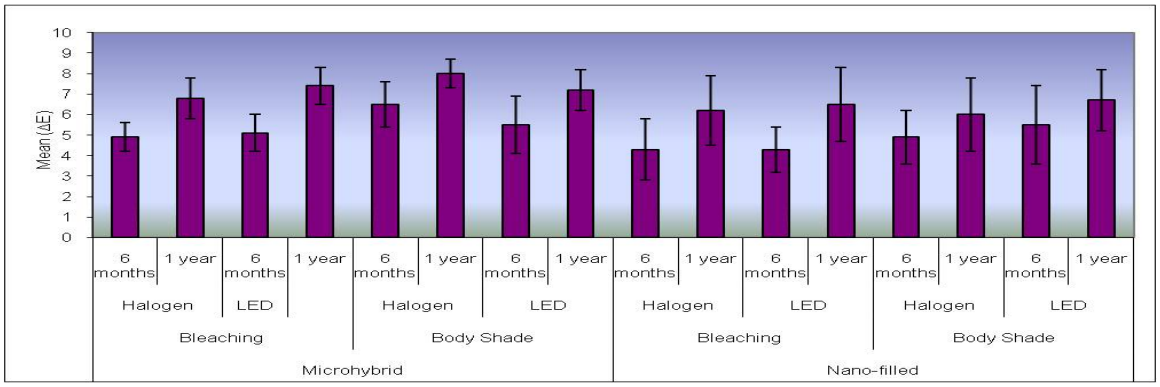

Figure 2. Bar chart representing means and SD for comparison between ΔE values using spectroradiometer with different interactions

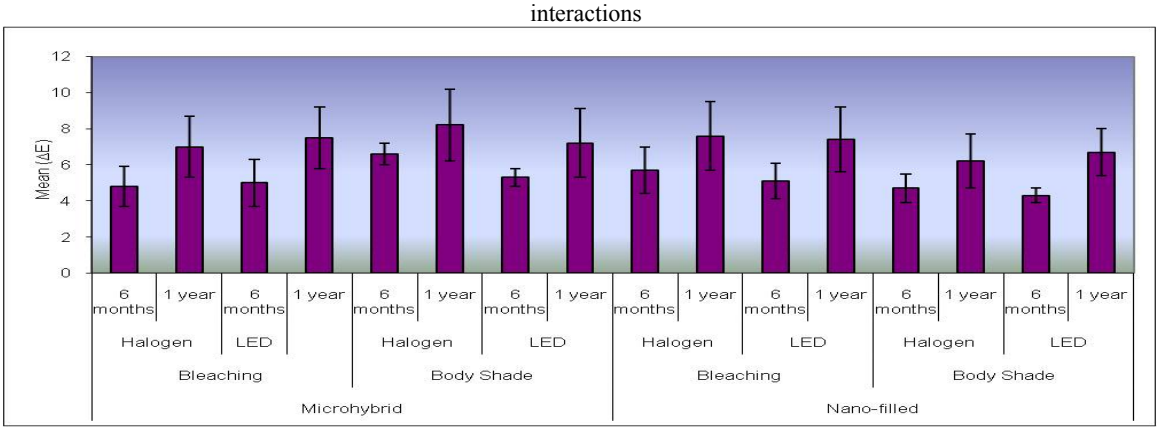

Table 2. Regression model results for the effect of different variables on ΔE using spectrophotometer

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	151.121	15	10.075	5.733	<0.001*
Resin	24.794	1	24.794	14.109	<0.001*
Shade	11.708	1	11.708	6.663	0.011*
Curing	0.276	1	0.276	0.157	0.693
Time	97.408	1	97.408	55.429	<0.001*
Resin x Shade x Curing x Time	10.004	1	10.004	8.386	<0.001*

R Squared = 0.934 (Adjusted R Squared = 0.912), df: degrees of freedom, *: *Significant at p ≤ 0.05*

3.2.Using Digital Spectro-radiometer:

The mean ΔE recorded for Filtek Supreme XT (R2) was lower than that recorded for Artemis (R1) (5.8 ± 1.6 for R2, and 6.5 ± 1.8 for R1). The mean ΔE recorded for the bleaching shade (S1) was lower than that recorded for Enamel Shade (S2) (5.8 ± 1.7 for S1, and 6.5 ± 1.8 for S2). There was no statistically significantly difference between mean ΔE between the usage of the curing lights, Halogen light curing unit (L1) and high-performance light emitting diode (HP LED) curing unit (L2). The mean ΔE recorded for 6 months (T2) was lower than that recorded for 1 year (T3) (5.3 ± 1.4 for T2, and 6.9 ± 1.7 for T3).

There was a statistically significant difference between different materials R1 and R2 at an F-value of 6.665 and p-value < 0.011; between different shades S1 and S2 at an F-Value of 6.896 and p-value < 0.010; between different times T2 and T3 at an F-value of 38.995 and p-value < 0.001. There was no significant difference between different curing lights L1 and L2. When the four variables interacted together, the difference was significant at F-value of 19.224 and p-value < 0.001 (Table 3).

Tukey's test was used to rank and test the significance of differences between the mean ΔE of different subgroups. The lowest mean ΔE recorded was R2S2L2T2 (4.3 ± 0.4) and R2S2L1T2 (4.7 ± 0.8). While the highest mean ΔE recorded was R1S2L1T3 (8.2 ± 2.0) (Figure 2)

Table 3. Regression model results for the effect of different variables on ΔE using digital spectroradiometer

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	146.921	15	9.795	4.581	<0.001*
Resin	14.249	1	14.249	6.665	0.011*
Shade	14.744	1	14.744	6.896	0.010*
Curing	1.983	1	1.983	0.928	0.338
Time	83.371	1	83.371	38.995	<0.001*
Resin x Shade x Curing x Time	20.278	1	20.278	19.224	<0.001*

R Squared = 0.880 (Adjusted R Squared = 0.872), df: degrees of freedom, *: *Significant at p ≤ 0.05*

Table 4. Results of Cronbach's alpha and intra-class correlation coefficient (ICC) for the agreement between ΔE measurements with the two devices (Contemporary Spectrophotometer and digital Spectroradiometer)

Cronbach's alpha	ICC	P-value
0.715	0.502	0.001*

3.3.Agreement between the Contemporary Spectrophotometer and the Digital Spectroradiometer

Correlation was done to describe the agreement (Concordance) between the contemporary spectrophotometer and the digital spectroradiometer using Cronbach's alpha reliability coefficient. Cronbach's alpha reliability coefficient normally ranges between 0 and 1. The closer Cronbach's alpha coefficient is to 1.0, the higher the reliability. In table (4)Cronbach's alpha results indicate good agreement (0.715) between the two devices at ICC value of 0.502 and p-value < 0.001. This agreement is statistically significant.

4. Discussion

This study was conducted inside patients' mouth (in situ) where the oral environment has a powerful effect on the optical properties of resin composite. In the oral cavity, the influence of heat caused by hot drinks and hot food may be more noteworthy. In addition, the resin surface roughness was changed by the mastication process and the discolouring factors and deposits can stay longer on rough resin surfaces, Celik et al.,(2011).

Several studies have investigated the process of biodegradation of resin composites in the presence of salivary enzymes. Inflammatory [Cholesterol esterase (CE)] and salivary [pseudocholinesterase (PCE)] enzymes can cause the breakdown of Bisphenol- A glycidyl dimethacrylate (BisGMA) and triethylene glycol dimethacrylate (TEGDMA) components in resin composites and hence increase discoloration, Wiggins et al.,(2004).

It has been stated that the matrix-filler interface plays a major role in the uptake of water by composites. Changes in optical properties within the matrices or the matrix-filler interface therefore could have been responsible for the different values of ΔE, Lee and Powers(2006).

The change in color could also be due to some physical adsorption of specific drink components on the composite surface. Due to the hydrophilic behavior of the polymeric matrix, the penetration of drink components into the composite was facilitated by the action of water; the presence of water from saliva softens the polymer by swelling the network and reducing the frictional forces between the polymer chains Buchalla et al.,(2002). This water sorption of the resin composite may decrease the life of the restoration by expanding and plasticizing the resin component, hydrolyzing the silane, and causing micro-crack formations. These micro-cracks or the interfacial gaps at the interface between filler and matrix allow stain penetration and discoloration, Bagheri et al.,(2005); Barutcigil and Yıldız(2012).

Moreover, dietary factors containing tannin have shown high chromogenic potential. It has been suggested that the stain forms by precipitation of iron sulfide, and that the sulfur was provided by exposed thiol groups from denatured proteins and the iron originated from the diet. Besides, air voids in the resin material may lead to inhibition zones of unpolymerized material, resulting in lower color stability, Luiz et al.,(2007). Extrinsic factors for discoloration include staining by adsorption or absorption of colorants as a result of contamination from exogenous sources. Extrinsic factors for discoloration are known to cause staining of oral tissues and restorations, especially in combination with dietary factors and drinks, including coffee, tea, and nicotine, Khokhar et al.,(1991).

In this study, the results have shown clinically perceptible ($\Delta E \geq 3.3$) color change that occurred in both resin composite materials. This has been attributed to a wide variety of possible causes. Many color changes were described as the result of physical adsorption, absorption or physico-chemical reactions of the ingredients of the composite materials during exposure to the oral environment as previously mentioned. Inherent discoloration of polymeric materials could also be due to an oxidation of the unreacted carbon-carbon double bonds, producing colored peroxide compounds, Lim et al.,(2001).

In this study, the nanofilled Filtek Supreme XT resin composite demonstrated less color changes than the microhybrid Artemis resin composite. These results were in agreement with that of Sarafianou *et al.,(2007)*who found that nanocomposite Filtek Supreme XT had less color changes than microhybrid composite Tetric Ceram. They attributed their findings to the resin composition, type and size of fillers that influence the color stability of tooth-colored restorative materials. Where in Filtek Supreme XT, the partial substitution of TEGDMA for urethane dimethacrylate (UDMA) comonomer in BisGMA/TEGDMA resin matrix has been shown to reduce water uptake and stain susceptibility. Water uptake in BisGMA-based resins has been found to increase proportionally to TEGDMA concentration. In addition, Filtek Supreme XT contains UDMA and BisEMA monomers, both of which are less hydrophilic than BisGMA and TEGDMA. Filtek Supreme XT as a nanocomposite has in its composition zirconia/silica cluster fillers with particle size of 5-20 nm fillers and with filler weight 78.5%. These smaller filler particles together with the high filler loading shows overall high gloss and polishability of Filtek Supreme XT and hence, less stain susceptibility.

On the other hand, Artemis; the microhybrid resin composite, contains ytterbiumtri fluoride for

fluoride release. The fact that this component is water-soluble and leaches out after immersion in a solution might have affected the early color changes in Artemis, Gupta et al.,(2005).

The color changes, in this study, were clinically unacceptable because $\Delta E > 3.3$ for the two materials studied in different times of investigation six months and one year, having one week as the baseline. In this study, the color change of the resin composites after one year of investigation was higher than six months; in addition, there was significant increase in color change compared with one week of investigation (the base line). Based on these results, time was found to be a critical factor affecting the color stability of resin composite, as with increasing time of investigation, the color changes becomes more pronounced. These results are in agreement with that of Polyzois *et al*.,(1999) and Furuse *et al.,(2008)*who evaluated the color stability of restorative resin materials at various time intervals and found similar findings.

In this study, there was no difference between halogen and HP LED curing units on the discoloration of resin composite, this result was in agreement with that of Micali and Basting (2004) who explained these results based on the fact that LED has wavelength 430 – 480 nm with a peak 460 nm which is ideal for activating materials with camphorquinone as photo initiator. On the other hand, the halogen curing unit has a broad spectrum of wavelength 400-500 nm, which is efficient for photopolymerization of resin composite material activating camphorquinone as photo initiator. And since, adequate polymerization depends on light source intensity and wavelength, these parameters are sufficient enough to obtain similar results on color change of resin composite.

In this study, the bleach shade demonstrated higher color stability than the enamel shade; this was in agreement with Paravina *et al*.,(2004*)*who attributed the light shade of beaching-shade resin composites to originate from inorganic oxides added to match extralight tooth shades. Moreover, some initiators in the bleaching shade accelerate the polymerization of resin composite and decrease the aging process of polymerized materials, and enhance color stability.

5. Conclusions:

Biodegradation of resin composite by the oral environmental factor has a direct effect on its optical properties.

i. Discoloration properties of the resin composite are material, shade and time dependant. However, the curing unit has no effect on discoloration.
ii. Nanofilled resin composite shows better color stability compared to microhybrid resin composite.
iii. Curing of resin composite using HP LED curing unit has similar effects on color stability as halogen curing unit.
iv. Increasing the aging time from one week to one year, increase the color change of resin composite. It seems to be a linear relationship.
v. The digital spectroradiometer has proven to be an efficient alternative method for color determination.

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