Color stability and degree of conversion of a novel dibenzoyl germanium derivative containing photo-polymerized resin luting cement

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Abstract
Aim: To compare the color stability and degree of conversion (DC) of a resin cement containing a dibenzoyl germanium derivative photo-initiator (Variolink Esthetic) to resin cements containing conventional luting agents.

Materials and Method: Spectrophotometry and Fourier transform infrared spectroscopy (FTIR) were used to compare the color stability and DC, respectively, of Variolink Esthetic compared to Calibra, Variolink-N, and NX3 resin cements. Ten specimens (1 × 2 mm²) of each resin cement were photo-polymerized and then subjected to color stability assessments. In addition, 30 samples of each of the four resin cements were prepared and then immersed in three staining solutions (tea, coffee, and distilled water) for two weeks. Changes in color for the immersed versus non-immersed specimens (control specimens) were determined by comparing ∆L (lightness), ∆a, and ∆b (color components), and an overall ∆E (color difference) obtained from spectrophotometry assays.

One-way analysis of variance and a multiple comparison test (Tukey’s test) were used to analyze color stability and DC data. NX3 and Variolink Esthetic resin cements exhibited significantly lower values compared to the dual cured resin cements (Variolink-N and Calibra).

Results: The highest DC values were observed among the photo-polymerized samples of Variolink Esthetic (87.18 ± 2.90%), while the lowest DC values were observed among the Variolink-N samples (44.55 ± 4.33%).

Conclusion: The resin cement, Variolink Esthetic, containing a novel dibenzoyl germanium derivative photo-initiator exhibited superior color stability (p < 0.05) and a higher DC than other resin cements containing conventional luting agents in an in vitro setting.

Keywords
Dibenzoyl germanium derivative, color stability, degree of conversion, luting cements, in vitro study

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Introduction

To successfully meet the requirements of esthetic dentistry, mechanically durable and optically stable contemporary ceramics in the form of veneers, inlays, onlays, crowns, and fixed partial dentures are pivotal. Equally critical is the adhesive bonding of ceramic restorations to tooth structures with the use of contemporary resin luting agents. Resin cements are classified on the basis of the polymerization method (chemical cure, photo cure, dual cure), filler particle size (micro, macro, hybrid, nano), and method of bonding (total etch versus self-etch). A critical deterministic of a resin cement’s characteristics is the degree of conversion (DC) of monomers to polymers. A low DC is linked to transverse mechanical strength, bond stability, pulpal integrity, and clinical longevity. Both laboratory and clinical evidence suggest that the DC is influenced by many factors, including the size, shape, and distribution of filler particles, restorative placement technique, type of photo-initiator used, and frequency of firing.

Another criterion of adhesive resin cement is color stability and discoloration. In fact, color instability of esthetic restorations due to discoloration of luting agents is one of the primary reasons for replacement of these restorations. Material-dependent factors that influence discoloration by resin luting agents include the type of filler, resin matrix, photo-initiator, polymerization, and DC. In particular, dual cure (chemical and light cure) cements tend to darken over time. Therefore, light cure cements, rather than dual cure cements, are recommended for anterior esthetic restorations (i.e., for translucent anterior cementation of veneers). The compromised color stability of dual cure cements (chemical and light cure) is related to the oxidation of unreacted co-initiators from polymerization and to residual benzoyl peroxide, both of which alter the color and long-term aesthetics of dual cure cements. In contrast, light cure resin cements use a Norrish type II photo-initiator (camphorquinone, CQ) to produce free radicals (chain reactions in which the addition of a monomer molecule to an active chain-end regenerates the active site at the chain-end) during the polymerization reaction. Since tertiary amines are not utilized for chemical activation, the co-initiators in light polymerizing cements are relatively more stable and, thus, cause less color variation over time. Furthermore, accumulating evidence suggests that changes in color stability are also associated with the DC of luting cements. The DC of monomer to polymer influences the physical and chemical stability of a material. For example, the presence of non-converted double carbon bonds (formed when two atoms share two pairs of electrons; double bonds are made of one pi bond and one sigma bond) in a material make it more susceptible to degradation, color instability, and the by-products, formaldehyde and methacrylic acid, which compromise the biocompatibility of luting cements.

Ongoing research has led to the development of contemporary cements that exhibit improved color stability and DC with resin-based adhesive agents. Variolink Esthetic (Variolink-E) is one such photo-polymerized resin cement. It contains a thiocarbamide photo-initiator derived from dibenzoyl germanium, called Ivocerin. This Norrish type II photo-initiator was designed to improve the DC and color stability of resin cements by acting as an inhibitor booster. It has been reported that exposure of Ivocerin to light induces the splitting of chemical bonds within the photo-initiator to form a polymerized network. Furthermore, initial studies of bulk fill composites have shown that Ivocerin displays an increased DC and reactivity to light (between wavelengths ranging from 370 to 460 nm) compared to other photo-initiators.

To date, inadequate evidence is available regarding the color stability and DC of Variolink-E compared to commonly used luting cements. It is hypothesized that Variolink-E will exhibit better resistance to staining (color stability) and a higher DC compared to conventional luting cements based on its use of a dibenzoyl germanium derivative initiator. To investigate this hypothesis, we compared the color stability and DC of Variolink-E, Calibra, Variolink-N, and NX3 resin cements.

Materials and methods

In this non-clinical, in vitro experimental study, color stability and DC were assessed for four resin cements by using Fourier transform infrared spectroscopy (FTIR) and spectrophotometry, respectively. The study was approved by the ethical committee of King Saud University and follows checklist for reporting in vitro studies (CRIS) guidelines. The study was carried out from June 2018 to December 2018, that is, for a period of six months.

Color stability

The color stability of light shade resin luting cements, Variolink-E, NX3, Calibra, and Variolink-N, was assayed. Briefly, specimens were prepared by placing a Teflon mold ring on top of a 1 mm thick glass slide. The cements were mixed according to their manufacturer’s instructions and were placed in the molds. After removing the excess cement from each mold, another glass slide was placed on top of each ring using a load of 10 g to standardize the thickness. Thirty disks (diameter, 5 mm; thickness, 1 mm) were prepared with each luting agent. The tip of a light-emitting
diode (LED) source (Woodpecker I-LED dental wireless LED curing light, 2300 mw/cm²) was slightly touching the glass slide placed perpendicularly so that it covered each specimen entirely for 40 s while curing.

The specimen color was initially evaluated after 24 h, and this was defined as the baseline, or control, evaluation. Briefly, each specimen was placed in a desktop X-Rite ColorEye 7000A Spectrophotometer (Grand Rapids, MI, USA) with a resin jig for reproducibility and standardization of position. The spectrophotometer employs a dual beam pulsed xenon light source and was calibrated according to National Institute of Standards and Technology (NIST) tiles. The wavelength interval of the spectrophotometer was 10 nm and its spectral range extended from 360 to 750 nm. Reflectance measurements were made at a 45° angle with illumination at 0° (built in). The trap latch of the sample holder was colored black and it served as the background for all of the samples. The specimens were thoroughly cleaned with absorbent paper and ethyl alcohol and then dried prior to their placement and measurement in the spectrophotometer. To minimize loss of light through the margins of the specimens (referred to as edge-loss), a drop of distilled water was added to the space between the specimen and the backing to seal the airspace and confirm that the water was added to the space between the specimens (referred to as edge-loss), a drop of distilled water and this was defined as the baseline, or control, evaluation.

The trap latch of the sample holder was colored black and it served as the background for all of the samples. The specimens were thoroughly cleaned with absorbent paper and ethyl alcohol and then dried prior to their placement and measurement in the spectrophotometer. To minimize loss of light through the margins of the specimens (referred to as edge-loss), a drop of distilled water was added to the space between the specimen and the backing to seal the airspace and confirm that the specimen was in optical contact with the backing during the reflectance measurements collected. Three measurements were recorded for each specimen and the corresponding mean values were used to represent the average values of the color parameters of each sample. All the experimentation was done under room temperature of 37°C. Color differences were measured in three axes (L*, a*, b*) by using the CIELAB color space. “L*” represents a brightness to darkness range from 0 to 100, the “a*” axis is colored red to green to represent coordinates ranging in value from 90 to 70, and the “b*” axis is colored yellow to blue to represent coordinates ranging in value from –80 to 100.

A total of 120 samples were assessed for color stability based on power calculations. Therefore, 30 samples of each of the four luting resin agents were immersed in each of three solutions (10 samples/solution): tea (Lipton, tea bag solution, Unilever, Saudi Arabia), coffee (Instant black, Nescafe, Switzerland), and distilled water. The tea solution was prepared by immersing two tea bags in 300 ml boiling water for 5 min. To prepare the coffee solution, 5 g of coffee crystals were mixed with 300 ml boiling water and then the solution was filtered after 1 min. Each solution was replaced with a freshly prepared solution each day for one week. After two weeks, each specimen was rinsed under distilled water for 5 min and then was dried with absorbent paper. Color was assessed with a X-Rite Color Eye 7000A Spectrophotometer, as described above. Differences in color between the specimens that had been immersed in staining solutions versus the controls were determined by comparing ΔL, Δa, and Δb values. The CIELAB color difference value, ΔE*, between two colors was assessed based on a Euclidean distance determined between the points representing them in space. Accordingly, an overall ΔE* value was calculated based on the formula adopted from the CIE technical report on colorimetry (8.21)

\[ ΔE^*_{ab} = \left( ΔL^2 + (Δa^*)^2 + (Δb^*)^2 \right)^{1/2} \]

Normal distribution of data was assessed by applying the Kolmogorov–Smirnov test. One-way analysis of variance (ANOVA) was used to analyze color difference and DC data. A post hoc multiple comparison test, Tukey’s test, was applied to compare acquired mean values among the study groups.

**DC**

The DC was evaluated at the top of 10 specimens for each of the four cements examined (Calibra, NX3, Variolink-N, and Variolink-E) by using FTIR combined with attenuated total reflectance (FTIR/ATR; Spectrum 100; PerkinElmer, Shelton, CA, USA) (FTIR spectroscope: Model 4100, Jasco Corporation, Tokyo, Japan) (Pike Miracle ATR Unit, Diamond ZnSeW Technology). The DC methodology used was adopted from previous studies. Resin cement samples were placed into vinyl molds (thickness, 1 mm; inner diameter, 2 mm). Mylar strips (DuPont Mylar 0.002 gauge/60 mm thick) were used to inhibit oxygen polymerization and facilitate placing of the ceramic discs prior to photo-polymerization. The LED tip of a Woodpecker I-LED dental wireless curing device (2300 mw/cm²) was applied through the ceramic disks containing the light cured resin cements (Variolink-E and NX3) for 40 s to achieve photo-polymerization. For the dual cure cements (Calibra and Variolink-N), equal lengths of the two pastes of cement were mixed and then were allowed to auto-polymerize for 120 s. The cements were subsequently photo-polymerized through the ceramic discs for 40 s (a similar light cure protocol was applied to all of the samples) prior to their placement in the FTIR chamber to calculate their cured spectrum.

A spectrum for un-polymerized resin was also calculated in absorbance mode at four wave number resolutions obtained from 16 scans. To prevent internal reflectance patterns, background spectra were collected for an empty mold and for a glass slide before running samples. The spectra of the monomers and their respective polymers were compared to determine the conversion rate of the double bonds into simple carbon bonds. The aliphatic (C–C double bond absorbance peak intensity at 1638 cm⁻¹) and aromatic (C–C at 1608 cm⁻¹) absorbance peak intensities of carbon–carbon double bonds formed during polymerization were recorded.
Both peak area and peak height were determined by a swift-quant software computer program (Philips, Germany). The DC% value for each specimen was calculated according to the following equation:

\[
DC\% = \left(1 - \frac{\text{Peak Area}_{\text{uncured}}}{\text{Peak Area}_{\text{cured}}}\right) \times 100
\]

### Results

**Color stability**

Mean ± standard deviation values for color change (\(\Delta E\)) among the resin cements after their immersion in three different staining solutions are presented in Table 1.

Immersion in coffee produced the greatest color change (\(\Delta E\) values) in all cement types, Calibra (7.11 ± 2.15), Variolink-N (5.65 ± 2.17), NX3 (4.55 ± 1.33), and Variolink-E (3.51 ± 1.36), followed by immersion in tea, Calibra (6.13 ± 2.23), Variolink-N (4.59 ± 2.23), NX3 (3.65 ± 1.73), and Variolink-E (3.07 ± 1.17) (<0.05). Immersion in distilled water resulted in minimal color change among all four resin cements. Variolink E and Calibra exhibited minimum and maximum color changes in all of the immersion solutions (<0.05). Meanwhile, Variolink-E and NX3 exhibited comparable color changes in all three immersion solutions (<0.05).

### Discussion

The present study was conducted to test the hypothesis that the DC and color stability of the light cure resin cement, Variolink E, which contains a novel dibenzoyl germanium derivative photo-initiator (Ivocerin), exceed that of resin cements that do not contain a dibenzoyl germanium derivative initiator (NX3, Calibra, and Variolink N). The results obtained confirm this hypothesis.

A statistically higher DC value (i.e., percentage of reacted aliphatic C=C bonds from the dimethacrylate monomers present in their polymeric matrices) was observed for Variolink E compared to the other materials tested (Varioink N, Calibra, and NX3). In general, the mechanical characteristics of luting cements that were evaluated include surface micro-hardness, flexural, compressive, and...
tensile strengths. Moreover, all of these characteristics are influenced by the DC of luting cements. When inadequate polymerization occurs, the corresponding reduction in DC impacts both bond strength and dimensional stability, and can possibly lead to allergic reactions. In addition, DC has been reported to be influenced by the translucency and dimensions of ceramics restorations.

We analyzed the DC with FTIR since this technique provides rapid scanning at high wavelengths and the results have better resolution, stability, and accuracy. To promote clinical simulation, luting cements were polymerized through ceramic discs.

When Ivocerin is exposed to light, it reacts with monomers to promote polymerization. The incorporation of this novel photo-initiator into bulk fill resin composite (such as Variolink E) has resulted in an increase polymerization, improved reactivity to curing light, and greater cure depth. The DC findings for Variolink E in the present study showed it to be superior to other resin cements examined, consistent with the results of previous studies. In addition, the findings that the NX3 samples exhibited a superior DC compared to the Variolink-N and Calibra cements are also consistent with previous findings. A possible explanation for the latter results may be that NX3 has a redox initiator system, which lacks both a tertiary amine and benzoyl peroxide. Redox initiators are also compatible with acidic adhesives, thereby making NX3 compatible with total and self-etch adhesives. The low DC observed for Variolink-N and Calibra compared to Variolink-E and NX3 may also be associated with the instability of these dual cure cements due to the presence of tertiary amines and benzoyl peroxide as initiators.

To compare the color stability of the four luting cements in the present study, spectrophotometry with the CIE \(L^* a^* b^*\) system and a color difference formula was employed. Currently, the CIE system is the most reproducible and comparable color measuring system. It is also recommended according to the standards described in the CIE technical report on colorimetry (8.21). Among the luting agents that were compared, Variolink E had the lowest \(\Delta E\) values, which indicated the greatest color stability. In a previous study conducted by Singh et al., Ivocerin was considered to be 100% amine free since it incorporates shade stability and bleaching properties into a composite resin. In the present study, the dual cure resin cements (Variolink N and Calibra) showed higher \(\Delta E\) values (thereby indicating lower color stability) compared to NX3 and Variolink-E. A possible reason for these results is the presence of benzoyl peroxide as an initiator in the Calibra cement, which can cause changes in shade since the reaction between benzoyl peroxide and amines has low efficacy. In addition, during the polymerization process, initiator mobility is reduced due to increasing viscosity, thereby leaving unreacted benzoyl peroxide within the polymer network.

The poor color results observed for Variolink-N may be associated with its tertiary amine and use of a CQ initiator. Amine molecules pair up with CQ molecules and are directly involved in determining the optical properties of polymerized materials. Furthermore, the co-initiator, CQ, undergoes photo bleaching during polymerization, and this can lead to a slight color change. Meanwhile, both Variolink N and Calibra contain bisphenol A-glycidyl methacrylate (Bis-GMA) as a monomer, and this compound is vulnerable to hydrolysis. Therefore, hydrolytic degradation and a hygroscopic (resulting in microstructural surface damages) effect are possible factors that may influence color instability in Variolink N and Calibra resins.

The comparable color stability results obtained for NX3 and Variolink E may be due to their use of a proprietary redox initiator system, which prevents color shifting in NX3 before, during, and after the curing process.

It is noteworthy that the degree of color change that was observed among the luting cements varied following their immersion in tea, coffee, and distilled water. Greater discoloration was observed following immersion in coffee compared with tea. In contrast, distilled water did not induce any significant discoloration among all four cement groups. These results suggest that the degree of color change associated with the resin luting cements tested is due to the material composition of the cement rather than the immersion solutions used. These findings are also consistent with the outcomes of similar previous studies.

Considering that Variolink E exhibited an overall higher DC and greater color stability compared to other resin cements (Variolink N and Calibra), it is recommended for use with restorations that have less thickness in an esthetic zone, such as laminate veneers, as increased ceramic thickness may reduce cement DC. Interestingly, a correlation between the DC and polymerization shrinkage of resin luting cements has previously been reported. It is important to note that other possible factors, such as wear resistance, bond strength,
and/or polymerization shrinkage, may also contribute to the observed characteristics of Variolink E. Therefore, further in vitro experiments for biological characterization and randomized controlled trials are needed to assess the viability of Variolink E in a clinical setting.

Conclusions

Variolink E, which contains a novel dibenzoyl germanium derivative photo-initiator (Ivocerin), exhibited greater color stability and a higher DC compared to conventional resin-based luting cements in an in vitro setting.

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Author contributions

Conceptualization and methodology, original draft preparation, FV. Methodology, formal analysis, investigation and project administration, FA (Fahad Alkhudhairy). Investigation, software and validation, and original draft preparation, MN. Preparation of draft and resources, review, and editing, MMO, AHBA, and KBA.

Data availability

The data used to support the findings of this study are available from the corresponding author upon request.

Declaration of conflicting interests

The authors have no conflicts of interest to declare.

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