Effect of Different Light-Curing Modes on Degree of Conversion, Staining Susceptibility and Stain's Retention Using Different Beverages in a Nanofilled Composite Resin

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ABSTRACT

Statement of Problem: It is unknown whether the staining pigment concentration would affect the color of composite resin and whether the absorption of the staining pigment is related to the degree of conversion (DC).

Purpose: The purpose of this study was to evaluate the effect of light-curing units (LCUs) on DC, superficial staining (ΔE), and pigment concentration (PC) in a nanofilled composite resin (Z350, 3M ESPE) using different beverages.

Materials and Methods: Specimens were polymerized for 20 seconds using four LCUs ($N = 50$): quartz–tungsten–halogen (QTH)—450 mW/cm^2; laser (LAS)—300 mW/cm^2; second-generation light-emitting diode (LED)—1100 mW/cm^2; and third generation LED—700 mW/cm^2. DC (%) was measured using Fourier transform infrared spectroscopy. Specimens concerning each group ($N = 10$) were then immersed in one of the solutions (distilled water, red wine, whisky, coffee, and cola—40 min/day, for 40 days). Specimen's color was measured before and after exposure to solutions using a colorimeter (*Commission Internacionale de I'Eclairaga L* a* b** color scale), and ΔE was calculated. Specimens were then prepared for the spectrophotometric analysis to measure PC. Data were submitted to two-way analysis of variance and Tukey's test ($p = 0.05$).

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Results: DC: QTH presented the lowest DC, with statistical differences for LAS, LED 2, and LED 3. Whisky and wine showed lower PC mean values than cola and coffee. No statistical difference was observed for LCUs regarding PC and all staining solutions, except cola. Whisky showed the highest values for ΔE regarding all LCUs. Wine showed statistically lower ΔE than whisky, with water presenting the lowest ΔE. LAS and QTH showed higher values than LED 2 concerning ΔE.

Conclusion: LCUs interfered with DC and altered the PC and ΔE of the composite resin submitted to different staining solutions. There was no correlation among DC, PC, and ΔE.

CLINICAL SIGNIFICANCE

Light-curing modes might interfere with staining susceptibility, stain's retention, and DC of a composite resin, compromising the clinical performance. The highest pigment absorption was not associated with the highest superficial staining of the composite resin. Alcoholic drinks lead to greater superficial staining and non-alcoholic solutions lead to a higher pigment concentration.

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INTRODUCTION

Adequate polymerization is a crucial factor for obtaining optimal physical and clinical performance of any hybrid composite resin.[1] The degree of monomer conversion is considered crucial to the clinical success of composite resins,[2] and is related to numerous physical properties.[3]

Many are the polymerization modes used for photocuring composite resin, involving different radiant exposure times and protocols. Quartz–tungsten–halogen (QTH) and light-emitting diodes (LEDs) are still the most common light-curing units used to convert the monomer molecules into a polymer network. However, different polymerization modes can affect the polymer's internal structures, degree of conversion (DC), and cross-link density. These three factors may affect the sorption degree of the composite resin, leading to color alteration of the restorative material.

Photoinitiators, such as camphorquinone (CQ), the most commonly used for resin polymerization, can also result in such color alteration. CQ has a particularly bright, yellowish color that influences the final color of the polymerized material.[4,5] Amine, another substance found in composite resins, can also alter the final color of the restorative material, resulting in a yellowish to brownish-red color.

Alteration in the final color of the restorative material might affect the longevity of the restoration.[6] Color alteration in resin-based materials may be caused by intrinsic and extrinsic factors. The intrinsic factors involve the photoinitiator system, the composition of the resin matrix, the filler loading and particle size distribution, the light-curing device, the irradiation exposure time, and the degree of remaining $C=C$ bonds (% remaining double bonds [RDBs]).[7–9]

Extrinsic factors are related to substances and conditions such as water, foods, dyes, Ultraviolet radiation, heat, smoking, and oral hygiene habits.[5,8–10] Therefore, the quality of the polymer and the DC of the resin are important factors that might be associated with the resin color alteration[9,10] and the residual yellowing observed in photocured restorations.[4,8] Studies have suggested that color alteration in restorative materials should be

$\Delta E > 1$ to be visually perceptible, and that $\Delta E > 3.3$ would be considered an undesired value for clinical success of the restoration.[11–13]

The present study was aimed at evaluating the influence of different light-curing modes on the DC and staining of a nanofilled composite resin, using different beverages. It was hypothesized that: (1) different polymerization modes with different energy sources could lead to different degrees of conversion, (2) different polymerization modes could alter the staining susceptibility of the restorative material and its stain retention, and (3) there might be a correlation between its staining susceptibility and stain retention.

MATERIALS AND METHODS

A nanofilled composite resin (Z350, 3M ESPE Dental Products, St. Paul, MN, USA) was used to prepare 200 cylindrical specimens in Teflon ring molds, 5.0 mm in diameter and 2.0 mm in thickness, held between two glass slabs separated by Mylar matrix strips, and then pressed with a 500-g load. Cavities were filled in using one-increment restorative material, which was randomly polymerized according to the four polymerization modes ($N = 50$): QTH at 450 mW/cm^2 for 20 seconds (XL 3000, 3M ESPE, Grafenau, Germany); laser (LAS) at 300 mW/cm^2 for 20 seconds (AccuCure 3000, LaserMed, West Jordan, UT, USA); second-generation light-emitting diode (LED 2) at 1,100 mW/cm^2 for 20 seconds (Bluephase 16i, Vivadent, Bürs, Austria); third generation LED (LED 3) at 700 mW/cm^2 for 20 seconds (Ultralume LED 5, Ultradent, South Jordan, UT, USA). The irradiance (Table 1) was measured with Demetron Research Corporation model 100, serial 105415 (Kerr Corporation, Orange, CA, USA). All specimens were stored in light-proof containers at 37°C for 24 hours and the top surface of each specimen was polished with flexible aluminum oxide disks (Sof-Lex Pop-on®, 3M ESPE) under water spray.

DC

Twelve specimens of each experimental group were randomly submitted to the DC test. To measure the absorption spectra of the uncured material, resin was inserted in a circular metallic matrix (7 mm in diameter and 1 mm in thickness), covered with a polyester strip, and held together between two glass plates under a weight of 10 kgf. A spectrometer (Nexus 670 FT-IR, Nicolet Instrument Corp., Madison, WI, USA), operating with 64 scans at a resolution of 4 cm^{-1} and a wavelength of 4,800–4,500 cm^{-1}, was used to obtain the absorption spectra for both the polymerized and the uncured composite resins.

To determine the DC, the monomer was converted into polymer and the absorbance intensity of the stretching band of the carbon-to-carbon double bonds ($C = C$) of the methacrylate (4,743 cm^{-1}) was measured using a spectrometer; the reduction in the absorbance intensity was then recorded.

TABLE 1. IRRADIANCE AND RADIANT EXPOSURE OF THE LIGHT-CURING UNITS USED.

Light-curing unit	Manufacturer	Code	Irradiance (mW/cm^2)	Radiance exposure (J/cm^2)
Bluephase 16i	Vivadent (Bürs, Austria)	LED 2	1,100	22
Ultra-Lume LED 5	Ultradent (South Jordan, UT, USA)	LED 3	700	14
XL 3000	3M ESPE (Grafenau, Germany)	QTH	450	9
AccuCure 300	LaserMed (West Jordan, UT, USA)	LAS	300	6

This spectroscopic procedure depends on an absorption band that is not modified with the polymerization and serves as a spectrum normalization of both the monomer and the polymer. For example, composite resins that have aromatic monomers with an absorption band of 4,623 cm^{-1} can serve as internal standards of normalization, regardless of the thickness of the sample.[14]

The DC value was calculated by standard methods using changes in the ratios of aliphatic-to-aromatic $C = C$ absorption peaks in the uncured and cured states obtained from the infrared spectra.[14]

Superficial color measurement

The baseline color of all specimens were measured with a reflection spectrophotometer (model UV-2450, Shimadzu, Kyoto, Japan) using the *Commission Internacionale de I'Eclairaga* (CIE) L^* a^* b^* system, according to the CIE. This color system consists of two axes, which have right angles and represent the dimension of tone or color (a^*: red-green content; b^*: yellow-blue content) and another, the third axis, represented by the letter L^*, perpendicular to a^* b^*, referring to brightness.

After the baseline measurement was taken, all specimens were submitted to the staining process and then measured again using the same method described earlier. Changes in overall color (ΔE) were calculated as follows:

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

Staining

Specimens concerning each polymerization mode were randomly assigned to five staining groups ($N = 10$): distilled water; red wine; whisky; coffee; and cola (Table 2). Each specimen was immersed in the staining solution for 3 hours/day at room temperature, for 40 days.[15] Only the top surface of each specimen was in contact with the staining solution as the other surfaces (bottom and lateral) were protected with two coats of a cyanoacrylate adhesive (Superbond, Henkel Loctite Adhesives, Ltda., Itapevi-SP, Brazil). Each specimen was immersed in separated vials containing 50 mL of each staining solution. The vials were sealed with parafilm to prevent evaporation. After each staining period, the specimens were gently rinsed with distilled water, air-dried, and stored in distilled water at 37°C.[15]

Stain retention measurement

Each specimen (red wine, whisky, coffee, and cola) was weighed and ground into powder using a mill (Marconi Equip. Ltda., Piracicaba, SP, Brazil). The resulting powder was weighed again, and the difference between the initial and final weight should not be greater than 10% to be included in the present study. To dilute the stain, the powder from each specimen was individually immersed in a glass tube containing 4 mL of absolute PA alcohol for 24 hours. Next, the solutions were centrifuged (Tomy–IC 15AN, Tomy Ind., Tokyo, Japan) at 3,000 rpm for

TABLE 2. THE STAINING SOLUTIONS USED.

Staining solution	Presentation	Brand
Coffee	8 g of ground coffee were dissolved in 100 mL of boiling distilled water	Café Pilão, União, São Paulo, SP, Brazil
Red Wine	Bottle with 750 mL, harvest 2003	Reserva, Cabernet Sauvignon, Miolo Wine Group, RS, Brazil
Whisky	Bottle with 1 L	Red Label, Jonnie Walker
Cola	Bottle with 600 mL	Coca Cola, Co.
Distilled water	—	—

3 min. The supernatant (floating solution) was analyzed using a spectrophotometer (Beckman DU-65, Instruments, Inc., Fullerton, CA, USA) adjusted to a measurement wavelength, which varied according to each staining agent (Table 2).

The wine, whisky, and cola solutions were separately submitted to the vacuum rotary evaporator to remove alcohol and water from the solution, leaving only the pigment, which was added to the alcohol to prepare solutions with known concentrations. To calibrate the spectrophotometer, the absorbance values concerning each standard solution (red wine, whisky, coffee, and cola), at different concentrations (2, 4, 6, 10, 20 μg/mL), were determined at wavelengths ranging from 200 to 700 nm. The highest wavelength value was considered to measure the stain retention and the absorbencies for the standard solutions. Based on these values, a coefficient of linear correlation and a straight-line equation ($y = a + bx$) were determined (Table 2). To measure the dye concentration (μg/mL) in the experimental samples, the "y" was changed for the absorbency value of each specimen (Table 3).

Data were submitted to two-way analysis of variance and Tukey test, at a significance level of 0.05.

RESULTS

DC values are shown in Table 4. The QTH polymerization mode showed the lowest DC values when compared with LAS, LED 2, and LED 3. No statistically significant difference was observed among the modes.

Table 5 shows mean values of stain retention. In general, whisky and wine showed lower pigment concentrations than cola and coffee. No statistical difference was observed for the light-curing modes concerning all staining solutions, except for cola, showing the highest pigment concentration when QTH was used.

Table 6 shows mean values for color alteration (ΔE) regarding all experimental groups. Whisky showed the highest ΔE values, considering all light-curing modes. Wine and water showed statistically lower ΔE values than whisky, without differences when compared with cola and coffee. When the light-curing modes were analyzed, LAS and QTH showed the highest ΔE values. LED 3 showed intermediary results; no statistical differences were found among the groups tested.

DISCUSSION

The first hypothesis was accepted. There were statistical differences

TABLE 3. THE REGRESSION EQUATION, COEFFICIENTS OF CORRELATION, AND WAVELENGTHS OF THE STAINING SOLUTIONS USED.

Staining solution	Regression equation	Coefficients of correlation (r)	Wavelengths
Coffee	$y = 0.0304x + 0.2634$	0.9798	288
Red wine	$y = 0.0454x + 0.3323$	0.9845	294
Whisky	$y = 0.035x + 0.2147$	0.9342	288
Cola	$y = 0.0071x + 0.3191$	0.9556	287

TABLE 4. MEANS VALUES (STANDARD DEVIATIONS) OF DEGREE OF CONVERSION OF THE EXPERIMENTAL GROUPS.

Groups of polymerization mode	Degree of conversion % (standard deviation)
Second generation LED	76.96 (0.53) A
Third generation LED	77.01 (1.25) A
Laser	76.84 (0.86) A
Quartz–tungsten–halogen	75.63 (1.13) B

Groups with the same lower letter were not statistically different ($p = 0.05$).

TABLE 5. MEANS VALUES (STANDARD DEVIATIONS) FOR STAIN RETENTION CONSIDERING ALL GROUPS.

Staining solutions	Polymerization modes			
	LED 3	LAS	QTH	LED 2
Whisky	07.06 (1.79)Ab	04.77 (1.81)Ac	5.74 (1.62) Ac	5.07 (2.01)Ab
Cola	25.92 (6.79) Ba	12.18 (7.71)Cb	44.89 (11.25)Aa	25.23 (6.81) Ba
Red Wine	07.59 (2.29)Ab	05.93 (1.40)Ac	9.49 (1.79) Ac	8.59 (1.56)Ab
Coffee	21.10 (3.45)Aa	17.24 (3.05)Aa	23.07 (2.50) Ab	19.89 (2.30)Aa

Groups with same lower case letter were not statistically different for the same staining solution ($p = 0.05$). Groups with same upper case letter were not statistically different for the same polymerization mode ($p = 0.05$).

TABLE 6. MEANS VALUES (STANDARD DEVIATIONS) OF COLOR CHANGE VALUES (ΔE_{AB}) OF THE EXPERIMENTAL GROUPS.

Staining solutions	Polymerization modes				
	LED 3	LAS	QTH	LED 2	
Whisky	1.36 (0.30)	1.39 (0.30)	1.41 (0.31)	1.28 (0.22)	a
Cola	0.88 (0.11)	1.02 (0.24)	0.99 (0.17)	0.87 (0.21)	bc
Water	0.84 (0.18)	0.92 (0.11)	0.98 (0.21)	0.83 (0.13)	c
Red Wine	0.97 (0.24)	1.12 (0.33)	1.16 (0.30)	1.02 (0.21)	b
Coffee	0.91 (0.16)	0.96 (0.22)	1.03 (0.15)	0.93 (0.19)	bc
	AB	A	A	B	

Groups with same lower case letter were not statistically different for the same staining solution ($p = 0.05$). Groups with same upper case letter were not statistically different for the same polymerization mode ($p = 0.05$).

among the light-curing modes concerning DC. QHT showed lower DC mean values when compared with LAS, LED 2, and LED 3. The radiant energy used for QTH was 9 J/cm^2, lower than that used for LED 2 (22 J/cm^2) and LED 3 (14 J/cm^2). Calheiros and colleagues,[16] measuring the DC of a composite resin light cured with different irradiance values, reported that DC increased according to the radiant exposure.

LED devices have a narrow spectral range with a peak around 470 nm, which matches the optimum absorption wavelength for the activation of the CQ photoinitiator, increasing the DC of the composite resin.

There was no statistically significant difference among the LED groups, considering all irradiance values. Such factors as the light polymerization mode, the high irradiance values, and the spectral range coincident with the optimum absorption wavelength for the activation of the CQ might have contributed to the improvement in the DC observed in the present study.

In a previous study, da Silva and colleagues[17] reported that polymerization modes with high initial irradiance values might lead to an increase in the exothermic heat and, consequently, in the mobility of the polymer chains, improving the DC.

Although both LED groups presented high irradiance values, no statistically significant difference was observed between LEDs and LAS. LAS used an irradiance value of 6.0 J/cm^2, lower than those used in the other groups; however, this unit might increase the temperature of the composite resin, accelerating the polymerization.[18]

Different polymerization modes might also alter the staining susceptibility[5] and stain retention of the composite resin. However, the stain retention is also dependent on the staining solution; in the present study, only one solution (cola) showed statistically significant differences considering polymerization modes. Therefore, the second hypothesis was partially accepted. Polymerization modes using a relatively low irradiance level showed the highest superficial staining values for all solutions tested. Clinically, it can be inferred that it would be wise to repair or replace restorations with polymerization modes using low irradiance.

Two laboratory methods were used to evaluate the staining susceptibility and stain retention of the resin. A reflection spectrophotometer (CIE L^* a^* b^* system) was used to measure the ΔE of the composite resin. An absorbance spectrophotometer was used to evaluate the amount of pigment absorbed by the composite resin, including its surface portion.

Extrinsic discoloration has been attributed mainly to the absorption of pigment from exogenous sources.[7] In the present study, no correlation was found between stain retention and staining susceptibility. Specimens stained with cola and coffee showed significantly higher pigment concentrations than those stained with whisky and wine; however, whisky showed the highest discoloration, followed by wine. Cola and coffee did not differ from water (control) and wine with regard to staining susceptibility. Although only the whisky and red wine staining solutions caused a visually perceptible ΔE ($\Delta E > 1$), all experimental groups presented a clinically acceptable ΔE ($\Delta E < 3.3$).[11–13] Thus, the third hypothesis was rejected.

The amount of pigment absorbed by the composite resin tested, considering all staining solutions, might be related to the water absorption rate, the content of resin monomer and filler particles, the quality of the polymer, and the type of staining agent.[7,19,20] Coffee and cola showed the highest pigment concentrations. Coffee contains yellow colorants with different polarities;[20] therefore, its pigment is absorbed by the composite resin and then adheres to the polymer due to its lower polarity components.[20] Cola, having some different properties, consists of sugar and caramel, substances containing a sticky effect[19] that increases its adsorption degree. Although cola and coffee did not present high staining rates, the high concentration of pigment long-term can result in replacement of the restoration without allowing more conservative procedures, such as finishing/polishing procedure. Red wine and whisky presented lower stain retention and higher staining rates. Unlike coffee and cola, red wine and whisky are alcoholic beverages. Alcohol causes the composite resin surface to soften by removing its polymer structure such as unreacted monomers, oligomers, and linear polymers,[21] facilitating the absorption of pigment agents and increasing wear.[22–24] In a composite resin, having the same monomer DC, the resin surface dissolution by ethanol might be more selective in a relatively linear polymer than in one that is expected to have a more cross-linked structure. Aguiar and colleagues,[25] using alcoholic and aqueous solutions to test the susceptibility of restorative materials to staining, showed higher staining mean values for alcoholic than aqueous solutions. Other authors, comparing red wine with coffee, water, and cola, reported that the alcoholic solutions caused more discoloration than the non-alcoholic ones.[12,19]

However, this effect may have occurred superficially, as the pigment was concentrated only on the surface of the specimens. Alcohol might affect only the surface of the resin because its subsurface contains water, which is absorbed during the staining process (3 hours in staining solution/21 hours in distilled water daily). Therefore, the alcoholic solution was ineffective in removing the monomers and oligomers of the resin subsurface, avoiding staining of the deep layers of the specimens. Bagheri and colleagues[20] stated that a 1-week pre-immersion of specimens in distilled water was able to reduce to staining potential of the food-simulating solutions. Further studies are needed to investigate the surface topography and roughness of the composite resin after its immersion in alcoholic media as well as to verify whether a finishing/polishing procedure is sufficient to remove the superficial staining from the material.

In the present study, whisky showed significantly higher staining rates than those observed for the red wine. The whisky (39 GL) tested had a higher alcohol volume than that of the red wine (12.8 GL), suggesting that the concentration of alcohol might have contributed to the high discoloration rates found.

The staining rates found might be related to the composite resin's affinity for stains and water sorption due to its low monomer DC as well as the quality of the polymer.[26] Water sorption is related to the degradation and type of polymer.[27]

The light intensity and polymerization time can modify the polymer structure formation.[21] Asmussen and Peutzfeldt[21] reported that polymerization with pulse delay starts in the center of the polymer, and, consequently, propagation will predominantly add one molecule of monomer after another to a growing polymer chain. This results in a more linear oligomer or polymer structure with relatively few cross-links. The final cure will activate a large part of the remaining CQ and thus give rise to a branched polymer.

The authors of the above-mentioned study credit the polymer structures formed with the pulse delay technique to the softness of the resin surface after being immersed in ethanol, making it more susceptible to staining. The results of the present study corroborate this statement.

In the present study, QTH and LAS modes showed the highest discoloration mean values for all solutions. These modes presented the lowest irradiance values and, consequently, the lowest energy density, resulting in a more linear polymer structure and highest staining rates. In contrast, LED 2 presented the highest values for irradiance and energy density, and the lowest staining rates. Yazici and colleagues[28] studied the influence of two different light-curing units (LED and QTH) on the staining of composite resin and found no statistical differences between the polymerization modes. The authors attributed this result to the similar radiant energy emitted by the light-curing units.

CONCLUSION

Within the limits of this study, it can be concluded that different polymerization modes with different radiant exposure could alter the composite resin's stain retention and its staining susceptibility to different staining solutions. The highest pigment absorption was associated with neither the highest superficial staining of the composite resin nor the DC. Alcoholic drinks might lead to higher superficial staining and non-alcoholic solutions to higher pigment concentration.

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