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RESEARCH AND EDUCATION

The influence of simulated aging on the color stability of composite resin cements

Nijad R. Mina, DDS, MSc,[a] Nadim Z. Baba, DMD, MSD,[b] Fahad A. Al-Harbi, BDS, MSD, DScD,[c] Moataz F. Elgezawi, BDS, MDSc, DDSc,[d] and Maha Daou, DDS, MS, PhD[e]

ABSTRACT

Statement of problem. Data for the color stability of dual-polymerized and light-polymerized resin cements used in esthetic dentistry are lacking.

Purpose. The purpose of this in vitro study was to evaluate the color stability of 4 types of composite resin cements after water aging.

Material and methods. Specimens (n=30) of each resin cement (Variolink Esthetic LC, RelyX Ultimate DC, Nexus 3 DC, Nexus 3 LC) were prepared. The shade selected was Light+ for Variolink Esthetic, B 0.5 for RelyX Ultimate, and White for both Nexus 3 DC and LC. All 120 specimens were aged by water for 30 days at 37°C under dark conditions, using a thermocycling machine. The specimens' color characteristics (L*, luminosity; a*, red-green; b*, yellow-blue) and color differences (ΔE) were measured with a spectrophotometer before day 0, after day 1, and after 30 days of immersion. Statistical analysis used ANOVA and Tukey post hoc tests (α=.05).

Results. Considering ΔE<3.3 as clinically acceptable, results showed significant color variations for all cements (RelyX Ultimate=3.69; Nexus 3 LC=3.76; Nexus 3 DC=5.34), except for Variolink Esthetic (0.88). However, this variation was significantly less when day 1 was considered the baseline measurement, showing clinically acceptable ΔE values for all types of cement.

Conclusions. Water aging had a significant effect on color stability; most color variations occurred in the first 24 hours of polymerization, with relatively nonsignificant variations afterwards. (J Prosthet Dent 2019;121:306-10)

One of the major challenges in dentistry is to recreate the optical properties of natural dentin and enamel using dental materials.[1] Ceramic restorations are popular because of their esthetic appearance,[2] and when used properly, these materials can provide a natural-looking restoration.[1]

More conservative tooth preparation and improvements in ceramic technology have led to the fabrication of thinner restorations with increased translucency. With current ceramic materials, the thickness of laminate veneers can range between 0.3 and 0.7 mm.[3] Factors that affect the esthetics of veneer restorations include the translucency and opacity of the ceramic material being used, the shade of the luting cement, and the shade of the underlying tooth structure.[1,4,5] For machinable lithium disilicate, shade matching is affected by both the shade and thickness of the luting cement.[6] Thus, the color stability of the luting cement is essential to achieve long-term esthetics with lithium disilicate restorations.[2,5,7,8]

The color of autopolymerizing and dual-polymerizing composite resin cements, both before and after polymerization, is more yellow than that of light-polymerizing composite resin cement, and the color change on polymerization is greater.[9] Viohl et al[10] attributed discoloration of composite resin cements to reactive chemical groups in amine accelerators and inhibitors, such as peroxide and tertiary aromatic amines. In autopolymerizing and dual-polymerizing composite resin cements, the shade change in the cement may be precipitated by the oxidation of these reactive chemical groups, which produces colored oxygen byproducts. As

[a]Private practice, Magrabi Hospital, Al-Khobar, Saudi Arabia.
[b]Professor, Advanced Specialty Education Program in Prosthodontics, Loma Linda School of Dentistry, Loma Linda, Calif.
[c]Professor and Dean, Department of Substitutive Dental Sciences, Imam Abdulrahman Bin Faisal University, Dammam, Saudi Arabia.
[d]Professor, Department of Substitutive Dental Sciences, College of Dentistry, Imam Abdulrahman Bin Faisal University, Dammam, Saudi Arabia.
[e]Director of Research, Department of Biomaterials, Saint Joseph University, Beirut, Lebanon.

Clinical Implications

Resin cements are not yet completely reliable in terms of color stability. Not all light-polymerized resin cements exhibit better color stability than dual-polymerized cements.

the amine inhibitors oxidize over time, the hue of the cement changes and discolors, causing a yellow color shift that could affect the long-term esthetics of the restoration.[2,10]

Visible light-polymerized composite resin cements are recommended for luting thin, translucent ceramic veneers because of their color stability and extended working time.[3] Their color stability is related to their amine accelerators. In light-polymerizing composite resin cements, the amine accelerators are aliphatic, which means they oxidize and discolor to a much lesser degree than tertiary amine accelerators.[2,9]

Despite the color change, the use of dual-polymerizing composite resin cements is still of interest because of their improved mechanical properties and the chemical polymerization, which is useful where direct light polymerization is difficult.[3] Development of a color-stable, dual-polymerizing resin cement with a lighter initial color and less dramatic color shift has been reported.[9]

The use of laminate veneers with minimal or no tooth preparation has been advocated but poses its own challenges.[11] This type of restoration exposes the resin cement to the oral environment, causing intrinsic and extrinsic discoloration.[12] Thus, color-stable resin cements must also withstand conditions in the oral cavity.

Aging of resin-based materials has been assessed using prolonged water storage and exposure to ultraviolet light.[3] To simplify the aging procedure, water has been separately analyzed as a factor in the optical alteration of dental resin-based restorative materials.[5,9,13-18] Whether color changes should be assessed by starting baseline measurements after 1 day of water storage[2,5,17,19,20] or immediately after specimen preparation (before any water immersion) is unclear.[9,14-16,18,21-24] Typically, the same shade of resin cements has been studied,[2,16,23,25,26] as color stability is affected by the resin shade[8,14,17,25,27-29] and composition.[3,14,18,24,29-32] Color change is usually measured and quantified by means of the CIELab system established by the Commission International de l'Eclairage.[1,5,8,29,30,33,34]

The purpose of this in vitro study was to evaluate the color stability of different types of composite resin cements after water aging tests with 2 different baselines of color measurements. The null hypotheses tested were that no significant differences in color would be found before and after aging of composite resin cements and that the color measurement baseline would have no significant effect on color variations.

Table 1. Composite resin cements materials tested

Composite Resin Cements	Manufacturer	Lot	Shade
Nexus 3 DC	Kerr Corp	4872926/4981767/ 5017603	White
Nexus 3 LC	Kerr Corp	4992589	White
Variolink Esthetic LC	Ivoclar Vivadent AG	V44691	Light+
RelyX Ultimate DC	3M ESPE	531904/567731	B 0.5

MATERIAL AND METHODS

A total of 120 square-shaped specimens were fabricated from 4 different types of composite resin cements (Table 1). Translucent cellulose sheets (Essix; Dentsply Sirona) were cut into 10×10×1-mm squares and embedded in polyvinyl siloxane putty (I-Sil; Spident Co Ltd) to form molds. For each type of composite resin cement, the molds were filled and covered with a polyester resin strip (Mylar; Henry Schein) and a glass slab and light polymerized for 20 seconds by using a light-emitting diode (LED) polymerizing unit (Satelec; Acteon) with a mean light intensity of 1250 mW/cm^2 (previously checked by radiometer [Bluephase; Ivoclar Vivadent AG]). The glass slab was removed, and direct light polymerizing of the composite resin cement was completed for 40 seconds (total of 60 seconds per specimen).

A digital caliper (Electronic Digital Caliper) was used to measure all specimens to ensure standardization. The glossy surface of the specimens was used as the reference. If the thickness of the specimen needed adjustment, the material was reduced on the nonglossy side with polishing disks (OptraFine; Ivoclar Vivadent AG). For each group, the specimens were numbered from 1 to 30 and stored in the dark.

Specimens were artificially aged in distilled water at 37°C for 30 consecutive days. Color measurements were made by using a spectrophotometer (Color-Eye 7000A; Gretag Macbeth) before and after each aging test. A black-colored light trap and a white tile provided by the manufacturer were used to calibrate the spectrophotometer before each study group. Values were measured according to the CIELab system: L* (lightness, "100" represents white and "0" represents black), a* (red-green chromatic coordinate), and b* (blue-yellow chromatic coordinate).

For the water aging test, baseline measurements (day 0) were made for each group of specimens, followed by other measurements after 1 day and 30 days of water aging. CIELab values were compared by using the formula: $\Delta E=[(\Delta L)^2+(\Delta a)^2+(\Delta b)^2]$ ½.

Statistical analysis was performed by using a software program (PASW Statistics v18.0; SPSS Inc) (α=.05). The primary outcome variable of the study was the ΔE (day

Table 2. Tukey pairwise comparisons in each group of resin cements

Factor	Mean
Nexus3 DC (n=30)	
NX3DC ΔE0/30	5.344[A]
NX3DC ΔE0/1	4.466[A]
NX3DC ΔE1/30	1.552[B]
Nexus3 LC (n=30)	
NX3LC ΔE0/30	3.762[A]
NX3LC ΔE0/1	2.886[A]
NX3LC ΔE1/30	1.816[B]
RelyX Ultimate (n=30)	
RlxUlt ΔE0/30	3.694[A]
RlxUlt ΔE1/30	2.786[B]
RlxUlt ΔE0/1	1.124[C]
Variolink Esthetic (n=30)	
VlkLC ΔE0/30	0.88[A]
VlkLC ΔE1/30	0.715[A,B]
VlkLC ΔE0/1	0.514[B]

Mean values with different uppercase letters indicate significant differences ($P<.05$).

0 to day 30) values. The variable was tested for normal distribution using the Kolmogorov-Smirnov test. Analyses of variance followed by Tukey post hoc tests were used to compare the mean ΔE (day 0 to day 30) values and ΔE (day 1 to day 30) among all 4 types of composite resin cements.

RESULTS

When day 0 of water storage was considered as baseline for color measurement, all materials showed ΔE>3.3, except for Variolink Esthetic. The mean ΔE (day 0 to day 30) was 3.76 Nexus3 LC, 5.34 for Nexus3 DC, 3.69 for RelyX Ultimate, and 0.88 for Variolink Esthetic. Color variations of Nexus3 DC were the highest (*P*<.05), whereas color variations of Variolink Esthetic LC were the lowest (*P*<.05) (Table 2). In the LC groups, Variolink Esthetic showed better color stability than Nexus3 LC. In the DC groups, RelyX Ultimate showed better color stability than Nexus3 DC (*P*<.05) (Fig. 1).

When day 1 of water storage was considered as baseline for color measurement, no clinically significant changes were detected in ΔE (day 1 to day 30) for all materials used. ΔE was less than 3.3 for all 4 types of resin cements tested. The mean ΔE (day 1 to day 30) was 1.81 for Nexus3 LC, 1.55 for Nexus3 DC, 2.78 for RelyX Ultimate, and 0.71 for Variolink Esthetic. Color variations of Variolink Esthetic were the lowest, whereas color variations of RelyX Ultimate were the highest.

In the DC groups, Nexus3 showed better color stability than RelyX Ultimate, whereas in the LC groups, Variolink Esthetic showed better color stability than Nexus3 LC. The Tukey test for comparison of ΔE day 0 to day 30 to ΔE day 1 to day 30 showed that mean ΔE values at day 0 to day 30 were significantly different than the mean ΔE values at day 1 to day 30 with Nexus 3 DC (*P*<.001), Nexus 3 LC (*P*<.001), RelyX Ultimate (*P*<.001), except for Variolink Esthetic (Table 2).

DISCUSSION

The null hypothesis that no significant differences in color would be found with water storage or with different color baselines was rejected, as the results showed significant impact of water aging and different baselines choices (1 day difference) on the color stability of most of the resin cements tested.

Composite resin cements have an influence on the esthetic outcome of ceramic restorations[2,8] and have improved to meet esthetic requirements, including their color stability.[9] Janda et al[35] examined the consequence of the constant or exponential polymerization mode of a halogen light-polymerizing device on the color stability of resin-based restorative materials. They concluded that the extent of discoloration depends on the polymerizing time, polymerizing mode, aging condition, and material.

When a LED is used to polymerize the specimens, color change is less than with a conventional halogen unit[36] or a photo-polymerizing oven system.[37] LED polymerizing units emit a narrow wavelength, achieving a high absorption rate of camphorquinone, and the degree of conversion depends on the LED energy density. When polymerizing composite resin using an LED light, color stability depends on the polymerizing mode, polymerizing time, and aging condition.[8,21]

A spectrophotometer was used in this study to measure color. Spectrophotometers have replaced colorimeters for the measurement of transmission (transparent object) and reflectance (opaque object) because the color measurements are more accurate.[1,4,8,29]

When the CIELab system is used to measure color changes,[1,5,8,17-20] different ΔE values have been used for clinical unacceptability. These values have included ΔE>3.7[38] and ΔE>3.[3] Sabatini et al[23] considered ΔE>3.3 as the threshold most applied as the indicator for clinical unacceptability of color change.[8,9,13,34,39]

In the current study, the spectrophotometer recorded the color of the specimens placed against a black background and exposed to a standard light source. Similar to the studies of Kilinc et al,[2] Diamantopoulou et al,[16] and Ural et al,[25] this study used specimens that were 1 mm thick (according to International Organization for Standardization standard 7491[40]), and the spectrophotometer requires a certain specimen thickness for reliable results.[2]

Different methods have been used to model clinical aging.[9,15,16] Storing the specimen in water under dark conditions for 1 day at 37°C before baseline measurements has been recommended.[2,5,17] Other studies have started the baseline measurements before any water storage.[9,14,15,21] Lepri et al[19] stored specimens for 1 day in

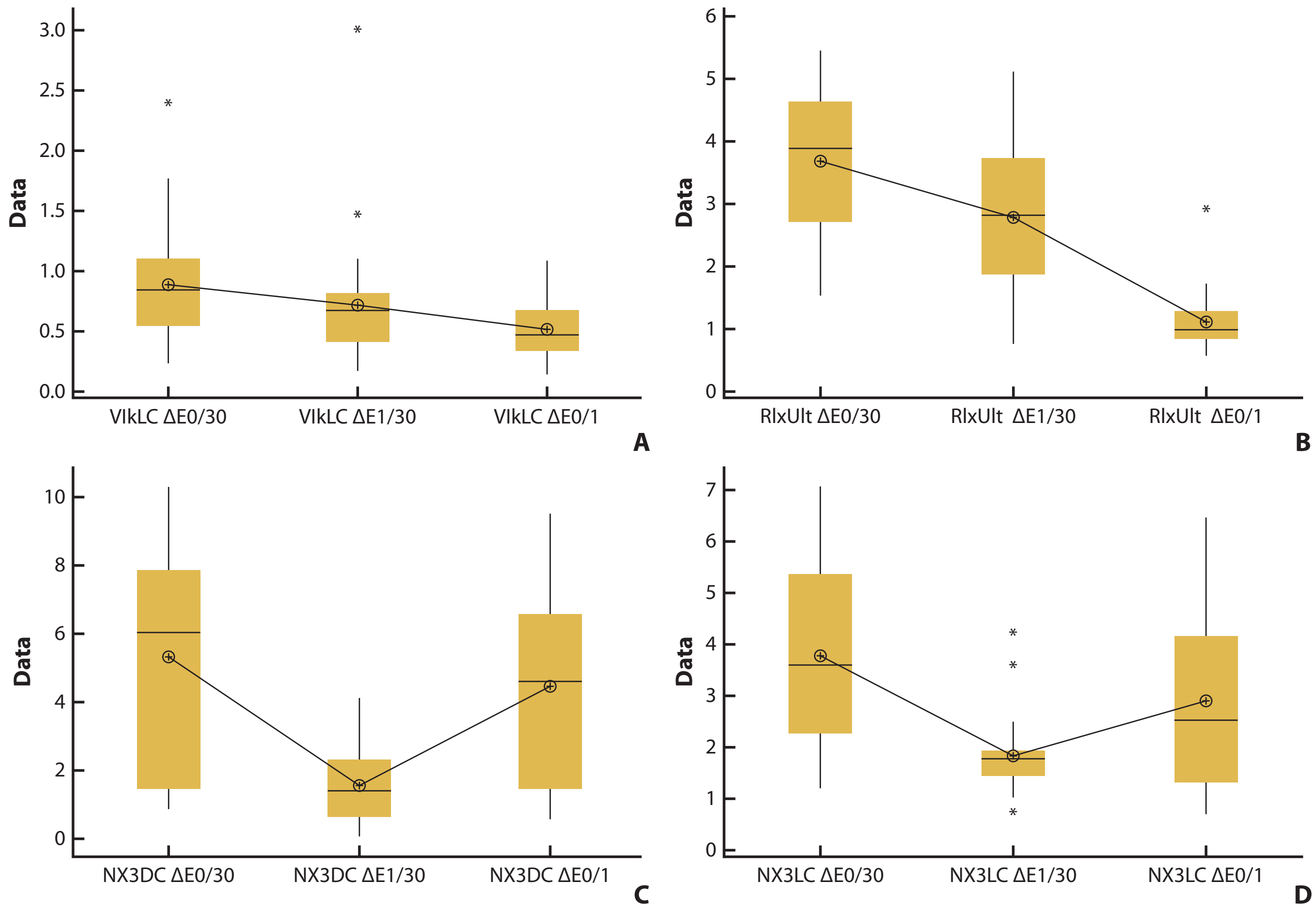

Figure 1. Color changes (ΔE) based on different baseline measurements. A, Variolink Esthetic LC. B, RelyX Ultimate DC. C, Nexus3 DC. D, Nexus3 LC.

artificial saliva at 37°C, whereas Samra et al[22] stored them for 1 hour before any experiment. In the current study, measurements were made before and after 1 day of storage at 37°C under dark conditions to determine how the color variation was affected by the related baseline.

The lightest shade of each type of resin cement was tested: the white shade for both Nexus3 LC and DC, the B0.5 shade for RelyX Ultimate, and the Light+ shade for Variolink Esthetic LC. This is consistent with studies using the same shade category for different types of resin cement and dividing the resin cement specimens into different groups depending on the lightest, middle, and darkest shades.[2,23,25-27]

Results of the current study showed that ΔE (day 0 to day 30) had significant color changes after water aging, with unacceptable values for Nexus3 LC and DC and RelyX Ultimate composite resin cements. These results are consistent with those of other studies,[5,13] which concluded that water aging can cause color change when the stability of dual polymerizing composite resin cements, composite resin, flowable composite resins, and composite resins combined with adhesives is tested.

For the DC groups, the ΔE (day 0 to day 30) values of RelyX Ultimate showed better color stability than Nexus LC and DC, which had a similar white shade color, although both were above the acceptable ΔE (3.3).

As for the LC groups, the ΔE (day 0 to day 30) values of Variolink Esthetic showed better color stability than Nexus3 LC and were the only values under the clinically acceptable ΔE (3.3). Results of this study are consistent with those of Archegas et al[3] and Berrong et al.[15] Most of the related studies attributed the color stability of resins to their resin matrix and filler compositions.[3,14,24,29-32] Boaro et al[14] and Vichi et al[30] found that the smaller the particles, the lower the water aging susceptibility. Schneider et al[41] concluded that a matrix with lower water solubility, such as silorane, was related to hardness but not necessarily to color stability.

Color change has been reported to be material- and shade-dependent.[17,24] Other investigators have concluded that lighter shades of composite resins are likely to exhibit higher color degeneration after a short period of water storage.[14,27,28,33] When the baseline of color measurement was considered after 24 hours of water storage, all ΔE (day 1 to day 30) color variations were less than the clinically acceptable level of ΔE (3.3). Nexus3 DC color variations went from being the highest value to the second lowest value after Variolink Esthetic;

the latter remained the most color stable material regardless of the baseline considered. This finding is consistent with those of Smith et al[17] and Ural et al.[25]

When color changes were compared after 24 hours and after 1 month, the results of this study were similar to the results of Sabatini et al,[14] who found that most of the clinically relevant color changes occurred after initial polymerization and 24 hours later. After 24 hours, color changes were found to be negligible.[14,22,42]

Limitations of the current study included use of a single aging and color assessment protocol and use of luting cement specimens that were much thicker than the luting cement film thickness beneath laminate veneers in clinical situations.

CONCLUSIONS

Within the limitation of this in vitro study, the following conclusions were drawn:

1. All composite resin cements tested showed significant changes in color after 30 days in water storage, except for Variolink Esthetic LC.
2. Less color change was found for Variolink Esthetic LC and RelyX Ultimate DC than for Nexus3 LC and Nexus3 DC of the same mode and shade opacity.
3. The most color changes occurred in the first 24 hours of polymerization, whereas color changes afterward remained relatively negligible.

REFERENCES

1. Turgut S, Bagis B, Turkaslan SS, Bagis YH. Effect of ultraviolet aging on translucency of resin-cemented ceramic veneers: an in vitro study. J Prosthodont 2014;23:39-44.
2. Kilinc E, Antonson SA, Hardigan PC, Kesercioglu A. Resin cement color stability and its influence on the final shade of all-ceramics. J Dent 2011;39 suppl 1:30-6.
3. Archegas LR, Freire A, Vieira S, Caldas DB, Souza EM. Color stability and opacity of resin cements and flowable composites for ceramic veneer luting after accelerated aging. J Dent 2011;39:804-10.
4. Xing W, Jiang T, Ma X, Liang S, Wang Z, Sa Y, et al. Evaluation of the esthetic effect of resin cements and try-in pastes on ceromer veneers. J Dent 2010;38 suppl 2:87-94.
5. Gaintantzopoulou M, Kakaboura A, Loukidis M, Vougiouklakis G. A study on colour stability of self-etching and etch-and-rinse adhesives. J Dent 2009;37:390-6.
6. Niu E, Agustin M, Douglas RD. Color match of machinable lithium disilicate ceramics: Effects of cement color and thickness. J Prosthet Dent 2014;111:42-50.
7. Azer SS, Rosenstiel SF, Seghi RR, Johnston WM. Effect of substrate shades on the color of ceramic laminate veneers. J Prosthet Dent 2011;106:179-83.
8. Ghavam M, Amani-Tehran M, Saffarpour M. Effect of accelerated aging on the color and opacity of resin cements. Oper Dent 2010;35:605-9.
9. Oei JD, Mishriky M, Barghi N, Rawls HR, Cardenas HL, Aguirre R, et al. Development of a low-color, color stable, dual dental resin. Dent Mater 2013;29:405-12.
10. Viohl J. Color stability of dental resins. Quintessence Int 1980;3:71-8.
11. Gürel G. Porcelain laminate veneers: minimal tooth preparation by design. Dent Clin North Am 2007;51:419-31.
12. Shiozawa M, Takahashi H, Asakawa Y, Iwasaki N. Color stability of adhesive resin cements after immersion in coffee. Clin Oral Investig 2015;19:309-17.
13. Harorli OT, Barutcigli Ç, Bayindir YZ, Bayindir F. Effect of water storage and additional polymerization on the color parameters of flowable resin composites. J Contemp Dent Pract 2013;14:1109-14.
14. Boaro LC, Gonçalves F, Guimarães TC, Ferracane JL, Pfeifer CS, Braga RR. Sorption, solubility, shrinkage, and mechanical properties of “low-shrinkage” commercial resin composites. Dent Mater 2013;29:398-404.
15. Berrong JM, Weed RM, Schwartz IS. Color stability of selected dual-cure composite resin cements. J Prosthodont 1993;2:24-7.
16. Diamantopoulou S, Papazoglou E, Margaritis V, Lynch CD, Kakaboura A. Change of optical properties of contemporary resin composite after one week and one month water ageing. J Dent 2013;41 suppl 5:62-9.
17. Smith DS, Vandewalle KS, Whisler G. Color stability of composite resin cements. Gen Dent 2011;59:390-4.
18. Salgado VE, Cavalcante LM, Silikas N, Schneider LF. The influence of nanoscale inorganic content over optical and surface properties of model composites. J Dent 2013;41:45-53.
19. Lepri CP, Palma-Dibb RG. Surface roughness and color change of a composite: influence of beverages and brushing. Dent Mater J 2012;31:689-96.
20. Topcu FT, Sahinkesen G, Yamanel K, Erdemir U, Oktay EA, Ersahan S. Influence of different drinks on the colour stability of dental resin composites. Eur J Dent 2009;3:50-6.
21. Rüttermann S, Suyoun K, Raab WH, Janda R. Effect of exposure time on the color stability of resin- based restorative materials when polymerized with quartz-tungsten halogen and LED light. Clin Oral Investig 2010;14:599-605.
22. Samra AP, Pereira SK, Delgado LC, Borges CP. Color stability evaluation of esthetic restorative materials. Braz Oral Res 2008;22:205-10.
23. Sabatini C, Campillo M, Aref J. Color stability of ten resin-based restorative material. J Esthet Restor Dent 2012;24:185-99.
24. Festuccia MS, Garcia Lda F, Cruvinel DR, Pires-De-Souza Fde C. Color stability, surface roughness, and microhardness of composites submitted to mouthrinsing action. J Appl Oral Sci 2012;20:200-5.
25. Ural Ç, Duran I, Tatar N, Özturk Ö, Kaya I, Kavut I. The effect of amine-free initiator system and the polymerization type on color stability of resin cements. J Oral Sc 2016;58:157-61.
26. Noie F, O'Keefe KL, Powers JM. Color stability of resin cements after accelerated aging. Int J Prosthodont 1995;8:51-5.
27. Uchida H, Vaidyanathan J, Viswanadhan T, Vaidyanathan TK. Color stability of dental composites as a function of shade. J Prosthet Dent 1998;79:372-7.
28. Khokhar ZA, Razzoog ME, Yaman P. Color stability of restorative resins. Quintessence Int 1991;22:733-7.
29. Turgut S, Bagis B. Color stability of laminate veneers: an in vitro study. J Dent 2011;39 Suppl 3:e57-64.
30. Vichi A, Ferrari M, Davidson CL. Color and opacity variations in three different resin-based composite products after water aging. Dent Mater 2004;20:530-4.
31. Benetti AR, Ribeiro de Jesus VC, Martinelli NL, Pascotto RC, Poli-Frederico RC. Colour stability, staining and roughness of silorane after prolonged chemical challenges. J Dent 2013;41:1229-35.
32. Ren YF, Feng L, Serban D, Malmstrom HS. Effects of common beverage colorants on color stability of dental composite resins: the utility of a thermocycling stain challenge model in vitro. J Dent 2012;40 suppl 1:48-56.
33. Paravina RD, Ontiveros JC, Powers JM. Accelerated aging effects on color and translucency of bleaching-shade composites. J Esthet Restor Dent 2004;16:117-26.
34. Dozic A, Tsagkari M, Khashayar G, Aboushelib M. Color management of porcelain veneers: influence of dentin and resin cement colors. Quintessence Int 2010;41:567-73.
35. Janda R, Roulet JF, Latta M, Kaminsky M, Rüttermann S. Effect of exponential polymerization on color stability of resin-based filling materials. Dent Mater 2007;23:696-704.
36. Domingos PA, Garcia PP, Oliveira AL, Palma-Dibb RG. Composite resin color stability: influence of light sources and immersion media. J Appl Oral Sci 2011;19:204-11.
37. Santos PH, Souza FI, Guedes AP, Pavan S. Effect of postpolymerisation method on the color stability of composite resins submitted to ultraviolet aging. Int J Periodontics Restorative Dent 2012;32:95-100.
38. Saygili G, Sahmali S, Demirel F. Colour stability of porcelain repair materials with accelerated ageing. J Oral Rehabil 2006;33:387-92.
39. Silami FD, Mundim FM, Garcia Lda F, Sinhoreti MA, Pires-de-Souza Fde C. Color stability of experimental composites containing different photoinitiators. J Dent 2013;41 suppl 3:62-6.
40. International Organization for Standardization. ISO 7491:2000. Dental materials – determination of color stability. Geneva: ISO. 2017. Available at: https://www.iso.org/standard/26857.html.
41. Schneider LF, Cavalcante LM, Silikas N, Watts DC. Degradation resistance of silorane, experimental ormocer and dimethacrylate resin-based dental composites. J Oral Sci 2011;53:413-9.
42. Hekimoğlu C, Anil N, Etikan I. Effect of accelerated aging on the color stability of cemented laminate veneers. Int J Prosthodont 2000;13:29-33.

Corresponding author:
Dr Nadim Z. Baba
Loma Linda School of Dentistry
Advanced Specialty Education Program in Prosthodontics
11092 Anderson Street
Loma Linda, CA 92350
Email: nbaba@llu.edu

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