# Flexural strengths and color stability of bis-acryl resin materials for provisional restorations

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Objectives: This study evaluated the flexural strength and color stability of 3 bis-acryl resins (Luxatemp Fluorescence<sup>TM</sup>, Protemp  $4^{TM}$  and Integrity<sup>TM</sup>) and a polymethyl methacrylate-based resin (Unifast Trad<sup>TM</sup>) after different aging conditions.

Materials and Methods: Twenty bar-shaped specimens of each material were prepared for flexural strength test performed at 30 minutes after mixing, 24 hour and 7 day water storage with or without thermocycling. For color stability test, color parameters were measured on 20 disc-shaped specimens, 5 specimens for each material, at the same aging conditions.

Results: All bis-acryl resins showed low flexural strengths at 30 minutes after mixing but increased after 7 day water storage with or without thermocycling. Polymethyl methacrylate-based resin showed similar flexural strengths at all-time intervals. Color differences ( $\Delta E$ ) of all bis-acryl resins were less than that of polymethyl methacrylate-based resin. The greatest  $\Delta E$  was observed in a polymethyl methacrylate-based resin after 24 hour water storage. No significant differences of  $\Delta E$  among bis-acryl resins were detected in all conditions. Conclusions: Bis-acryl resins showed higher flexural strengths and less  $\Delta E$  than polymethyl methacrylate-based resin. Thermocycling did not alter flexural strengths and color changes of these materials.

**Keywords**: acrylic resin, bis-acryl resin, color stability, flexural strength, provisional material, polymethyl methacrylate

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## Introduction

Provisional or interim restorations should provide patient comfort, function and a good periodontal health, and also stabilize occlusal relationship and aesthetics [1-2]. Acrylic resins have been used as provisional restorations for decades. They are classified into two groups: poly (methyl methacrylate) (PMMA) and poly (ethyl

methacrylate) (PEMA). Their delivery systems are commonly in powder/liquid form that requires a hand-mixing of the two components. Although these materials provide an acceptable color stability, esthetics and relatively low expense; they are difficult to manipulate [2]. In addition, there was an exothermic temperature release, especially for PMMA, that possibly caused pulpal and tissue damage [3].

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In recent years, resin composites have been introduced and developed to overcome some disadvantages of acrylic resins for provisional restorations. The chemistry of provisional resin composites is bis-acryl-methacrylate-based resin (e.g. dimethacrylate bis-GMA, or urethane resins or resin containing at least 2 acrylic groups in the monomer). They were used in forms of light-cured, self-cured, or dual-cured. These materials are dispensed in the double-barrel cartridge where the catalyst and base pastes are mixed in automixing tips [4]. This cartridge delivery method was not only convenient but also more accurate and consistent mix [5].

Clinicians usually select a provisional material based on factors that include ease of manipulation, cost effectiveness, esthetics, strength, and marginal accuracy. Among mechanical properties, the flexural strength is one of the important properties of the provisional restoration, particularly in a long-span provisional prosthesis with short pontic height and connectors. Additionally, in the patients with parafunctional habits such as bruxism and/or clenching and the patients using provisional restorations for a long period of time (e.g. full mouth reconstruction), [6] high strength provisional restorations are required. Provisional restorations prepared chair-side by direct technique are usually cemented and exposed to occlusal forces immediately after fabrication. It is important that the provisional materials should have sufficient strength shortly after the initial polymerization to prevent failure of the restorations [7-8]. Several studies reported that flexural strengths of provisional materials depended on time after mixing [7-8] and materialspecificity rather than category-specificity [7, 9-10]. Flexural strengths of the bis-acryl-methacrylatebased resins at 24 hours after mixing have been reported to be in a range of 51.8-110.1 MPa [7-8].

Color stability of provisional restorations is a critical concern especially in the esthetic zone. The provisional material should provide an initial shade match and maintain the selected shade over the service period [11]. Discoloration of the provisional restorations in esthetic area may lead to patient dissatisfaction, additional expense and time for fixation [12]. Several studies have shown varying degree of discoloration of bis-acryl provisional resins [11-16]. Most of the studies were conducted to expose the provisional materials to external colorants such as drinks, foods or oral rinses or to subject the materials to accelerated aging conditions.

Frequently, aging protocols used for color stability study of provisional materials are immersion of specimens in 60°C water for 1 month [17] or exposure of specimens to UV light [11-12]. These methods seem to be appropriate for accelerated color change of long-term used materials such as restorative resin composites, but might be inappropriate for relatively short-term used provisional materials. Thermocycling has also been used for aging of restorative resin composite specimens in color stability test [18] or mechanical property test [8]. However, no information is available regarding the effect of thermocycling on color stability of provisional resin materials.

The purpose of this study was to evaluate the flexural strength and color stability of 3 bis-acryl resins and a methyl/ethyl methacrylate-based resin used as provisional restorations after different storage time and after aging with 5,000 cycles of thermocycling. The null hypothesis of this study was that the flexural strengths and color changes were not affected by type of materials, and the storage times including thermocycling.

#### Materials and methods

## I. Specimen fabrication for flexural strength test

Eighty bar-shaped specimens (25x2x2 mm), 20 specimens for each provisional material, were prepared using a stainless steel split mold according to ISO 4049 [19]. The materials used in this study are chemically-polymerized type (Table 1).

Table 1 The materials used in the study

Material	Manufacturer	Composition	Shade	Lot number
Unifast Trad <sup>a</sup>	GC America, IL, USA	Powder: Methyl methacrylate & Ethyl methacrylate copolymer  Liquid: Methyl methacrylate,  N,N dimethyl-p-toluidine	lvory	Powder: 1208081 Liquid: 1208022
Luxatemp Fluorescence <sup>b</sup>	DMG, Hamburg, Germany	Glass powder and silica, Urethane dimethacrylate, Aromatic dimethacrylate, Glycol methacrylate	A2	704894
Protemp 4 <sup>b</sup>	3M ESPE, Seefeld, Germany	Catalyst paste: Ethanol,  2,2'-[(1-methylethylidene)bis (4,1 phenyleneoxy)]bis-,diacetate, Benzyl-phenyl-barbituric acid, Silane treated silica, Tert-butyl peroxy-3,5,5-trimethylhexanoate Base paste: Dimethacrylate (BISEMA6), Silane treated amorphous silica, Reaction products of 1,6-diisocyanatohexane with 2-[(2-methacryloyl)ethyl] 6-hydroxyhexanoate and 2-hydroxyethylmethacrylate (DESMA), Silane treated silica	A2	530568
Integrity <sup>b</sup>	Dentsply Caulk, DE, USA	Barium boron alumino silicate glass, Hydrophobic amorphous fumed silica, Polymerizable dimethacrylate resins	A2	130708

<sup>&</sup>lt;sup>a</sup> Methacrylate resin: powder-liquid hand-mix

For bis-acryl specimens, each provisional material was dispensed using the manufacturer's mixing tip and directly syringed, with slightly overfilled, into the mold which placed on a glass plate. For methacrylate-based resin, powder was weighed using a micro-balance (Mettler-Toledo AE163, Giessen, Germany) and liquid was measured using a Nichiryo Nichipet micropipette (100 - 1000 μl; Nichiryo America, MO, U.S.A.). The powder / liquid ratio is 1g of powder to 0.5mL of liquid. The liquid was dispensed into a resin-mix bowl (Scheu Dental, Iserlohn, Germany) containing the weighted powder and then hand-mixing was performed and the material was dispensed into

the mold using a stainless steel spatula. After material placement, another glass plate was placed upon each unset material and the plates were clamped together with four clamps.

Once the material set, the mold was disassembled prior to ejection of the specimens. Any flash at the border of specimen was removed by gently abrading with 320-grit silicon carbide paper [19]. The specimens were kept dry at room temperature for 30 min. After that, 20 specimens of each material were randomly divided into 4 groups of 5 specimens each and assigned to 4 different aging conditions as shown in Table 2.

<sup>&</sup>lt;sup>b</sup> Bis-acryl resin: paste-paste auto-mix

Time after mixing	Water storage	Time / conditions
30 min	None	30 minutes, 23°C
24 h	$\sqrt{}$	24 hours, 37°C
7 d	$\sqrt{}$	7 days, 37°C
7 d, TC	$\sqrt{}$	7 days, thermocycling
		(5000 cycles; 5-55°C; 15 s dwell time)

Storage times / conditions after mixing before flexural strength and color stability testing

## II. Specimen fabrication for color stability test

Twenty disk-shaped specimens (10 mm in diameter, 2 mm in thickness) were prepared from 4 provisional materials, 5 specimens each, using a stainless steel mold. The materials were applied into the mold in the same way as previously mentioned in the fabrication for flexural strength test. To achieve standardized smooth surfaces, the set specimens were wet-ground using 2,500grit silicon carbide abrasive paper for 10 times, then rinsed with distilled water to remove any debris and wiped dry. After that, the specimens were measured for color at 30 min after mixing (baseline-BL). Then the specimens were continuously immersed in distilled water and color measurement was performed after each aging condition as shown in Table 2.

## III. Material Testing

#### a) Flexural strength test

After each aging condition, the specimens were removed from the water bath. Remaining water on the specimen surfaces was removed by tissue paper and left dry in air for 5 min. The bar-shaped specimen width and height were measured by micrometer screw (Mitutoyo, Kawasaki, Japan) for flexural strength calculation. Specimens were loaded on a universal testing machine (Instron<sup>®</sup> 5565, Instron Corp., Canton, Mass, USA) for three-point bending test with a crosshead speed of 1 mm per minute and a support separation of 20 mm (Figure 1).

The forces at fracture were recorded in Newton and calculated for flexural strength in

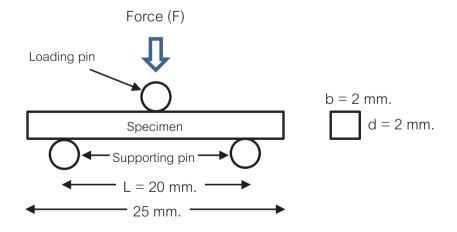


Figure 1 Three-point bending test

MPa using the following formula by the testing machine software [20]

FS=3FL/2bd<sup>2</sup>

Where FS=Flexural strength (MPa), F=Load at break or yield (N), L=Distance between supports (mm), b=Width of the specimen (mm) and d=Thickness of the specimen (mm)

### b) Color stability Test

Baseline color of each material was recorded before water storage at the time of 30 min after mixing using imaging spectrophotometer [SpectroShade Micro<sup>™</sup>; MHT S.p.A., Via Milano, Arbizzano di Negrar (VR), ITALIA]. Each specimen was placed against a white background and color was measured at the same central point for 3 times. The image of each measurement was captured with a digital camera/LED spectrophotometer combination and analyzed for L\*, a\*, and b\* values using SpectroShade software version 2.40 (Microsoft Corporation).

After each storage period and condition, color of the specimen was measured using the imaging spectrophotometer as described earlier. Color change ( $\Delta E$ ) was calculated as the color difference between the color at baseline and at different storage times and conditions using the following formula.

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

Where  $\Delta \mathsf{E}$  represents the color difference in all dimensions (L\*a\*b\*), and  $\Delta$ L\*,  $\Delta$ a\*, and  $\Delta$ b\* represent color difference along the individual axes.

#### IV. Statistical analysis

The mean and standard deviation of the flexural strengths and the color difference values  $(\Delta E)$  were calculated and subjected to the Kolmogorov-Smirnov test to check for normal distribution (p>0.05). Levene test was performed to check the homogeneity of variances (p>0.05). Flexural strength data were analyzed using twoway ANOVA test (p<0.05) to identify significant differences between the materials and different

aging conditions, followed by Tukey HSD multiple comparison test (p<0.05) to identify dependent variables contributing to the flexural strength values.

The mean color difference values ( $\Delta E$ ),  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  were analyzed using two-way ANOVA test (p<0.05) to identify significant differences between materials and different aging times and conditions. The  $\Delta E$ ,  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$ values were subjected to Tukey HSD multiple comparison test (p<0.05) to identify dependent variables contributing to the color change.

## Results

The mean values and standard deviations of the flexural strengths are shown in Table 3.

Two-way ANOVA test (p<0.05) indicated that materials, aging conditions, and interaction between "materials" and "aging conditions" affected the flexural strengths of provisional materials. Unifast Trad showed no significant differences of the flexural strengths at any aging conditions, whereas the flexural strengths of Protemp 4, Luxatemp Fluorescence, and Integrity at 30 min after mixing were significantly lower than those at other aging conditions. At the time of 30 minutes after mixing, the flexural strength of Unifast Trad was significantly higher than those of the bis-acryl resin materials tested, contrary to after 7 days and thermocycling which were significantly lower. In addition, Unifast Trad showed significantly lower flexural strength than Protemp 4 and Integrity after 24 h and 7 d water storage respectively. There were no significant differences of the flexural strengths among bis-acryl resin materials at all aging conditions. After 7 d water storage with and without thermocycling, Integrity showed the highest flexural strength values in contrast to Unifast Trad which showed the lowest strength.

Mean Flexural strengths (MPa) and standard deviations (in parentheses) after different storage times/ Table 3 conditions

Materials	Times and conditions			
	30 min	24 h	7 d	7 d, TC
Unifast Triad	55.68 (4.03) <sup>bc</sup>	69.88 (8.33) <sup>bcde</sup>	69.84 (10.89) <sup>bcd</sup>	60.79 (3.60) <sup>bc</sup>
Protemp 4	23.36 (2.88) <sup>a</sup>	94.65 (4.87) <sup>f</sup>	89.51 (6.85) <sup>def</sup>	93.10 (5.78) <sup>def</sup>
Luxatemp Fluorescence	19.66 (1.29) <sup>a</sup>	81.25 (11.37) <sup>bcdef</sup>	79.89 (16.88) <sup>bcdef</sup>	93.27 (9.84) <sup>def</sup>
Integrity	19.39 (3.89) <sup>a</sup>	89.63 (10.69) <sup>cdef</sup>	109.95 (6.11) <sup>f</sup>	101.71 (12.47) <sup>def</sup>

Different superscript letters denote significant difference (two-way ANOVA test: p<0.05)

The mean color difference values ( $\Delta E$ ) and standard deviations are shown in Table 4.

Two-way ANOVA test (p<0.05) indicated materials, aging conditions, and interaction between "materials" and "aging conditions" affected the color change of the provisional materials. Unifast Trad showed the largest color change after 37°C water immersion for 24 h and significantly decreased in color change after 7 d water storage with and without thermocycling. All bis-acryl resins exhibited low color difference values and there were no significant differences of color change after 24 h and 7 d water storage with and without thermocycling compared to baseline color. After 24 h water storage, Unifast Trad showed significantly higher in color difference value than all bis-acryl resins and there were no significant color difference values among bis-acryl resin materials. Luxatemp Fluorescence and Integrity showed significantly lower color difference values compared to Unifast Trad after 7 d water storage and only Luxatemp Fluorescence showed significantly lower color change than Unifast Trad after 7 days with thermocycling.

The mean values and standard deviations of  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  are shown in Table 5.

All provisional materials showed lower L\* values after 24 h and 7 d water storage with and without thermocycling compared to baseline (negative  $\Delta L^*$  values). Unifast Trad showed the largest  $\Delta L^*$  values after 24 h storage in 37°C water and decreased in  $\Delta L^*$  values after 7 d water storage with and without thermocycling. The a\* values of Unifast Trad after all aging conditions were higher than baseline (positive  $\Delta a^*$  values). On the contrary, all bis-acryl resins showed lower a\* values than baseline (negative a\* values). The  $\Delta b^*$  values of Unifast Trad after all aging conditions were higher than baseline (positive  $\Delta b^*$  values). The  $\Delta b^*$  values of all bis-acryl resin materials were significantly lower than that of Unifast Trad and there were no statistically significant differences of  $\Delta b^*$  values among bisacryl resin materials. Two-way ANOVA (p<0.05) indicated that materials, aging conditions, and interaction between "materials" and "aging conditions" affected  $\Delta L^*$  and  $\Delta a^*$  values of the provisional materials, whereas only materials and interaction between "materials" and "aging conditions" have influenced on  $\Delta b^*$  value.

Table 4 Mean color difference values (ΔE) and standard deviations (in parentheses) after different storage times/ conditions compared to baseline color (30 min after mixing)

Materials	Times / conditions			
	24 h	7 d	7 d, TC	
Unifast Triad	5.40 (0.62) <sup>e</sup>	2.35 (0.47) <sup>d</sup>	2.43 (0.62) <sup>d</sup>	
Protemp 4	1.09 (0.70) <sup>abc</sup>	1.59 (0.42) <sup>abcd</sup>	1.79 (0.12) <sup>cd</sup>	
Luxatemp Fluorescence	0.60 (0.24) <sup>a</sup>	0.74 (0.26) <sup>ab</sup>	1.28 (0.31) <sup>abc</sup>	
Integrity	0.83 (0.33) <sup>abc</sup>	1.20 (0.41) <sup>abc</sup>	1.70 (0.65) <sup>bcd</sup>	

Different superscript letters denote significant difference (two-way ANOVA test: p<0.05).

Table 5 Mean values and standard deviations (in parentheses) of  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  evaluated after different storage times/conditions compared to baseline color (30 min after mixing)

Materials	Times and conditions	ΔL*	∆a*	Δb*
Unifast Triad	24h	-4.35 (0.41) <sup>d</sup>	2.33 (0.31) <sup>a</sup>	2.12 (0.77) <sup>a</sup>
	7d	-1.53 (0.44) bc	0.47 (0.18) ab	1.68 (0.37) <sup>a</sup>
	7 d, TC	-1.55 (0.55) bc	0.25 (0.17) bc	1.82 (0.50) <sup>a</sup>
Protemp 4	24h	-1.03 (0.67) abc	-0.07 (0.14) <sup>cd</sup>	0.25 (0.28) b
	7d	-1.13 (0.59) abc	-0.95 (0.28) <sup>e</sup>	-0.03 (0.46) <sup>b</sup>
	7 d, TC	-1.13 (0.31) abc	-1.06 (0.21) <sup>e</sup>	-0.63 (0.64) <sup>b</sup>
	24h	-0.35 (0.25) <sup>a</sup>	-0.17 (0.10) <sup>d</sup>	-0.35 (0.29) <sup>b</sup>
Luxatemp	7d	-0.59 (0.28) ab	-0.09 (0.09) <sup>cd</sup>	0.07 (0.45) b
Fluorescence	7 d, TC	-1.19 (0.37) abc	-0.14 (0.15) <sup>cd</sup>	0.15 (0.41) <sup>b</sup>
Integrity	24h	-0.61 (0.36) ab	-0.26 (0.08) <sup>d</sup>	-0.37 (0.33) <sup>b</sup>
	7d	-1.17 (0.43) abc	0.00 (0.12) <sup>cd</sup>	-0.02 (0.27) <sup>b</sup>
	7 d, TC	-1.63 (0.66) °	-0.14 (0.19) <sup>cd</sup>	-0.05 (0.46) b

Different superscript letters denote significant difference (two-way ANOVA test: p<0.05).

## Discussion

Flexural strength is an important property of provisional restorations for crowns and bridges [6]. Clinically, provisional restorations are usually finished and cemented to the abutment shortly after setting and then initially exposed to oral environment, temperature change and occlusal loading along the service period. Therefore, the strength should be sufficient at an early stage of

setting and sustainable to prevent failure of the restorations until the final restorations have been finished and placed. This study tested the flexural strengths of materials at 30 min after mixing to simulate the time interval of the fabrication for provisional restorations. Other storage conditions aimed to simulate the clinical situation from days to weeks using different water storage times and thermocycling.

All provisional resin materials tested in this study showed flexural strengths greater than the limit specified in ISO 4049, 50 MPa at 24 hour after mixing [19]. All bis-acryl resins showed significantly lower flexural strengths at 30 min after mixing than polymethyl methacrylate-based resin. The strengths of all bis-acryl resins significantly increased after 24 hour water storage and remained at these high values after 7 day water storage with or without thermocycling. These results agreed with the studies by Balkenhol et al, [7-8] which found that all auto-polymerized bis-acryl resin materials have low flexural strength at 10 minutes after mixing and the strength increased after 1to 72 hours of 37°C water storage and also after thermocycling. These might be due to lack of cross-linking between oligomers at an early stage of setting. The crosslinkings are increased when the time progressed, resulting in higher fracture resistance of the materials. The other reason might be due to stress development inside the polymeric network during the polymerization, thus the materials are more susceptible to fracture at the early setting stage. The increased temperature from 37°C water storage and thermocycling may decrease stresses inside the material by relaxation processes resulting in increase of its strength. This study concluded that the differences in mechanical properties of provisional materials are related to the monomer system used [7] and the chemical nature of the materials. [8]

Unifast Trad showed high flexural strength at 30 min after mixing and no significant difference after 24 hour and 7 days water storage with or without thermocycling. This result is different from the study of Balkenhol et al, [8] which found that the monomethacrylate resin used in their study showed low flexural strength at 10 min after mixing and increased after 2, 16, 24 and 72 hour water storage, and also after 7 days with thermocycling. The monomethacrylate resin used in their study contained iso-butylmethacrylate (i-BMA), which presented a longer side chain compared to methyl

methacrylate monomer. The side chain in the structure of i-BMA increases the backbone separation of the polymer molecules and thus acts as an internal plasticizer for its copolymerization, resulting in strength reduction [21]. Consideration of the monomethacrylate resin used in this study, Unifast Trad contains methyl methacrylate and ethyl methacrylate copolymers, which form linear polymerization without cross-linking. These copolymers have short length of side chain and narrow distribution of the molecular weight of the polymer (MW=100 for methyl methacrylate and 114 for ethyl methacrylate), resulting in more stable copolymerization, chemically stable to heat and external plasticizers which was water for this study, and better physical and mechanical properties [21] compared to the monomethacrylate resin used in the study of Balkenhol et al [8].

The flexural strengths of all bis-acryl resin materials in this study were higher than those of Unifast Trad. All bis-acryl resin materials tested contain glass powder and silica, whereas Unifast Trad does not have such composition. It could be considered that filler contents might have influenced the strengths of materials. A study of Haselton et al [9]. compared the flexural strengths of methacrylate resins and bis-acryl resins used to fabricate provisional fixed restorations after immersion in 37°C artificial saliva for 10 days. They stated that two important factors contributing to the mechanical properties of those materials are the monomer chemistry and the use of fillers. Conventional methacrylate-based materials use monomers that are less rigid and contain little or no fillers, while the bis-acryl resins contain more rigid monomers, such as bis-GMA, and addition of inorganic fillers.

Internal color change of resin-based materials has been reported to be the result of water sorption and photo-oxidation [17, 22-24]. Water plays a role in oxidative and hydrolytic reactions which were the chemical degradation process, [25] resulting in changes of the optical properties of provisional materials [12]. This study tested the color change of provisional materials, resulting from water sorption at different time periods of temporization by immersion in 37°C distilled water with and without accelerated aging by thermocycling.

As previously mentioned, the accelerated aging method often used for color stability test of provisional resin materials is immersion of specimens in 60°C water for 1 month, which is suggested to be equivalent to nearly 1-year in 37°C water [17]. This method seems to be too long comparing to the duration of using provisional materials. The other aging method is subjecting the specimens to UV light for different durations and cycles [11-12]. Nevertheless, a concern of non-reliable test for the use of UV light in color stability test of resin composites was also mentioned [18, 22]. The accelerated aging using thermoycling was suggested to be more clinical relevant. The 5,000 cycles of thermocycling used for accelerated aging in this study is the same as the method used in a previous study for color stability test of direct and indirect resin composites [18]. A dwell time of 15 second, however, was used in this study to simulate more appropriate the clinical situation [26-27].

The results of this study exhibited that the use of 5,000 thermocycling after 7 d, 37°C water immersion slightly increased  $\Delta E$  value, but not significantly different compared to the same storage condition without thermocycling. This may imply that the intrinsic color change of these provisional materials was insignificant after several weeks in oral conditions with various temperatures of food and drink consumption. It might be also suggested that most of the intrinsic color changes of these provisional materials occurred in the first week. It is also interesting to note that, after thermocycling, the results of color changes of similar provisional materials used in this study compared to the previous studies using another aging method were in the same range of  $\Delta E$ 1.5-2.8 [9, 11-12].

In the CIE L\*a\*b\* color system, the color change between different conditions is given by  $\Delta E$ . Previous studies have reported different ranges of  $\Delta E$  value which were perceptible to the color changes.  $\Delta E$  value of one unit is a color difference value that is visually perceptible to 50% of observers under controlled conditions [24]. Other study demonstrated that values of  $\Delta E$ between 0 and 2 represented imperceptible to color change, whereas values of  $\Delta E$  in the range of 2 to 3 defined perceptible color differences [28].  $\Delta E$  values greater than or equal to 3.3 have been reported to be visually perceptible and clinically unacceptable to 50% of trained observers [29]. For this study, color difference values greater than 3.3 were regarded as clinically unacceptable.

All provisional materials showed lower L\* values after 24 hour and 7 day water storage with and without thermocycling compared to baseline, which means that the colors of all materials became darker after different aging times and conditions than their colors at baseline. With regard to Unifast Trad, this methacrylate resin showed the darkest change after 24 hour water storage and became brighter after 7 day water storage with and without thermocycling, but was still darker than the color at baseline. The largest change of a\*and b\* values also presented in Unifast Trad after 24 hour water storage compared to baseline values. This material showed higher in red and yellow chroma. Nevertheless, this change decreased after longer water storage time. From these findings, the color change,  $\Delta E$ , of Unifast Trad (Table 4) after 24 hour water storage was larger than 3.3 (5.4) which was clinically unacceptable [29]. Its color difference values reduced to be below 3.3 (2.35 and 2.43 after 7 day water storage with and without thermocycling respectively) which were clinically acceptable. However, the color change of this material can still be detected [28].

For bis-acryl resins, there were no significant differences of color change among these materials after all aging times and conditions. Also, there were no significant differences of color change among different aging times and conditions for each bis-acryl resin material. All bis-acryl resins showed significantly lower color change than Unifast Trad. Besides, all bis-acryl resins demonstrated small changes of  $\Delta L^*$ ,  $\Delta a^*$  and  $\Delta$ b\* as shown in Table 5, which resulted in small changes of color difference values. They demonstrated  $\Delta E$  values lower than 2, which were imperceptible to color change [28].

From the aforementioned findings, bis-acryl resin materials seem to be more color stable than methacrylate resin after 37°C water storage and thermocycling. The results of this study in term of internal color change cannot be directly compared with other studies because of different materials, aging times and testing time intervals. However, the results of color stability from this study are in agreement with previous studies, [12, 30-31] which found higher color stability of bis-acryl resins compared with polymethyl methacrylate resins. The high color change of methyl/ethyl methacrylatebased resins, which was clinically unacceptable, might be due to their higher water sorption compared to bis-acryl resins.

Unifast Trad showed larger color changes after immersion in water and thermocycling compared to other bis-acryl resins, especially after 24 hours of 37°C water storage. The large change of Unifast Trad might be due to the effect of amine activators contained in the liquid part of this material, N,N-dimethyl-p-toluidine, which is a chemical activator in the induction stage of an additional polymerization chain reaction process. Asmussen [17] found that the amount and type of amine affected the color change of resin materials. The color change depends on a ratio between amine and benzoyl peroxide (BPO) which is an initiator of the polymerization reaction. With regard to amine type, it was found that the resins cured with DEPT (N, N-diethanol-p-toluidine) were less color stable than resins cured with DEBA (N,N-diethanol-3,5-di-tert-butyl-alinine) which has a bulky structure with a steric hindrance for color forming oxidizing reaction on the benzene ring of the amine. Amount of BPO was not indicated in the Unifast Trad's MSDS, thus the amount of amine that might have affected color stability of Unifast Trad could not be discussed. Nevertheless, *N*,*N*-dimethyl-*p*-toluidine containing in Unifast Trad which has a smaller structure than DEBA, or even DEPT, might be the cause of color instability of Unifast Trad compared to other provisional resins containing larger structure of amine. The color change of Unifast Trad decreased after 7 day water storage and after thermocycling compared to after 24 hour storage time. This color change pattern of methacrylate resin could not be directly compared with other studies because there were no studies simulated the same aging conditions nor tested the color change in the same time intervals as those of the present study. A study of Bayindir et al $^{32}$  found that  $\Delta E$  values of PMMA resin decreased after 7 day storage in 37°C distilled water compared to 24 hour storage time; however, this study did not mentioned about the actual time for baseline color measurement in the study. Therefore, their result could not be directly compared to the result of this study.

Based on the results of this study, significant differences of the flexural strength and color changes were found among the provisional materials, and aging conditions. Therefore, the null hypothesis was rejected.

In conclusion, the methyl/ethyl methacrylatebased resin provided higher flexural strength than the bis-acryl resin at 30 min. The bis-acryl resins, however, exhibited higher flexural strength and less color changes than the methyl/ ethylmethacrylate-based resin at 7 days. Aging process using 5,000 cycles of thermocycling after 7 days did not change flexural strength and color of these materials significantly. Because the low flexural strengths of bis-acryl resins were exhibited at 30 minutes after mixing, therefore, clinical use of these provisional restorations at the early stage of delivering should be concerned. The results of color stability could be used to predict the long-term color changes of these provisional materials.

## References

- 1. Vahidi F. The provisional restorations. *Dent Clin North* Am 1987; 31: 363-381.
- 2. Perry RD, Magnuson B. Provisional materials: key components of interim fixed restorations. Compend Contin Educ Dent 2012; 33: 59-60, 62.
- 3. Michalakis K, Pissiotis A, Hirayama H, Kang K, Kafantaris N. Comparison of temperature increase in the pulp chamber during the polymerization of materials used for the direct fabrication of provisional restorations. J Prosthet Dent 2006; 96: 418-423.
- 4. Strassler HE. Fixed prosthodontics provisional materials: making the right selection. Compend Contin Educ Dent 2013; 34: 22-24, 26.
- 5. Young HM, Smith CT, Morton D. Comparative in vitro evaluation of two provisional restorative materials. J Prosthet Dent 2001; 85: 129-132.
- 6. Rosenstiel SF, Land MF, Fujimoto J. Contemporary Fixed Prosthodontics 4<sup>th</sup> ed. Philadelphia: Mosby; 2006: 466-504.
- 7. Balkenhol M, Ferger P, Mautner MC, Wostmann B. Provisional crown and fixed partial denture materials: mechanical properties and degree of conversion. Dent Mater 2007; 23: 1574-1583.
- 8. Balkenhol M, Mautner MC, Ferger P, Wostmann B. Mechanical properties of provisional crown and bridge materials: chemical-curing versus dual-curing systems. J Dent 2008; 36: 15-20.
- 9. Haselton DR, Diaz-Arnold AM, Vargas MA. Flexural strength of provisional crown and fixed partial denture resins. J Prosthet Dent 2002; 87: 225-228.
- 10. Kerby RE, Knobloch LA, Sharples S, Peregrina A. Mechanical properties of urethane and bis-acryl interim resin materials. J Prosthet Dent 2013; 110: 21-28.
- 11. Doray PG, Li D, Powers JM (2001) Color stability of provisional restorative materials after accelerated aging. J Prosthodont 2001; 10: 212-216.

- 12. Sham AS, Chu FC, Chai J, Chow TW. Color stability of provisional prosthodontic materials. J Prosthet Dent 2004: 91: 447-452.
- 13. Givens EJ Jr., Neiva G, Yaman P, Dennison JB. Marginal adaptation and color stability of four provisional materials. J Prosthodont 2008; 17: 97-101.
- 14. Guler AU, Yilmaz F, Kulunk T, Guler E, Kurt S. Effects of different drinks on stainability of resin composite provisional restorative materials. J Prosthet Dent 2005; 94: 118-124.
- 15. Haselton DR, Diaz-Arnold AM, Dawson DV. Color stability of provisional crown and fixed partial denture resins. J Prosthet Dent 2005; 93: 70-75.
- 16. Turgut S, Bagis B, Ayaz EA, Ulusoy KU, Altintas SH, Korkmaz FM, Bagis N. Discoloration of provisional restorations after oral rinses. Int J Med Sci 2013; 10: 1503-1509.
- 17. Asmussen E. An accelerated test for color stability of restorative resins. Acta Odontol Scand 1981; 39: 329-332.
- 18. Lee YK, Yu B, Lim HN, Lim JI. Difference in the color stability of direct and indirect resin composite. J Appl Oral Sci 2011; 19: 154-160.
- 19. International Organization for Standardization. Dentistry--Polymer-based filling, restorative and luting materials. ISO 4049:2000, Geneva.
- 20. Sakaguchi RL, Powers JM. Craig's Restorative Dental Materials. 13th ed. St. Louis: Mosby; 2011: 83-107.
- 21. Rawls HR. Dental polymers.In: Anusavice KJ, editor. Phillips' science of dental materials, 11th ed. St. Louis: Saunders Co; 2003: 143-169
- 22. Asmussen E. Factors affecting the color stability of restorative resins. Acta Odontol Scand 1983;
- 23. Ferracane JL, Moser JB, Greener EH. Ultraviolet light-induced yellowing of dental restorative resins. J Prosthet Dent 1985; 54: 483-487.
- 24. Seghi RR, Gritz MD, Kim J. Colorimetric changes in composites resulting from visible-light-initiated polymerization. Dent Mater 1990; 6: 133-137.
- 25. Ruyter IE. Physical and chemical aspects related to substances released from polymer materials in an aqueous environment. Adv Dent Res 1995; 9: 344-347.
- 26. Li H, Burrow MF, Tyas MJ. The effect of thermocycling regimens on the nanoleakage of dentin bonding systems. Dent Mater 2002; 18: 189-196.

- 27. Harper RH, Schnell RJ, Swartz ML, Phillips RW. In vivo measurements of thermal diffusion through restorations of various materials. J Prosthet Dent 1980; 43: 180-185.
- 28. Gross MD, Moser JB. A colorimetric study of coffee and tea staining of four composite resins. J Oral Rehabil 1977; 4: 311-322.
- 29. Ruyter IE, Nilner K, Moller B. Color stability of dental composite resin materials for crown and bridge veneers. Dent Mater 1987; 3: 246-251.
- 30. Wang RL, Moore BK, Goodacre CJ, Swartz ML, Andres CJ. A comparison of resins for fabricating provisional fixed restorations. Int J Prosthodont 1989; 2: 173-184.

- 31. Watanabe H, Kim E, Piskorski NL, Sarsland J, Covey DA, Johnson WW. Mechanical properties and color stability of provisional restoration resins. Am J Dent 2013; 26, 265-270.
- 32. Bayindir F, Kurklu D, Yanikoglu ND. The effect of staining solutions on the color stability of provisional prosthodontic materials. J Dent 2012; 40, Suppl 2, e41-46.