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Influence of Curing Light Type and Staining Medium on the Discoloring Stability of Dental Restorative Composite

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Abstract: Discoloration kinetics of 4 commercial nano-filled resin composites polymerized with LED and QTH source types in 4 different staining media held at 37°C are investigated. Quartz Tungsten Halogen (QTH) and Light Emitting Diode (LED) were used. Dental composites color change was monitored with a digital spectrophotometer at one, two and four weeks for quantitative comparison. The kinetics and intensities of the color change in accelerated aging tests in staining media were monitored and discussed. Discoloring followed diffusive driven kinetics. The four composites polymerized with QTH light showed higher differences in color change intensities compared to LED cured ones. All the QTH cured samples have shown significant higher variations of color changes intensities compared to LED samples ($p<0.05$). LED cured composite showed comparatively more stable initial shade persistence than QTH cured ones, which may be explained in terms of higher level of polymerization and surface hardness reached by the LED light cure resinous composite matrix.

Keywords: Bioengineering, Biomaterials, Biotechnology, Biochemistry, Bioactive Scaffolds

Introduction

Tooth coloured resin based composites have the benefit of better aesthetic properties and are widely used aesthetic restorative materials in modern dentistry (Rajkumar *et al*., 2011). Initially, resin composites were manufactured as chemically activated materials; two pastes system based on initiator (benzoyl peroxide) and activator (a tertiary amine such as N,N-dimethyl p toluidine) system. The key drawbacks of chemically activation are the limited working time, difficulty in finishing and colour stability (Anusavice, 2003). These drawbacks led to the development of light cured resin composites, which could provide a better control over working and finishing times while improving colour stability. In addition, light cured resin materials are activated by visible blue light (wavelength = 400-470 nm) hence avoiding from the hazardous effects of radiation. Light cured composites contain canphorquinone as photo-initiator which facilitates better control over working time and enhances colour stability (Rajkumar *et al*., 2011; Anusavice, 2003), but discolouration still remains the main concern.

In order to cure light activated resin composites, various types of light sources have been used including Quartz Tungsten Halogen (QTH) and Light Emitting Diode (LED).

The QTH system is comprised of a quartz bulb and tungsten filament that works in the halogen environment. In order to activate the canphorquinone initiator, the emitted light is filtered to dissipate heat and to transmit only the violet-blue part of the visible light spectrum. These systems produce a spectrum of 400-500 nm with an output range of 400-800 mW/cm² (Filipov and Vladimirov, 2006). There are several disadvantages of using QTH system, among these there are the prolonged curing time, the source larger size and the need of filters and fans to dissipate the generated heat (Yazici *et al*., 2007; Aversa *et al*., 2016a; 2016b; 2016c; 2016d; 2016e; 2016f; 2016g; 2016h; 2016i; 2016j; 2016k; 2016l; 2016m; 2016n; 2016o).

The LED systems include Gallium nitride blue diodes and silicon carbide diodes with a power output of 7 µW (Park *et al*., 2005). The LEDs sources are generally cordless, lightweight, of compact size and powered by batteries. The wavelength is usually in the range of 400-500 nm. All the emitted condensed light is fully utilized and no additional filters or cooling fan are required. Finally, due to the electronic natue of the emitted light, there is no drop in intensity and diodes do not need replacement (Christensen, 2002). The main disadvantages are the relatively high cost of the equipment and that the batteries require recharging.

Although no significant mechanical differences have been reported in the curing performance of QTH and LED systems (Campregher *et al*., 2007), the QTH light curing system tends to exhibit more yellowing of the resin composites upon curing (Brackett *et al*., 2007). Moreover, LED systems are often considered similar or comparatively better than QTH system in terms of the degree of polymerization (Hasler *et al*., 2006), polymerization shrinkage (Uhl *et al*., 2005), micro-leakage (Attar and Korkmaz, 2007), wear rate (Ramp *et al*., 2006), flexural properties (Keogh *et al*., 2004) and hardness (de Araújo *et al*., 2008).

Currently, there are no reports that compare these two systems in terms of colour changes during aging in staining media (Imirzalioglu *et al*., 2010; Rajkumar *et al*., 2011; Seghi *et al*., 1990). In these works it was hypothesized that both the source type and the amount of energy provided by the curing light can not only change the colour of the dental restorative materials but also its discolouration aptitude in staining beverages. The aim of this study was to investigate the variations in shade change in various beverages of dental nano-filled resin composite cured with QTH and LED light curing systems (Paul *et al*., 2002; Zafar, 2013; Zafar and Ahmed, 2015; Ullah and Zafar, 2015).

Materials and Methods

Two different light curing systems were evaluated on various resin composites for colour stability. A QTH light cure unit (Optilight Plus, Curing Light, Gnatus, Ribeirão Preto, SP, Brazil) and a LED cure unit (Elipar™ LED Curing Light, 3M ESPE, Deutschland, GmBH, 41453, Germany). The cured samples were conditioned in various staining beverages and analysed for time dependency of their colour changes (Table 1).

Sample Preparation

For each material, 100 disc shaped specimens (6 mm diameter and 3 mm thickness) were prepared using specially tailor made Teflon moulds. A glass slide was used to compress and flatten the materials and to remove the excess resin. Before curing, all samples were divided randomly in to two study groups: QTH and LED (n = 50 for each group). QTH group samples were cured for twenty seconds using a halogen light source (Optilight Plus, Curing Light, Gnatus, Ribeirão Preto, SP, Brazil). LED groups samples were light cured for twenty seconds using a diode electronic light source (Elipar™ LED Curing Light, 3M ESPE, Deutschland, GmBH, 41453, Germany). Light sources were held at a distance of 2 mm from the surface of sample (Petrescu and Calautit, 2016a; 2016b; Petrescu *et al*., 2015; 2016a; 2016b; 2016c; 2016d; 2016e). All samples were inspected carefully for uniformity. Samples with any defect, visible crack or voids were excluded. Each sample was polished using a finishing discs kit (Sof-LEX disks; 3M ESPE, ST Paul.MN) and slow speed air turbine handpiece (uniform pressure and circular motions were used) according to manufacturer's guidelines. The final thickness of samples after polishing was 2.0±0.01 mm. Prepared samples from each materials under investigation (n = 100) were randomly divided into various study groups for longevity analysis (Fig. 1).

Preparation of Beverage Solutions and Aging Treatment

The effects of four hot beverages (karkada, tea, yansoon and coffee) on the colour stability after light curing were investigated (Table 1). All beverage solutions were prepared by boiling tea leave/coffee granules in deionized distilled water for 5 min. Final concentration of each solution was maintained at 3.5% and pH in the range of 3.5-4.5. The pH of the solution was maintained throughout the experimental period using a digital pH meter (Clean PH500, First Clean Co. 14656 Valley Blvd, City of Industry, 91746). Additives such as sugar, milk or flavourings were not added to any experimental solutions.

Specimens were immersed in 50 mL of ageing media in air tight containers and were stored in an incubator at 37°C. Ageing beverage medium was changed cyclically after every 6 h to mimic the daily consumption of the beverage daily. Specimens were rinsed using deionized water before colour shade measuring and change of the ageing beverage media.

Five groups of samples for each curing light source type were prepared: Karkada [Group I; n = 10]; black tea [Group II; n = 10]; yansoon [Group III; n = 10]; coffee [Group IV; n = 10]; deionized distilled water used as control group for aging (n = 10).

Assessment of Colour Longevity

The longevity of the dental composites in staining media was evaluated by recording the colour changes. A digital spectrophotometer Chroma Meter (CR 400; Konik Minolta, Japan) has been used to perform a colorimetric analysis to quantitatively monitor the colour changes in each restorative composites sample aged for one, two and four weeks of staining liquid conditioning.

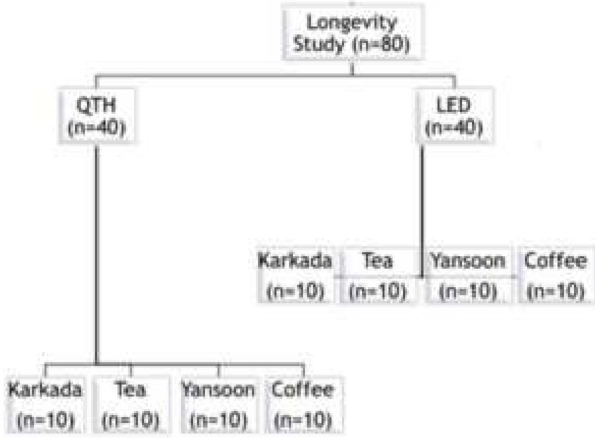

Fig. 1. Schematic presentation of study design, groups and sample distribution

Table 1. Restorative materials and beverages

		Description	Manufacturer
Restorative materials			
Filtek Z350 XT	A	Resin based nanocomposite	*3M ESPE, St. Paul, MN 55144-1000 USA*
Filtek Z250 XT	B	Resin based nanocomposite	*3M ESPE, St. Paul, MN 55144-1000 USA*
Artiste	C	Nanocomposite resin composite	*Pentron Clinical Orange CA, 92867, USA*
GC Fuji II LC	D	Capsule Glass ionomer	*GC Corporation, Tokyo, Japan*
Beverages used			
Karkada	I	Herbal Tea	*Al Diafa, J.N. Spain*
Tea	II	Black Tea	*Al Diafa, J.N. Spain*
Yansoon	III	Herbal Tea	*Lipton yellow label, Riyadh, Saudi Arabia*
Coffee	IV	Instant freeze dried coffee	*Nescafe, Original, USA*

The spectrophotometer returned a precise quantitative measurement of surface colours by recording the spectral reflectance/transmittance curve of the specimen.

A prism disperses tungsten-filament bulb light into a spectrum of wavelength (5 and 20 nm) bands (Bowen and Marjenhoff, 1992; Kim-Pusateri *et al*., 2007; Dozić *et al*., 2007; Al-Dharrab, 2013; Eldiwany *et al*., 1995; Ruchi *et al*., 2010). The colour changes (ΔE) of each specimen were calculated by analysing the coordinate values before and after beverage ageing. Colour changes were quantified using the following equation (Imirzalioglu *et al*., 2010):

$$\Delta Eab = \left[(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2\right]^{1/2}$$

Where:

- ΔEab = The overall colour difference
- ΔL = The lightness
- Δa = The red colour on the positive and the green colour on the negative side
- Δb = The yellow colour on the positive and the blue colour on the negative side

The results were statistically analysed by using SPSS version 20 and 95% Confidence Intervals (CI). Chi-square tests were used to evaluate the experimental data significance. A *P-value* < 0.05 was considered significant.

The regression parameter (R^2), which measures the relationship between the two variables colour change and time, was evaluated for all staining tests at significance $p<0.05$.

Results

All samples exhibited increasing discoloration with time upon ageing in the staining liquids.

Discolouring kinetics was investigated by plotting the digital spectrophotometry colour change values as a function of the exposure time.

The colour changes linearly related to the square root of time (Fig. 2) with a near unity regression parameter (last column of Table 2).

All colour changes data were grouped for type of composite resin, cure light and staining beverage and plotted as a function of the square root of time. Significant differences in staining properties among the four beverages (karkada, tea, coffee and yansoon were observed (Fig 3a through d).

Figure 3a-c show the colour changes observed for GC Fuji II LC, Filtek Z250 XT and Filtek Z350 XT, respectively. The colour differences between LED and QTH cured samples become higher and statistically significant at week 2 and week 4 for all beverages.

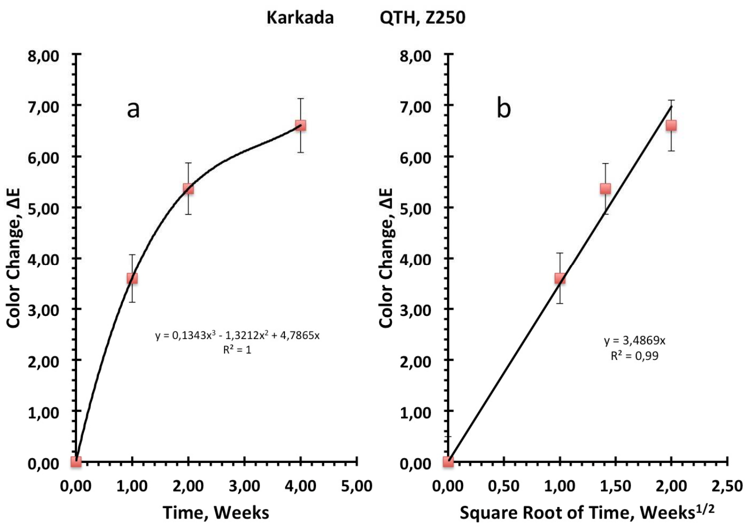

Fig. 2. Discolouring kinetics of QTH cured Z250 restorative resin composite in karkada beverage plotted as a function of time (a) and of the square route of time (b)

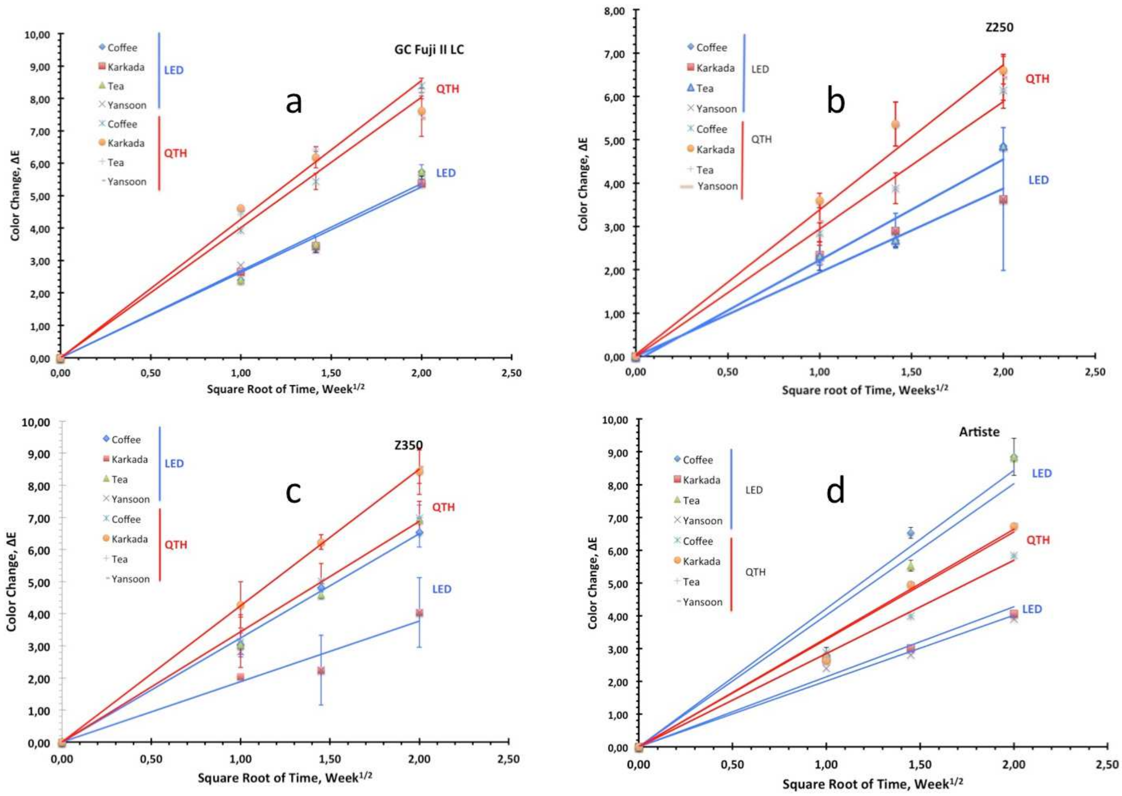

Fig. 3. Diffusion controlled discoloring kinetic for QTH cured (red lines) and LED cured (blue lines) for (a) GC Fuji II LC, (b) Z250, (c) Z350, (d) Artiste resin composites in different staining media

The colour differences following aging in any of the staining media (Fig. 3a and raw data in Table 2) were significantly higher for QTH light cured samples (red regression lines in the figure) than for LED cured specimens (blue regression lines in the figure). QTH and LED cured samples reached at four weeks mean grade colour changes for GC Fuji II LC of 15.60±0.19 and 7.90±0.47, respectively. Filtek Z250 XT reached 6.33±0.23 and 4.24±0.71 respectively for QTH and LED. Tea and coffee produced slightly higher discolouring compared to yansoon and karkada both for QTH and LED cured composites.

Table 2. The summary statistics of response variable (ΔE_{ab} values) at week1, week 2 and week 4 by light type with four different types of beverages for restorative materials

Cure light	Beverage	Week = 1 $Week^{1/2}$ = 1	Week = 2 $Week^{1/2}$ = 1.4	Week = 4 $Week^{1/2}$ = 2	Linear Regr., R^2
	Artiste				
LED	Coffee	2.80±0.23	6.53±0.17	8.85±0.57	0,955
	Karkada	2.70±0.05	3.01±0.31	4.05±0.91	0,964
	Tea	2.80±0.23	5.53±0.17	8.85±0.57	0,957
	Yansoon	2.56±0.16	3.01±0.27	4.05±0.79	0,981
QTH	Coffee	2.88±0.20	4.00±0.34	5.84±0.52	0,998
	Karkada	2.63±0.24	4.95±0.62	6.74±0.48	0,983
	Tea	2.72±0.35	4.00±0.34	5.84±0.52	0,997
	Yansoon	2.70±0.12	5.01±0.56	6.80±0.22	0,985
	GC Fuji II LC				
LED	Coffee	2.40±0.25	3.49±0.22	5.74±0.07	0,992
	Karkada	2.65±0.42	3.46±0.31	5.44±0.14	0,997
	Tea	2.40±0.25	3.49±0.22	5.74±0.07	0,992
	Yansoon	2.85±0.09	3.34±0.16	5.44±0.14	0,992
QTH	Coffee	3.40±0.15	5.44±0.12	8.40±0.03	0,998
	Karkada	4.62±0.38	6.18±0.58	7.62±0.82	0,990
	Tea	4.43±0.08	6.44±0.12	8.20±0.46	0,996
	Yansoon	4.57±0.32	6.18±0.58	7.41±0.40	0,987
	Z250				
LED	Coffee	2.32±0.33	2.69±0.16	4.86±0.01	0,969
	Karkada	2.34±0.21	2.90±0.40	3.63±1.65	0,973
	Tea	2.32±0.33	2.69±0.16	4.86±0.01	0,969
	Yansoon	2.20±0.10	2.90±0.40	3.63±1.65	0,983
QTH	Coffee	2.84±0.10	3.88±0.35	6.14±0.41	0,993
	Karkada	3.60±0.47	5.36±0.51	6.60±0.53	0,988
	Tea	2.83±0.26	3.88±0.35	6.14±0.41	0,993
	Yansoon	3.12±0.16	5.36±0.51	6.44±0.32	0,979
	Z350				
LED	Coffee	3.03±0.38	4.81±0.13	6.93±0.45	0,998
	Karkada	2.05±0.03	2.24±1.09	4.04±1.09	0,959
	Tea	3.03±0.38	4.60±0.14	6.93±0.45	0,993
	Yansoon	2.83±0.10	2.24±1.09	4.04±1.09	0,876
QTH	Coffee	3.13±0.76	5.03±0.54	6.58±0.25	0,996
	Karkada	4.28±0.72	6.23±0.23	8.44±0.73	0,872
	Tea	3.15±0.82	5.03±0.54	7.00±0.51	0,844
	Yansoon	3.52±0.38	6.23±0.23	8.58±0.52	0,808

The Z350 showed similar behaviour with yet statistically significant grade change values at four weeks between 7.75±0.88 and 5.39±1.56 for QTH and LED light curing, respectively. It has been observed that both Z250 and Z350, either for QTH and LED light curing, were more sensitive to discolouring induced by karkada and yansoon.

A more complex staining behaviour was observed for LED cured Artiste composite (Fig. 3d); a higher sensitivity to discolouration of the QTH cured Artiste composite with respect to the LED cured ones was observed for staining induced by karkada and yansoon (3.98±0.11for LED cured and about 6.5±0.54 for QTH), while, contrarily to all the previous discussed systems, LED cured composite presented significantly higher discolouring compared to QTH ones when exposed to coffee and tea (8.86±0.01 for LED and about 5.84±0.01 for QTH light cures, respectively). Less significant differences in discolouring properties of the staining media were observed among the QTH cured samples (mean discolouring grade values ranged from 5.84±0.01 to 6.5±0.54).

Discussion

The resin composites gained popularity as restorative material due to their aesthetic properties such as translucency and colour matching to natural teeth (Rajkumar *et al*., 2011). Ideally, restorative materials should have a matching shade that is stable for colour changes over a long period of time. Nevertheless, these materials have been described to change their colour with time in response to various oral environmental, intrinsic and extrinsic factors (Rajkumar *et al*., 2011).

Colour changes in the composite polymeric matrix are induced by the diffusion and absorption of increasing amount of chromogenic staining substances. A square root of the time dependency of the colour change was observed in our tests. All mass transfer processes driven by diffusive phenomena, in fact, are described by a linear behaviour in the early sorption stages when plotted as a function of the square root of the time (Crank, 1975). Sorption and diffusion of the chromogenic substance in the composite polymeric matrix will increase the concentration of the staining substance, which, then, will progressively alter the colour shade.

Moreover, colour change of resin composites on curing has been described to show characteristic variations in terms of chrome values (Seghi *et al*., 1990). In the current study, the intrinsic colour stability of dental resin composites curing using QTH or LED light has been evaluated.

The effect of aging on cured materials in different staining beverages was also studied. A large number of electronic devices (spectrophotometers, colorimeters, digital colour analysers, or their combinations) are presently available for clinical use (Paul *et al*., 2002).

In terms of curing light source, LED curing resulted in less colour changes and better stability for all composites.

In QTH curing system, the light is mainly dissipated as heat. The excessive heat may increase the temperature of restoration and likely favour oxidation processes affecting the subsequent staining substances sorption attitude.

Conversely, the energy emitted by LED curing system is fully used with less heat dispersion avoiding potential oxidation processes. QTH cured composites have been described to exhibited more yellowing of upon curing (Brackett *et al*., 2007). In our accelerated aging tests, QTH curing showed more intrinsic colour changes attitude than LED curing for all tested composites materials. GC Fuji II LC showed the more remarkable colour variations upon ageing. This composite belongs to the class of the glass ionomers that are used for their fluoride release in the oral cavity (Zafar, 2013; Zafar and Ahmed, 2015). Based on the fact that GC Fuji II LC materials are more prone to colour changes upon curing and aging, they should not be considered as the best choice for aesthetic restorations (Bowen and Marjenhoff, 1992).

A number of factors including nature of light, its intensity, type of measuring tools and experimental procedures during shade assessment may lead to inaccurate measurements (Apicella *et al*., 2005; Simeone *et al*., 2005; Kim-Pusateri *et al*., 2007; Dozić *et al*., 2007).

Although the effects of curing by LED and QTH have been compared in previous studies (Al-Dharrab, 2013), there is no published complete study comparing the effects in terms of colour changes during aging in staining media.

Recently, QTH and LED have been compared for their curing performance and effectiveness in terms of hardness and depth of cure. In terms of depth of cure, LED has been reported to cure similar or to increase depths through the resin composites (Apicella *et al*., 2002; 2005; Simeone *et al*., 2005; Ruchi *et al*., 2010). Considering the surface micro-hardness, several studies (Yaman *et al*., 2011) reported that LED-curing results is better micro-hardness than QTH. These results are suggesting that the type of curing light source may affect the level of polymerization and cross linking of the resin through the composite leading to higher surface hardness and glass transition temperatures.

Although our current study has a number of limitations due to the lack of dynamic stimulation of a real oral environment and clinical conditions (Aversa *et al*., 2009; Sorrentino *et al*., 2007; 2009; Perillo *et al*., 2010; Gramanzini *et al*., 2016; Marrelli *et al*., 2015), the base mechanisms and kinetics of resin composite staining have been identified and discussed.

QTH cured materials have shown almost in all tested samples higher discolorations than LED cured materials (with the exception of coffee and black tea in LED cured Artiste). The colour changes induced by the different beverages (karkada, tea, coffee or yansoon) were different for each material and exposure time leading to the conclusion that we are dealing with intrinsic discolouring driven by staining species sorption and diffusive phenomena in the polymeric composite matrix.

The specific sensitivity of each material could depend both on the resinous chemical nature and on the levels of polymerization and undesired chemical changes that are singularly reached by the resinous systems upon curing.

Conclusion

Discoloration kinetics of 4 commercial nano-filled resin composites polymerized with LED and QTH source types in 4 different staining media held at 37°C are investigated. Quartz Tungsten Halogen (QTH) and Light Emitting Diode (LED) were used. Dental composites color change was monitored with a digital spectrophotometer at one, two and four weeks for quantitative comparison.

The kinetics and intensities of the color change in accelerated aging tests in staining media were monitored and discussed. Discoloring followed diffusive driven kinetics. The four composites polymerized with QTH light showed higher differences in color change intensities compared to LED cured ones. All the QTH cured samples have shown significant higher variations of color changes intensities compared to LED samples ($p<0.05$). LED cured composite showed comparatively more stable initial shade persistence than QTH cured ones, which may be explained in terms of higher level of polymerization and surface hardness reached by the LED light cure resinous composite matrix.

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Author's Contributions

All the authors contributed equally to prepare, develop and carry out this manuscript.

Ethics

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved.

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