

Physical properties of two bis-acryl interim materials: color stability, flexural strength and shear bond strength to flowable composite resin as add-on material

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Aims: The objective of this study was to evaluate the mechanical properties of two bis-acryl interim resin materials, such as color stability, flexural strength and shear bond strength to flowable composite resin, simulating clinical situations when this material has to be used for repair as add-on. Two shades of two bis-acryl interim resin materials [Structur 2 SC (shades Bleach and A2); Protemp 4 (shades A1 and A2)] were evaluated. Discs (5 x 1 mm) were fabricated and baseline color was determined after 1 h. Ten specimens were immersed at 37°C in solutions of distilled water (control) and cola-based soft drink (Coca-Cola). Color measurements were performed with a spectrophotometer using CIELab parameters. Color readings were again measured after 2 hours, 4 hours, 24 hours and 7 days. Flexural strength was determined using the three-point bending test (10 x 1 x 2 mm) on a universal testing machine (0.5 mm/min) (n = 10). Discs of bis-acryl resin were embedded in acrylic resin, planned and distributed in 2 groups: G1 - Filtek Z350 Flow/Protemp4 and G2 - Grandio SO Flow/Structure 3 (n = 15). Cylinders (3.5 x 2 mm) were made with the flowable composite resins and polymerized for 20 s. The specimens were stored in distilled water at 37°C for 24 h and subjected to shear bond strength test. Data were analyzed using one-way ANOVA and Tukey's test ($\alpha = 0.05$). ΔE values were higher for Structur Bleach (3.08)^a compared with Protemp 4 (shade A1, 2.22)^b (shade A2, 2.25)^b. There were no significant differences between Structur Bleach and Structur A2 (2.62)^{ab}. Coca-Cola presented higher ΔE values (3.08)^a than (2.00)^b. Regarding time,

ΔE values increased from 1.84^a after 2 h to 2.31^b after 4 h. The higher values were observed after 24 h and 7 days (2.93^c and 3.09^d, respectively). No significant differences were observed for the flexural strength of Structur (22.05 MPa)^a and Protemp 4 (19.01 MPa)^a. The repairs executed with Structur/Grandio flow (9.21 MPa)^a were similar to those performed with Protemp 4/Z350XT flow (10.71 MPa)^a. It can be concluded that the two bis-acryl resins evaluated showed similar physical and mechanical properties.

Keywords: mechanical properties, provisional materials, shear strength, flexural strength, color stability.

Introduction

Bis-acryl interim resin materials have been extensively applied for prosthetic treatments. Properties such as fastness of set process, ease of handling and aesthetic quality of these materials allow its use in several clinical situations during prosthetic rehabilitation, such as temporary restorations and construction of dental mock up for aesthetic and functional evaluations. As interim material, it is essential that they remain stable over the period of rehabilitation. In order to achieve these purposes, they should present good mechanical properties and good color stability¹⁻⁴.

In the case of aesthetic rehabilitations, such as anterior interim restorations, the color stability of bis-acryl interim resin may be considered one of the most important factors for the clinical success. Different composite resins or different luting cements present distinctive degree of the color change^{5,6}. Thus, different bis-acryl interim resin materials may also present distinctive color stability. Color changes over time due to the use of beverages with staining potential (e.g., water, wine, and cola-based soft drink) have been previously described to resin-based materials^{5,6}. In addition, inherent properties of different bis-acryl materials can interfere in their color stability. Materials with smoother surfaces and less hydrophilicity tend to present less sorption of staining solutions, therefore, present better color stability⁷. In this way, the effect of different solutions with staining potential on bis-acryl interim resin must be evaluated.

In some clinical situations, interim restorations need to stay in function for a longer period than originally planned. Besides color stability, another important aspect in these cases of long-span interim restoration is their flexural strength⁴. Low flexural strength can lead to fractures, resulting in functional and esthetic problems. However, there is a lack of sufficient information about the mechanical properties of bis-acryl interim resin materials⁴.

Additionally, bis-acryl interim resins may need add-on material for repair, modification of form or reduce discrepancies at the margin⁸. According to the manufacturer recommendation and Lee and Lee⁸ (2015), bis-acryl-based interim material has favorable add-on properties when used with a flowable composite resin.

Thus, the aim of this study is to evaluate the mechanical properties of two bis-acryl interim resin materials, such as color stability, flexural strength and shear bond strength

to flowable composite resin, simulating clinical situations when this material has to be used for repair as add-on. The hypotheses evaluated were: i) there would be differences between the two bis-acryl materials evaluated regarding color stability, flexural strength and shear bond strength to flowable composite resin; and ii) immersion in different staining solution would induce color changes in the two bis-aryl resins.

Material and Methods

The materials used in this study and their composition are described in Table 1. Optical and mechanical properties of two bis-acryl materials were evaluated by three steps: evaluation of color stability after immersed in different storage solutions, by means of a spectrophotometer; three bending point assay to test flexural strength and shear bond strength between the bis-acryl materials and two flowable composites.

Color Stability

Two shades of two bis-acryl interim resin materials [Structur 2 SC (shades Bleach and A2) VOCO, Germany; Protemp 4 (shades A1 and A2) 3M ESPE, St. Paul, MN, USA] were evaluated in this study.

All specimens were prepared at the same laboratory with controlled humidity ($55 \pm 5\%$), temperature ($23 \pm 1^\circ\text{C}$) and illumination conditions. Twenty disk specimens (5 mm in diameter and 1 mm in thickness) were prepared for each shade and material. Each specimen was made by inserting the bis-acryl resin in a Teflon mold ring and pressed between two 1-mm-thick glass slides (separated by Mylar strips) under finger pressure. All samples were prepared according manufacturers' instructions (4 minutes). The samples were subjected to polishing procedures as described by the manufacturers.

The color measurements were performed with a spectrophotometer (EasyShade Advance, Vita Zahnfabrik, Bad Sackingen, Germany) according to the CIELab coordinates, under a standardized white background. Afterwards, ten specimens were immersed at 37°C in solutions of distilled water (control) and cola-based soft drink (Coca-Cola). The specimens were then stored in dark canisters containing water or Coca-Cola at 37°C , and the color parameters were again measured after 2 hours, 4 hours, 24 hours and 7 days. The CIELab coordinates were used to calculate the color difference (ΔE) between the "before" (baseline) and "after" periods.

Table 1. Compositions of materials used in this study

Material	Composition
Protemp 4 (3M ESPE, USA)	Bis-GMA, Dimethacrylate polymer, zirconia silica, fumed silica, silane
Structur 2 SC (Voco, Germany)	Bis-GMA, BHT, amines, benzoyl peroxide, dimethacrylates, glass particles
Filtek Z350 (3M Espe, USA)	BisGMA, BisEMA, UDMA, TEGDMA, sílica, zirconia, clusters, zirconia/silica aggregated particles
Grandio SO Flow (Voco, Germany)	Bis-GMA, Bis-EMA, TEGDMA, glass ceramic, functionalized SiO_2 nano-particles

Before each color measurement, the disks were ultrasonic cleaned for 60 seconds and then specimens were dried with absorbent paper. The ΔE for each experimental time was calculated using the equation:

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

where ΔL , Δa and Δb are the differences in the respective values before and after aging. The results of ΔE were analyzed by three-way ANOVA with repeated measures (material, solution and time) and Tukey's HSD test ($\alpha = 0.05$).

Flexural Strength

Ten specimens were made for each interim restorative material for the evaluation of the flexural strength. A split stainless steel was used to produce the specimens with 10 x 2 x 1 mm. Bis-acryl resins were injected into the mold according, as described previously for disks. Excess material was removed, the specimens were polished and then stored in distilled water at 37°C for 24 hours. The flexural strength was determined using the three-point bending test on a universal testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil) at a crosshead speed 0.5 mm/min. The flexural strength (S) in MPa was calculated by the following formula:

$$S = 3Fl/2bh^2$$

where F is fracture load (N), l is the span length (6 mm), b and h are, respectively, the width and height of the specimen (mm). The results of the flexural strength were analyzed by Student's t test ($\alpha = 0.05$).

Shear Bond Strength

Thirty bis-acryl specimens of 6 mm of diameter and 2 mm of thickness were fabricated, as described previously (n = 15 for Structur and Protemp4). After polishing, the specimens were embedded in PVC cylinders with acrylic resin (Jet, Artigos Odontológicos Clássico, São Paulo, Brazil). A Teflon matrix (3.5 mm in diameter and 1 mm in thickness) were placed onto the bis-acryl resin surface and filled with two flowable composite resins (shade A2, Grandio Flow, for Structur samples and Filtek Z350 XT Flow for Protemp samples, 3M ESPE, St. Paul, MN, USA). The specimens were light-cured through for 20 s with a LED curing unit (Poly Wireless, Kavo, Joinville, SC, Brazil) at 1100 mW/cm². Before adding the flowable resins on bis-acryl specimens, no surface treatment was performed. The specimens were then stored in distilled water at 37°C for 24 h. After the storage period, the specimens were submitted to shear bond strength test in a universal testing machine at a crosshead speed of 0.5 mm/min. Data were analyzed using Student's t test ($\alpha = 0.05$).

Results

Color Stability

Means and standard deviations for ΔE values are presented in Table 2. Significant differences were observed for material (p = 0.008822), solution (p = 0.000001) and time (p = 0.000001). All double (p < 0.0001) and triple (p = 0.040033) interactions were also significant. ΔE values were higher for Structur Bleach ($\Delta E = 3.08 \pm 2.27$)^a compared with

Table 2. Means and standard deviations for ΔE values.

Material	Solution	ΔE			
		2 h	4 h	24 h	7 days
Structur Bleach	Water	1.18±0.46 ^{de}	0.97±0.72 ^e	2.32±1.26 ^{bcd}	1.61±0.93 ^{cde}
	Coca-Cola	2.25±0.53 ^{bcd}	3.92±1.42 ^b	6.51±1.66 ^a	5.91±1.29 ^a
Structur A2	Water	2.60±1.77 ^{bcd}	2.91±1.80 ^{bcd}	1.77±0.73 ^{bcd}	3.15±1.12 ^{bcd}
	Coca-Cola	1.70±0.84 ^{cde}	2.21±0.63 ^{bcd}	3.15±1.56 ^{bcd}	3.46±2.19 ^{bc}
Protemp A1	Water	1.34±0.75 ^{cde}	1.65±0.54 ^{cde}	1.75±0.6 ^{bcd}	1.80±0.4 ^{bcd}
	Coca-Cola	1.99±1.20 ^{bcd}	2.78±1.0 ^{bcd}	3.10±0.78 ^{bcd}	3.41±0.9 ^{bc}
Protemp A2	Water	2.04±0.65 ^{bcd}	1.84±0.79 ^{bcd}	2.37±0.93 ^{bcd}	2.77±0.94 ^{bcd}
	Coca-cola	1.65±1.05 ^{cde}	2.25±0.94 ^{bcd}	2.47±0.79 ^{bcd}	2.65±0.84 ^{bcd}

*Means follow by different superscript letter are significantly different ($p < 0.05$).

Protemp 4 (shade A1, $\Delta E = 2.23 \pm 1.04$)^b (shade A2, $\Delta E = 2.26 \pm 0.86$)^b. There were no significant differences between Structur Bleach and Structur A2 ($\Delta E = 2.62 \pm 1.48$)^{ab}. It was observed that Coca-Cola presented higher ΔE values ($\Delta E = 3.08 \pm 1.85$)^a when compared to water ($\Delta E = 2.00 \pm 1.09$)^b. When the storage times were considered, the mean ΔE values increased from 1.84 ± 1.03 ^a after 2 hours to 2.32 ± 1.30 ^b after 4 hours. The higher values were observed after 24 hours and 7 days ($\Delta E = 2.93 \pm 1.78$ ^c) and 3.10 ± 1.65 ^d, respectively.

Flexural Strength

Student's t-test showed no significant difference between the flexural strength of Structur and Protemp4 ($p = 0.115$). The Structur flexural strength (22.05 ± 5.71 MPa) was similar to the one presented by Protemp 4 (19.01 ± 3.06 MPa).

Shear Bond Strength

There were no statistically significant differences between the materials tested ($p = 0.228$). The repairs executed with Structur/Grandio flow (9.21 ± 3.72 MPa)^a were similar to those performed with Protemp4/Z350XT flow (10.71 ± 2.86 MPa)^a. All the failures were adhesive.

Discussion

The hypotheses tested about color stability in the present study were accepted. There were significant differences between the shades evaluated, the immersion solutions used and different periods of time.

The staining of dental materials is the result of both extrinsic and intrinsic factors⁹. Cola-based soft drinks, wine, tea and coffee, for example, are commonly consumed beverages and other studies have demonstrated the discoloration of composite materials upon exposure to these solutions⁹⁻¹¹. Although the immersion for longer periods of time does not represent a clinical situation, this method provides a better understanding of the staining potencial of several solutions and color stability of materials subjected to immersion. Other studies used this method to verify color stability of materials¹⁰⁻¹³. In the present study, immersion in water and cola-based soft drink caused changes in color of both materials studied, however, the color

changed was significantly higher for the cola-based drink. The results of this study indicated that the dietary habits of the patient could present a potential discoloration risk to provisional restorations. Also, the water absorbed by the resin matrix can cause filler matrix debonding and hydrolytic degradation of the material^{14,15}. However, in the present study, the two bis-acryl materials tested demonstrated no significant differences for color change.

The effect of shades on the color stability of composite resins and luting cements was previously described^{6,15-18}. In the present study, the color change for the materials with lighter and less chromatic shades was higher. These results corroborate with those found by Uchida et al.¹⁷ (1998). These authors, by means of a quantitative analysis of color stability, verified a higher ΔE for the lighter shades of two composites. This study suggests that this fact may result from two factors: (a) discoloration through environmental breakdown of the polymer leading to release of monomers and the shift of color from the cured resin to that of the monomers and (b) the environmental effect on the retention and/or stability of pigments and other additives in the polymer formulations. The environmental effect on the pigments and other additives need clarification through additional research.

ΔE values showed a tendency to increase as immersion period increased, suggesting that the color of the material would tend to change over long-term clinical use^{6,15}. However, all the values reported color changes with ΔE values lower than the 3.3 threshold, being clinically acceptable¹⁹.

The flexural strength of an interim restorative material is considerably tested during mastication. Acceptable flexural strength is crucial to avoid repair procedures that can be time consuming or the fracture of these restorations can lead to functional and aesthetic problems. This factor is especially important in multiple-unit or long-span prosthesis, whose pontics are constantly submitted to flexural tensions during function⁴. In the present study, the materials tested presented similar acceptable flexural strength. Flexural strength test usually follow the ISO 4049 standard that state specimen with 25 x 2 x 2 mm. The dimensions used in the present study are different from the ISO, however also observed in prior studies²⁰⁻²². Other dimensions are also used in the literature, such as Vieira et al.²² (2012) and Firoozmand and Pagani²³ (2009).

Distinctive methods can evaluate the bond strength to determine the adhesion between different materials, such as microtensile, microshear or shear bond strength²⁴. Besides its irregular distribution of stress within the surface, shear bond strength tests are easier to perform when compared to microtensile and micro shear assays. Similarly to many studies^{8,25-28}, here, the shear bond strength method was used to determine the bond strength between bis-acryl interim resin materials and flowable composite resin, simulating an add-on repair or modification of form. This method has the advantage to mimic the clinical condition very closely because it results in stress on the interface between the materials^{8,24-28}. This study showed no differences between the two materials tested, regarding bond strength. Also, additional treatments have been proposed to improve the bond strength of these materials, such as, applying bonding agent or additional light polymerization⁸. Lee and Lee⁸ (2015), verified that the use of those methods increased the shear bond strength of add-on materials to bis-acryl resins when compared to the untreated specimens.

However, other studies are still necessary to corroborate the clinical use of these strategies, particularly in cases of interim restorations used in oral rehabilitation that remain in function for long periods of time.

This study has a number of limitations, mostly related to the conditions represented. In order to promote a better clinical correlation of the outcomes, more variables should be assessed, such as thermocycling of optical and mechanical properties specimens and immersion in solutions with different pH. In addition, different polishing and finishing protocols can be evaluated to identify the changes in color stability of bis-acryl resins. Thus, future studies must be performed to evaluate the clinical performance of these materials.

Therefore, based on the results of the present study, it can be concluded that the color stability of the bis-acryl resin tests decreased with storage time and storing in cola-based soft drink. There was no difference between materials regarding flexural strength and shear bond strength.

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