



# Color stability and degree of conversion of amine-free dual cured resin cement used with two different translucencies of lithium disilicate ceramics

Estabilidade de cor e grau de conversão de cimento resinoso dual sem amina usado com duas translucidezes diferentes de cerâmica de dissilicato de lítio

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## ABSTRACT

**Objective:** The present study aims to evaluate the color stability and degree of conversion of amine-free dual cured resin cement compared to light cured and amine-containing dual cured resin cements used with two different translucencies of thin esthetic restorations. **Material and Methods:** A total of 120 specimens were prepared for color stability testing (n=60). The specimens were divided into three main groups according to the resin cement type. Group 1: amine-free dual cured, Group 2: light cured, Group 3: amine-containing dual cured. Each group was further subdivided according to the ceramic translucency into two subgroups: high and low translucency. Color stability was assessed by a spectrophotometer before and after thermal aging. For the degree of conversion assessment (n=60), Fourier transform infrared spectroscopy was used at three different time intervals. Statistical analysis was performed using multi-factorial ANOVA, followed by one-way ANOVA with Bonferroni correction. **Results:** Amine-containing resin cement showed significantly higher  $\Delta E_{ab}$  and  $\Delta E_{00}$  in both translucencies ( $4.5 \pm 0.3$ ,  $3.5 \pm 0.3$  respectively for high translucency ceramic and  $3.8 \pm 0.4$ ,  $3.0 \pm 0.3$  respectively for low translucency) than the other tested cements ( $p < 0.001$ ). The highest degree of conversion (DC) was shown after 2 weeks by the amine-free dual cured resin cement ( $86.27 \pm 0.74$ ). **Conclusion:** Amine-free dual cured resin cement can be an alternative to light cured one for cementation of thin veneers since it showed comparable color stability and high degree of conversion.

## KEYWORDS

Resin cement; Color; Polymerization; Light curing of dental resins; Lithium disilicate.

## RESUMO

**Objetivo:** O presente estudo tem como objetivo avaliar a estabilidade de cor e o grau de conversão do cimento resinoso dual sem amina em comparação com cimentos resinosos fotopolimerizáveis contendo amina usados com duas translucidezes diferentes em restaurações estéticas definitivas. **Material e Métodos:** Um total de 120 espécimes foram preparados para teste de estabilidade de cor (n=60). Os espécimes foram divididos em três grupos principais de acordo com o tipo de cimento resinoso. Grupo 1: polimerização dupla sem amina, Grupo 2: fotopolimerização, Grupo 3: polimerização dupla contendo amina. Cada grupo foi ainda subdividido de acordo com a translucidez da cerâmica em dois subgrupos: alta e baixa translucidez. A estabilidade da cor foi avaliada por um espectrofotômetro antes e após o envelhecimento térmico. Para a avaliação do grau de conversão (n=60), a espectroscopia de infravermelho com transformada de Fourier foi usada em três intervalos de tempo diferentes. A análise estatística foi realizada usando ANOVA multifatorial, seguida de ANOVA um fator com correção de Bonferroni. **Resultados:** O cimento resinoso contendo amina apresentou  $\Delta E_{ab}$  e  $\Delta E_{00}$  significativamente maiores

em ambas as translucidezes ( $4,5 \pm 0,3$ ,  $3,5 \pm 0,3$  respectivamente para cerâmica de alta translucidez e  $3,8 \pm 0,4$ ,  $3,0 \pm 0,3$  respectivamente para baixa translucidez) do que os outros cimentos testados ( $p < 0,001$ ). O maior grau de conversão (DC) foi mostrado após 2 semanas pelo cimento resinoso dual sem amina ( $86,27 \pm 0,74$ ). **Conclusão:** O cimento resinoso dual sem amina pode ser uma alternativa ao cimento polimerizável na restauração de facetas finas, uma vez que apresentou estabilidade de cor comparável e alto grau de conversão.

## PALAVRAS-CHAVE

Cimento resinoso; Cor; Polimerização; Fotoativação de cimentos odontológicos; Dissilicato de lítio.

## INTRODUCTION

Recently, there is an increasing demand for esthetic restorations due to an increased esthetic conscious society [1]. Lithium disilicate is one of the most commonly used glass ceramics for the fabrication of laminate veneers, attributed to its excellent esthetic properties, adequate strength and bonding capacities which enable its use in thin esthetic veneers [2,3].

Laminate veneers are commonly delivered in 0.3-0.7-mm thicknesses and they represent a more conservative approach compared to full coverage all-ceramic restorations allowing superior translucency and esthetics [4,5]. Hence, they have been highly favored as an esthetic restoration for anterior teeth [6,7]. Nevertheless, the final esthetic outcome can be influenced by the color interaction with the underlying tooth substrate and luting resin cement [8-10].

Resin cements are the materials of choice for bonding ceramics to dental substrates and they are considered an essential part for the success and quality of esthetic treatments [11]. Resin cements are classified according to their method of activation into chemically cured, light-cured and dual-cured. In chemically-cured resin cements, color instability and lack of control over the working time make clinicians prefer to use either light-cured or dual-cured resin cements in luting of restorations [12].

Light-cured resin cements are preferred for luting ceramic veneers due to their good color stability, less possibility for air bubble incorporation and absence of tertiary amine chemical initiator. However, the possible attenuation of the light intensity by the thickness, variable degrees of translucency and type of ceramic may be a shortcoming [13].

Dual cured resin cements have the advantages of both light and chemically cured resin cements. They exhibit extended working time due to light-controlled polymerization in

addition to the chemical activators that ensure a high degree of polymerization in deeper areas where light cannot penetrate [14]. Moreover, it has been reported that they have a higher degree of conversion in comparison to the light cured cements [15].

Color stability of resin cements is a common problem, particularly when used with thin translucent restorations such as laminate veneers [16-18]. There are intrinsic and extrinsic factors that could affect the color stability of resin cements. The extrinsic factors such as smoking, beverages and food components may have the potential to stain restorative materials [19].

The intrinsic factors are material-related such as the composition of the resin matrix and fillers, filler particle size distribution, type of photo-initiator system, as well as degree of conversion percentage [20]. In addition, thermal change, UV irradiation, and humidity may cause intrinsic discoloration by physicochemical reaction. However, the evaluation of the color stability of resin cements after thermocycling is limited [21]. Thus, resin cement should have long-term color stability to guarantee acceptable esthetic results [22].

For color assessment, a spectrophotometer is used since it is among the most accurate tools [23]. In color science, a color difference formula ( $\Delta E$ ) is designed to give a quantitative representation, and there are two color difference formulas: CIELAB and CIEDE2000. For interpretation of these data into real-life scenarios, it should be compared to the acceptability threshold (AT), which is the limit above which the magnitude of color difference will be considered as clinically unacceptable for dental esthetics [24-27]. In addition, values above the AT were categorized as mismatch type [a] or moderately unacceptable ( $\leq AT \times 2$ ), mismatch type [b] or clearly unacceptable ( $\leq AT \times 3$ ), and mismatch type [c] or extremely unacceptable ( $> AT \times 3$ ) [26].

To solve the discoloration problem, new dual cured resin cements have been manufactured without a benzoyl peroxide/amine redox initiator system to be more color stable [28,29]. Variolink Esthetic is one of the amine free dual cured resin cements available in the market that was claimed by the manufacturer to be the first entirely amine-free with the new patented photo-initiator Ivocerin [30-32].

The most commonly used photo-initiator system in resin-based materials is camphorquinone (CQ), which needs an amine co-initiator such as ethyl 4-dimethylaminobenzoate (DMAB) to react with and create free radicals needed for the polymerization reaction [33]. These amine molecules go through oxidation reactions and result in color change. In dual cured resin cements, more color change occurs due to the presence of benzoyl peroxide and the tertiary amine initiator system of the chemical part, in addition to the oxidation of unreacted co-initiators from the light cured part [34].

Ivocerin, the new germanium-based photo-initiator is an alternative substitute for the CQ-amine photo-initiator system. It does not require amine molecules to initiate the reaction in dental resin-based materials [35,36]. It has been claimed to be more color stable and show enhanced curing depths due to its high photo-reactivity [34].

Adequate polymerization of resin cements is crucial since low degree of conversion could compromise their optical, physical and mechanical properties [37]. Fourier-transform infrared spectroscopy (FTIR) is used to measure DC% since it is one of the most precise methods with fast scanning capacity, good resolution, stability and accuracy [38,39].

The thickness and opacity of lithium-disilicate ceramic is known to compromise polymerization of the resin-based luting composite. However, studies regarding the effect of the ceramic interposition have focused mainly on traditional resin cements [40,41]. Currently, the debate is whether the amine free dual-cured resin cement would be as efficient as the light cured one considering color stability and degree of conversion when used with thin translucent esthetic restorations [29,42].

Accordingly, the research hypotheses of the current study are:

1. There is no difference in the color stability and degree of conversion of resin cements with different photo-initiator systems used in cementation of thin esthetic veneers.
2. The different translucencies of lithium disilicate ceramics have no effect on the final color stability of thin esthetic veneers.

## MATERIAL AND METHODS

The materials used in the present study, their composition, manufacturers and lot numbers are presented in Table I.

### Specimens' grouping

A total of 120 specimens were prepared for the present study: 60 bovine-bonded ceramic specimens were prepared for color stability assessment and 60 resin cement specimens were prepared and light cured through the ceramic specimens for the degree of conversion assessment. For each test, specimens were divided into three main groups (n=20) according to the resin cement type. Group 1 (G1): Variolink Esthetic amine-free dual cured resin cement, Group 2 (G2): Variolink Esthetic light cured resin cement and Group 3 (G3): Variolink N amine-containing dual cured resin cement. Each group was further subdivided into two subgroups (n=10), according to ceramic translucency: high translucent ceramic (HT) and low translucent ceramic (LT) (Figure 1).

### Color stability specimens' preparation

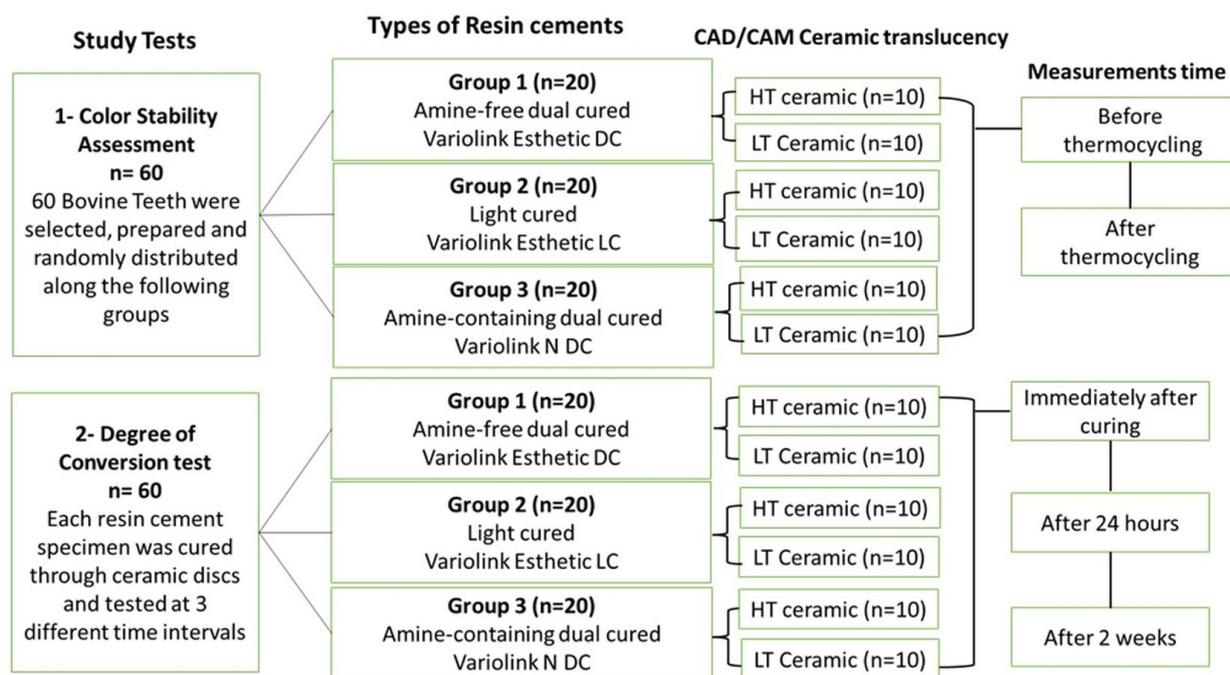
#### *Bovine teeth preparation*

Sixty bovine incisors were collected and selected with intact buccal enamel and average crown widths of 12 to 15 mm. The teeth were cleansed of soft tissue remnants and debris with a sharp scalpel, stored in water in order to remain hydrated, simulating the oral clinical situation of human teeth [16].

The roots were then removed 1 mm below the cemento-enamel junction (CEJ), using a high-speed handpiece. After separating the roots from their respective crowns, the enamel of the buccal surface of each tooth was flattened and polished with 320, 600 and 1200 grit SiC abrasive papers (3M of Brazil, Sumare, Brazil), under running water for 1 min [16,18].

**Table 1** - Materials used in the present study, their composition, manufacturer and lot numbers

Material	Description	Composition	Manufacturer	Lot number
Group 1:(G1) Variolink Esthetic DC	Amine-free dual cured resin cement Shade: Neutral	Matrix: UDMA, methacrylate monomers Inorganic fillers: ytterbium trifluoride and spheroid mixed oxide Ivocerin photo initiator and hydroperoxide/thiocarbamide self-cure initiator system Additional ingredients: stabilizers, pigments	Ivoclar/ Vivadent, Switzerland	W95564
Group 2:(G2) Variolink Esthetic LC	Light cured resin cement Shade: Neutral	Matrix: UDMA, methacrylate monomers Inorganic fillers: ytterbium trifluoride and spheroid mixed oxide Ivocerin photo initiator Additional ingredients: stabilizers, pigments	Ivoclar/ Vivadent, Switzerland	Y42601
Group 3:(G3) Variolink N DC	Amine containing dual cured resin cement Shade: Transparent	<b>Base:</b> Barium glass filler and mixed oxide, ytterbium trifluoride, dimethacrylates (BisGMA, UDMA, and TEGDMA) CQ-amine photoinitiator, stabilizers and pigments. <b>Catalyst:</b> Barium glass filler and mixed oxide, ytterbium trifluoride, dimethacrylates, benzoyl peroxide initiator, stabilizers and pigments.	Ivoclar/ Vivadent, Switzerland	Base X51772 Catalyst X49378
IPS e.max CAD/CAM Blocks: High translucent (HT) Low translucent (LT)	Lithium disilicate glass-ceramic blocks Shade: A2	SiO <sub>2</sub> : 57–80%; Li <sub>2</sub> O: 11–19%; in addition to K <sub>2</sub> O; P <sub>2</sub> O <sub>5</sub> ; ZrO <sub>2</sub> ; ZnO; Al <sub>2</sub> O <sub>3</sub> ; MgO; coloring oxides.	Ivoclar/ Vivadent, Switzerland	(HT) Y52153 (LT) Z0047Y

**Figure 1** - Specimens' grouping flowchart

Each prepared tooth was embedded in a polyvinyl mold filled with chemically cured acrylic resin (Acrostone, Egypt), with the exposed flat buccal surface flushing with the upper border of the mold to maintain the veneer in a horizontal level for cementation and color assessment [6,16].

### Ceramic specimens' preparation

To simulate the clinical use of laminate veneers, thin ceramic specimens were fabricated

from lithium disilicate ceramic CAD/CAM blocks (IPS e-max CAD), shade A2 with two different translucencies: high translucent (HT) and low translucent (LT) [6].

IPS e-max CAD blocks were milled into 10 mm diameter cylinders, then each cylinder was sectioned using a diamond-coated low speed precision saw (Isomet 1000, Buehler, Germany), under copious water coolant, producing disc shaped specimens of 10 mm diameters and  $0.3 \pm 0.05$  mm

thickness which mimics the lowest thickness that can be used for ceramic veneers. The final thickness of each sectioned slice was verified using a digital micrometer (Digimatic Micrometer Series 293 MDC-MX Lite, Mitutoyo, Japan).

Ceramic surfaces were then polished using 320, 600 and 1200 grit SiC abrasive papers (3M do Brazil, Sumare, Brazil). All the ceramic specimens were sintered in a ceramic furnace (Programat CS, Ivoclar Vivadent), at 850 °C for 10 min following the manufacturer's recommendations [9].

## Cementation

All cementation procedures were performed according to the manufacturer's instructions. The enamel surface of each bovine tooth was etched with 37% phosphoric acid (CharmEtch, Korea) for 30 s, then washed for 30 s under running water and air-dried. A layer of adhesive (All bond universal adhesive, Bisco, USA), was applied and light cured for 20 s using a LED light curing unit (3M™ ESPE Elipar™ S10, USA), with an output intensity of 1200 mW/cm<sup>2</sup>. The ceramic specimens were etched with 9.5% hydrofluoric acid (Porcelain etchant, Bisco, USA) for 90 s, then washed under running water for 30 s and air-dried for 30 s. Silane coupling agent (Monobond plus, Ivoclar Vivadent), was applied in a thin coat for 60 s. The luting material was applied on the treated surface of each ceramic specimen and carefully seated on the prepared enamel surface, covered with a clean glass slab with a 1kg weight on top for 20 s to form a standardized thickness of cement layer [19]. Next, final curing was performed for 20 s.

## Color stability measurement

Color measurements were performed for each specimen before and after aging using a spectrophotometer (Agilent Cary 5000 UV-Vis-NIR, USA). Aging was performed in the present study using a thermocycling machine (Thermo-Fisher Scientific, USA), for 5000 cycles at temperatures varying between 5 °C and 55 °C, with a dwell time of 30 s and transfer time of 5 s [21]. For each specimen, the average of three measurements were recorded and used in the data analysis. The CIE-lab coordinates were used to calculate the color difference ( $\Delta E$ ) between the baseline color measurement (before thermocycling) and the final color measurement (after thermocycling), by applying the following two equations [16,24]:

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

where  $L^*$  corresponds to the degree of lightness and darkness, whereas  $a^*$  and  $b^*$  coordinates correspond to  $+a^*$ =red,  $-a^*$ =green and  $+b^*$ =yellow,  $-b^*$ =blue, respectively.

$$\Delta E_{00} = \left[ (\Delta L / kL \cdot SL)^2 + (\Delta C / kC \cdot SC)^2 + (\Delta H / kH \cdot SH)^2 + RT (\Delta C^* \Delta H / SC^* SH) \right]^{1/2} \quad (2)$$

where:  $\Delta L$ ,  $\Delta C$  and  $\Delta H$  are the differences in lightness, chroma and hue for a pair of samples in CIEDE2000, and RT is a rotation function that accounts for the interaction between chroma and hue differences in the blue region. The SL, SC, and SH weighting functions adjust the total color difference for variation in the location of the color difference pair at the L, a, and b coordinates, and the kL, kC, and kH parametric factors are correction terms for experimental conditions. In the present study, the kL, kC, and kH were set to 1 [24].

The resultant  $\Delta E$  values were compared to the 50:50% perceptibility threshold (PT), and 50:50% acceptability threshold (AT). The corresponding CIELAB visual thresholds were 1.2 and 2.7 and for CIEDE2000, were 0.8 and 1.8, respectively.

## Degree of conversion specimens' preparation

Sixty resin cement specimens were prepared in a split Teflon mold (1 mm thickness x 2 mm inner diameter), supported by a metal ring. The G1 and G2 resin cements were packed directly into the mold, while for G3, an equal amount of base and catalyst pastes were mixed according to the manufacturer's instructions and packed into the mold. A Mylar strip (Quimidrol; Joinville, SC, Brazil), was then placed on the top surface of the mold to ensure smoothness of the specimens, allowing the ceramic disc to be easily placed over the resin cement and to avoid inhibition of polymerization by oxygen [37].

The ceramic specimen was then placed over the mylar strip. All curing procedures were performed through the thin ceramic specimen for 40 s to simulate the clinical condition. All cured resin specimens were dry stored in light-proof containers to avoid additional exposure to light.

## Degree of conversion measurement

For degree of conversion (DC) testing, attenuated-total-reflectance/Fourier Transform Infrared spectroscopy (ATR/FTIR VERTEX

70, Bruker; Ettlingen, Baden, Wurttemberg, Germany), was used at different time intervals; immediately after curing, after 24 hours and after 2 weeks [14]. The absorbance spectrum was acquired by scanning the specimens over a 1638-1608  $\text{cm}^{-1}$  range. The DC was calculated using the following equation [39]:

$$\text{DC (\%)} = 1 - \left[ \frac{C_{\text{aliphatic}} / C_{\text{aromatic}}}{(U_{\text{aliphatic}} / U_{\text{aromatic}})} \right] \times 100 \quad (3)$$

$C_{\text{aliphatic}}$  is the absorption peak at 1638  $\text{cm}^{-1}$ ,  $C_{\text{aromatic}}$  is the absorption peak at 1608  $\text{cm}^{-1}$  of the cured specimen.  $U_{\text{aliphatic}}$  is the absorption peak at 1638,  $U_{\text{aromatic}}$  is the absorption peak at 1608  $\text{cm}^{-1}$  of the uncured specimens.

### Statistical analysis and sample size calculation

Numerical data were explored for normality and variance homogeneity using the Shapiro-Wilk and Leven's tests, respectively. Data showed parametric distribution and homogeneity of variances across the groups, so they were represented as mean and standard deviation (SD) values and were analyzed using the multi-factorial ANOVA, followed by one-way ANOVA for analysis of simple effects with Bonferroni correction. The significance level was set at  $p \leq 0.05$  within all tests. Statistical analysis was performed with the R statistical analysis software version 4.0.3 for Windows.

Based on the results of a previous study [34]. Sample size calculation was performed using G\*Power version 3.1.9.7 [43]. By adopting an alpha level of (0.05) a beta of (0.2) i.e., power = 80% and an effect size (f) of (0.486). The predicted sample size was a total of (60) samples for each test, and (n=10) per each experimental subgroup and this was statistically approved.

## RESULTS

### Color stability

Two-way ANOVA for  $\Delta E_{\text{ab}}$  revealed a significant effect for the cement type and ceramic translucency on color stability, however, the interaction between the two variables was insignificant with a p value = 0.26. Mean and standard deviation (SD) values of color change ( $\Delta E_{\text{ab}}$ ) for different cement types and ceramic translucencies are presented in Table II. Pairwise comparisons for the effect of cement type showed that for both the HT and LT ceramic specimens, G3 showed significantly higher  $\Delta E$  values compared to the other tested groups ( $p < 0.001$ ), where G1 and G2 revealed no statistically significant difference. Regarding ceramic translucency, the HT ceramic specimens had significantly higher mean  $\Delta E$  values than the LT ceramic specimens in all tested groups.

When interpreting the results of the present study with 50:50% acceptability thresholds (AT) for the CIELab system, it was revealed that G1 and G2, only when used with low translucent ceramics, had  $\Delta E_{\text{ab}}$  values below 2.7 and therefore were considered clinically acceptable.

While for  $\Delta E_{00}$  values presented in Table III, it was revealed that for G1 and G3 (amine free and amine containing dual cured resin cements), high translucency samples had significantly higher values than low translucency samples ( $p < 0.05$ ). For high translucency samples, there were significant differences between values of different cements ( $p < 0.001$ ). For low translucency samples, there was also a significant difference with amine containing DC, having a significantly higher value than the other cements ( $p < 0.001$ ). Interpreting these results with the CIEDE2000 50:50% acceptability thresholds (AT), all groups had  $\Delta E_{00}$  values above 1.8 and

**Table II** - Mean  $\pm$  standard deviation (SD) of color change ( $\Delta E_{\text{ab}}$ ) values for different types of cements and ceramics

Cement type	Color change ( $\Delta E_{\text{ab}}$ ) (Mean $\pm$ SD)		p-value
	High translucency	Low translucency	
G1: Amine free DC	3.2 $\pm$ 0.5 <sup>B</sup>	2.3 $\pm$ 0.2 <sup>B</sup>	<0.001*
G2: LC	2.7 $\pm$ 0.1 <sup>B</sup>	2.2 $\pm$ 0.1 <sup>B</sup>	0.011*
G3: Amine containing DC	4.5 $\pm$ 0.3 <sup>A</sup>	3.8 $\pm$ 0.4 <sup>A</sup>	0.001*
p-value	<0.001*	<0.001*	

Means with different superscript letters within the same vertical column are statistically significantly different. \*Significant ( $p < 0.05$ ).

**Table III** - Mean  $\pm$  standard deviation (SD) of color change ( $\Delta E_{00}$ ) values for different types of cements and ceramics

Cement type	Color change ( $\Delta E_{00}$ ) (Mean $\pm$ SD)		p-value
	High translucency	Low translucency	
G1: Amine free DC	2.8 $\pm$ 0.3 <sup>B</sup>	2.1 $\pm$ 0.2 <sup>B</sup>	<0.001*
G2: LC	2.3 $\pm$ 0.1 <sup>C</sup>	2.1 $\pm$ 0.1 <sup>B</sup>	0.170
G3: Amine containing DC	3.5 $\pm$ 0.3 <sup>A</sup>	3.0 $\pm$ 0.3 <sup>A</sup>	0.001*
p-value	<0.001*	<0.001*	

Means with different superscript letters within the same vertical column are statistically significantly different. \*Significant ( $p < 0.05$ ).

were considered clinically unacceptable for both ceramic translucencies.

Results of the correlation between different color measurements presented in Table IV showed that there was a strong positive correlation between both measurements, which was statistically significant ( $r = 0.980$ ,  $p < 0.001$ ).

### Degree of conversion (DC)

Multiple repeated measures ANOVA showed a significant effect for time and cement type for the mean DC values; however, the effect of ceramic translucency and the interaction between the three variables was insignificant. Statistical analysis of the DC results recorded the highest mean DC% at 2 weeks' time interval followed by 24 hours, while those recorded immediately after curing showed the lowest mean values. For both G1 and G3 dual cured resin cements groups, there was a remarkable increase in DC in the first 24 hours compared to the G2 light cured group. After 2 weeks, all groups revealed comparable increase in DC values.

Mean and standard deviation (SD) values for degree of conversion % for different cements and ceramic translucencies are shown in Table V. Immediate records for both the HT and LT ceramic specimens presented significant differences between the different cement types ( $p < 0.001$ ). G2 showed the highest mean value followed by G1, while G3 showed the lowest mean value. Records after 24 hours and after 2 weeks showed no significant difference between the G1 and G2 cements, while the G3 cement showed the lowest mean value for both ceramic translucencies ( $p < 0.001$ ). There was no statistically significant difference between the HT and LT ceramic specimens regarding DC mean values for the different tested groups.

## DISCUSSION

Color is one of the integral parts of esthetic dentistry that affects patients' satisfaction and

**Table IV** - Correlation between the two-color change measurements

r	95%CI		Statistic	p-value
	Lower	Upper		
0.980	0.97	0.99	35.2	<0.001*

r = Pearson correlation coefficient. \*Significant ( $p < 0.05$ ).

self-esteem. Color stability of resin cements is one of the most challenging issues that clinicians encounter especially with thin translucent ceramic restorations. Since the color of resin cements influence the final color of ceramic restorations, neutral shades of resin cements were used in the present study to dismiss their influence on the final color. The thermocycling process was chosen as an aging method to simulate the thermal fluctuation in the intraoral conditions using 5000 cycles which corresponds to 6 months of clinical service [29].

There are two major thresholds for assessment of color difference: perceptibility threshold (PT) and acceptability threshold (AT). These thresholds can serve as a guide for evaluating the clinical performance of dental materials. The PT represents the magnitude of color difference that can be visually detected by a professional dental staff, while AT is the threshold above which the magnitude of color difference constitutes an unacceptable limit to dental esthetics [24,26].

Despite considerable efforts, the identification of  $\Delta E$  values for the "clinically acceptable threshold" is a difficult task and a widely accepted limit remains controversial since most of these thresholds were documented on the basis of *in vitro* studies [7]. In order to satisfy the increase of esthetic demands in recent years, a new CIEDE2000 color change formula had been developed to be more sensitive and comparable to visual observation [24]. The 50:50% perceptibility (PT) and the 50:50% acceptability (AT) thresholds for the CIEDE2000 and CIELAB were set 0.8 and 1.2 and 1.8 and 2.7, respectively [24,26,27].

**Table V** - Mean  $\pm$  standard deviation (SD) of degree of conversion values for different types of cements and ceramics

Time	Cement	DC% (mean $\pm$ SD)		<i>p</i> -value
		HT	LT	
Immediately after curing	G1 amine-free	61.3 $\pm$ 0.9 <sup>B</sup>	60.9 $\pm$ 1.6 <sup>B</sup>	0.581ns
	G2 light cure	74.7 $\pm$ 0.8 <sup>A</sup>	75.0 $\pm$ 0.5 <sup>A</sup>	0.487ns
	G3 amine-containing	42.7 $\pm$ 0.5 <sup>C</sup>	42.0 $\pm$ 0.8 <sup>C</sup>	0.102ns
	<i>p</i> -value	<0.001*	<0.001*	
After 24 hours	G1 amine-free	78.7 $\pm$ 0.4 <sup>A</sup>	78.7 $\pm$ 0.8 <sup>A</sup>	0.913ns
	G2 light cure	78.6 $\pm$ 0.3 <sup>A</sup>	78.3 $\pm$ 0.5 <sup>A</sup>	0.221ns
	G3 amine-containing	52.3 $\pm$ 0.9 <sup>B</sup>	53.4 $\pm$ 1.0 <sup>B</sup>	0.051ns
	<i>p</i> -value	<0.001*	<0.001*	
After 2 weeks	G1 amine-free	86.2 $\pm$ 0.4 <sup>A</sup>	85.7 $\pm$ 0.8 <sup>A</sup>	0.152ns
	G2 light cure	85.9 $\pm$ 0.9 <sup>A</sup>	85.2 $\pm$ 0.6 <sup>A</sup>	0.117ns
	G3 amine-containing	66.3 $\pm$ 0.8 <sup>B</sup>	65.7 $\pm$ 0.6 <sup>B</sup>	0.154ns
	<i>p</i> -value	<0.001*	<0.001*	

Means with different superscript letters within the same vertical columns are significantly different\*; significant ( $p \leq 0.05$ ) ns; non-significant ( $p > 0.05$ ).

The results of the present study invalidated both research hypotheses. The results showed that G3 amine-containing resin cement has significantly lower color stability than the other tested groups with  $\Delta E_{ab} = 4.5 \pm 0.3$  and  $3.8 \pm 0.4$  and  $\Delta E_{00} = 3.5 \pm 0.3$  and  $3 \pm 0.3$  for the HT and LT ceramics, respectively, which is considered clinically unacceptable. This could be attributed to its chemical composition that includes amines in the benzoyl peroxide/amine redox initiator system and CQ-amine photo-initiator system. These amines are easily oxidized causing discoloration. Moreover, the oxidation of the residual monomers in addition to the presence of Bis-GMA monomer could affect color stability since they are highly susceptible to hydrolysis [34].

These results are consistent with a previous study in which color stability of Variolink N cement bonded to IPS e.max Press HT ceramic was evaluated after 384 h in an accelerated weathering machine, which correspond to one year of clinical service and recorded  $\Delta E = 4.7 \pm 0.7$  [32]. On the contrary, a study concluded that Variolink N showed clinically acceptable values of  $\Delta E = 2.37$  after 10,000 thermal cycles. This could be attributed to the difference in the specimen preparation protocol since they used the cement base paste only as a light cured cement which is free of benzoyl peroxide initiator [21].

On the other hand, there was no significant difference in  $\Delta E_{ab}$  values between the other two groups (G1 and G2). These groups showed higher color stability compared to G3. This

could be explained by the presence of dibenzoyl germanium derivative photo-initiator (Ivocerin), which is an amine-free redox initiator/activator system which improves material properties including color matching and stability as a result of low pigmentation due to shorter wavelength range absorption. It also does not require co-initiators and its color is not as yellow as a QC photo-initiator [31,33].

Previous studies showed that resin cements lacking benzoyl peroxide and an amine-redox initiator system (amine-free), have enhanced color stability therefore, Ivocerin is considered to be amine-free since it imparts the bleaching properties and enhanced color stability of composite resin [21,35]. The color stability results of the present study agreed with other previous studies that evaluated the amine-free resin cements containing Ivocerin as a photo-initiator. This enhancement was justified by the absence of amine and the positive effect of the Ivocerin photo-initiator [17,28,29].

The translucency of the ceramic specimens had a significant effect on the final color of the restorations, especially in very thin ceramic thickness of 0.3 mm as used in the present study [7]. Results of the current study revealed that HT ceramic specimens had significantly higher mean values of  $\Delta E$  than that of LT ceramic specimens in all tested groups. For the HT ceramic, both  $\Delta E_{ab}$  and  $\Delta E_{00}$  values were above the acceptability threshold in all cement types, that's why for the very thin veneers, HT is not

recommended since it could easily reflect the color change of the underneath resin cement [8].

Whereas the  $\Delta E_{ab}$  values of the LT ceramic were below 2.7 and therefore, considered clinically acceptable. However, the  $\Delta E_{00}$  values were still above 1.8 and according to the classification of mismatch, it was considered mismatch type (a), which is moderately unacceptable [26]. It has been reported that ceramic veneers with lower translucency promote higher masking ability of the darkened background than high translucency veneers. In addition, it was recommended that specified color matching standards for ceramic veneers are needed when the thickness of veneers is less than 0.7 mm [4].

For resin cements to reach their optimal physical and mechanical properties, the highest degree of conversion (DC) should be achieved. Inadequate polymerization of a resin-based luting agent is associated with problems such as postoperative sensitivity, recurrent caries, degradation, discoloration and decrease in mechanical properties. The unreacted monomer may leach from the polymerized material and irritate the soft tissue. Accordingly, DC was one of the essential parameters evaluated in the present study [38]. In the current study, degree of conversion was assessed using the ATR/FTIR since this technique provides rapid scanning, good resolution and accuracy [34]. To simulate the clinical situation, resin cements were polymerized through ceramic discs.

Time is an important factor for evaluation of the degree of conversion, especially for dual-cured resin cements to indicate the maximum DC achieved. In the current study, DC was assessed at three different time intervals: immediately after light curing, after 24 hours and after 2 weeks since auto-polymerization is reported to continue up to 2 weeks [14]. Results of the current study revealed that DC % significantly increased with time for all tested groups. Mean values recorded after 2 weeks were the highest followed by the 24 hours, while the immediate mean DC values showed the least values, as shown in Table V. These results were in agreement with a previous study [14].

Different cement types showed a significant effect on DC% with time ( $p < 0.001$ ). Immediately after curing, results showed that both G1 and G3 have significantly lower DC% compared to G2. This may be due to the difference in curing modes where both G1 and G3 are dual

cured cements that have a chemical activation part which need time to reach the maximum polymerization compared to G2, which is a light cured cement [34].

Results of the 24 hours and 2 weeks revealed no significant differences between G1 and G2, however, both groups have significantly higher DC% than G3. This could be explained by the difference in chemical composition of the tested resin cements. Groups G1 & G2 contain a novel dibenzoyl germanium derivative photo-initiator (Ivocerin), thus they exhibited a significantly higher DC % than that of the conventional CQ- amine photo-initiator in G3. These results are consistent with previous studies [15,34]. Ivocerin has low energy bonds, which after cleavage, yields more active radicals and allow photo-polymerization by high energy, short wavelength of light curing units and do not require amine co-initiators which can improve material properties in dental resins [33]. The incorporation of this photo-initiator into bulk fill composite resin has resulted in increased DC, improved reactivity to curing light and greater depth of cure [35].

A study evaluated the effect of different photo-initiator systems on the efficiency of polymerization of resin cements through ceramic veneers and concluded that Ivocerin alone or associated with TPO was shown to be an effective alternative photo-initiator to substitute CQ. This is consistent with our results and with other previous studies [31,34].

In the literature, DC% has been reported to be influenced by the translucency and thicknesses of ceramic restorations. However, in the current study, ceramic translucency had no significant effect on the DC% at different time intervals and for different types of resin cements. This could be attributed to the thin ceramic thickness (0.3 mm) used in the present study which could not result in significant light attenuation to jeopardize the polymerization reaction [41]. The results of the current study agree with previous studies where they concluded that in up to or less than 1 mm thick ceramic, the DC showed similar behavior between dual-cured and light-cured resin cements [38,40].

Based on the results obtained in the present study, it could be highlighted that the final esthetic outcome of thin ceramic veneers could be affected by the type of resin cement and ceramic translucency. Additionally, the

polymerization quality of different resin cements is influenced by the modifications in the photo-initiator formulation and curing mechanisms. Nevertheless, further investigations are required using different resin cement/ceramic materials combinations with different thicknesses.

## CONCLUSIONS

Within the limitations of the present study, it was concluded that:

- 1- The first hypothesis was rejected since both amine free dual cured and light cured resin cements with the Ivocerin photo-initiator system exhibited better color stability and showed higher degree of conversion than the conventional photo-initiator.
- 2- The second hypothesis was also rejected since the ceramic translucency had a significant effect on color stability were HT ceramics revealed less color stability than LT ceramics.

## Author's Contributions

NMM, AEB, DIEK: Conceptualization. NMM, AEB: Methodology and Validation. NMM: Investigation and Resources. NMM, AEB, DIEK: Data Curation. NMM: Writing- Original Draft Preparation. AEB, DIEK: Writing- Review & Editing. NMM, AEB, DIEK: Visualization. AEB, DIEK: Supervision. NMM: Project Administrator.

## Conflict of Interest

The authors declare that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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## Regulatory Statement

This study was conducted and approved by the ethical committee of Faculty of Dentistry Ain Shams University with ethical approval number FDASU-RecEM 011934.

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