



Effects of Different Amine Activators on the Monomer Conversion, Biaxial Flexural Strength, and Color Stability of Experimental Provisional Dental Restorations

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Abstract

Objective The aim was to assess the effect of different amine activators including N, N-dimethyl-p-toluidine (DMPT) or Na-N-tolyglycine glycidyl methacrylate (NTGGMA) on chemical-activated monomer conversion, biaxial flexural strength (BFS), and color stability of composites for provisional dental restorations.

Materials and Methods Two formulations of composites containing either DMPT (D-temp) or NTGGMA (N-temp) were prepared. The degree of monomer conversion was assessed. The BFS of the materials was tested using the ball-on-ring testing jig. The color difference (ΔE_{00}) of the materials after immersion in water was also determined. The commercial comparisons were Unifast (UF), Protemp (PT), Luxacrown, and Luxatemp (LT).

Results The monomer conversion of D-temp ($57.4 \pm 1.3\%$) was comparable to that of N-temp ($59.0 \pm 1.3\%$). The conversion of both D-temp and N-temp were higher than that of PT ($48.1 \pm 3.4\%$) and LT ($48.0 \pm 1.6\%$). BFS of both D-temp (164.2 ± 18.1 MPa) and N-temp (168.6 ± 8.9 MPa) were comparable but higher than that of UF (119.8 ± 13.6 MPa). ΔE_{00} of D-temp (2.7 ± 0.7) and N-temp (2.5 ± 0.8) were comparable but higher than that of other commercial materials (0.6–1.2).

Conclusion The use of DMPT or NTGGMA showed negligible effect on monomer conversion, BFS, and color stability of the experimental provisional restorations. The conversion and BFS of the experimental materials were in the range of that obtained from commercial bis-acryl-based materials. However, the color stability of the experimental materials was lower than that of commercial materials.

Keywords

- ▶ composite resin
- ▶ temporary
- ▶ polymerization
- ▶ flexural strength
- ▶ color stability

Introduction

Provisional dental restorations are commonly used during the temporization period between preparing teeth and the placement of final restoration to help maintaining prepared margins, spaces, periodontal health, masticatory functions,

appearance, and vocal quality for the patient.^{1,2} The commonly used provisional materials include polymethyl methacrylate (PMMA) and bis-acryl resin composites which contain dimethacrylate monomers and fillers.^{3,4} These materials can be polymerized chemically which could facilitate the direct

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fabrication technique.³ The technique involves the injection of material into the silicone index placed over the prepared tooth intraorally. The clinicians then allow the material to set either by light or chemical activations. The use of light activation significantly enhances physical properties of the material.⁵ However, the light curing option may not be possible if the transparent silicone is not available. Thus, the ability to cure chemically is still vitally important for the materials.

Tertiary amine enables the dissociation of chemical initiator, such as benzoyl peroxide (BP), to initiate polymerization of the materials. N, N-dimethyl-p-toluidine (DMPT; molecular weight of 135 g/mol) is the commonly used tertiary amine to enable polymerization of chemical-activated resin composites or cements.^{6,7} The major concern of DMPT is its toxicity. Various studies have reported that DMPT can be absorbed and distributed to various sites inducing toxic and carcinogenic effects.⁸⁻¹⁰ Hence, an alternative activator is needed.

Na-N-tolylglycine glycidyl methacrylate (NTGGMA) consists of methacrylate and amine groups in the structure (►Fig. 1).¹¹ Hence, the monomer can be polymerized within the polymer network, thus reducing the risk of leaching of monomers. The monomer also contains carboxylic group which could potentially help to increase hydrophilic properties and promote the flow of material on the hydrophilic tooth surface. However, the hydrophilic group of NTGGMA may increase water sorption to the material that may subsequently reduce the strength and esthetic properties of the material.¹² The materials are required to exhibit sufficient mechanical strength and color stability to ensure patient satisfaction and function during temporization process.

The aim of this study was therefore to assess the effect of using different amine activator (NTGGMA vs. DMPT) on the degree of monomer conversion (MC), biaxial flexural strength (BFS), and color stability of the experimental provisional dental restorations. Commercial materials were used for comparison. The null hypothesis was that the use of different amine activators should not detrimentally affect the degree of MC, BFS, and color stability of the materials.

Materials and Methods

Material Preparation

The experimental composites for provisional restorations were prepared using the power-to-liquid mass ratio of 2.3:1. Powder phase of the experimental materials contained silanated borosilicate glass (Esstech Inc.; Essington, Pennsylvania,

United States). The liquid phase contained 70 wt% urethane dimethacrylate (UDMA; lot no. MKCG8230, Sigma-Aldrich, St. Louis, Missouri, United States), 24 wt% triethyleneglycol dimethacrylate (lot no. STBF9549V, Sigma-Aldrich), and 5 wt% 2-hydroxyethyl methacrylate (HEMA; lot no. STBG6525, Sigma-Aldrich). For imitator liquid, 1 wt% BP (lot no. MKCF7091, Sigma-Aldrich) was added. For activator liquid, 1 wt% of DMPT (lot no. MKBX9809V, Sigma-Aldrich) or 2 wt% of NTGGMA (lot no. X8630050, Esstech Inc) was added. The experimental composites containing DMPT and NTGGMA were referred to as D-temp and N-temp, respectively.

The liquid phase was mixed using a magnetic stirrer for 1 hour. The mixed liquid was left for 24 hours before mixing with powder phase. The powder and liquid phases were weighed using a four-figure balance. The powder and liquid were hand-mixed using a plastic spatula (~20 seconds until the paste was mixed homogeneously). The mixed initiator and activator pastes were then loaded into the double-barrel syringe. The syringe was left in the upright position at room temperature for 24 hours to allow the release of air bubbles incorporated in the paste.

The experimental materials were mixed and injected using mixing tip with a dispenser Sulzer Mixpac AG, Switzerland.¹³ Commercially available provisional dental restorations including PMMA and bis-acryl-based composites were used as comparisons (►Table 1). The commercial materials were prepared according to the manufacturer's instruction.

Monomer Conversion

A Fourier-transform infrared spectroscopy (FTIR; Nicolet iS5, Thermo Fisher Scientific, Massachusetts, United States) equipped with an attenuated total reflection (ATR; ID7, Thermo Fisher Scientific) was used to determine the MC of the materials ($n = 5$). The mixed materials were placed in the metal circlip (1 mm in thickness and 10 mm in diameter) on the ATR diamond. The specimens were then covered with an acetate sheet. The FTIR spectra were recorded initially and after 10 minutes. The spectra at the region of 700 to 4,000 cm^{-1} with the resolution of 8 cm^{-1} were obtained.¹⁴ The test was performed at $25 \pm 1^\circ\text{C}$. The degree of MC was calculated using the following equation:

$$MC(\%) = \frac{100(B_0 - B_t)}{B_0} \quad (1)$$

where B_0 and B_t are the absorbance of the C-O peak (1,320 cm^{-1}) above background level at 1,335 cm^{-1} initially and after time t .¹⁵

Biaxial Flexural Strength and Biaxial Flexural Modulus

The materials were loaded into the metal circlip (1 mm in thickness and 10 mm in diameter) ($n = 8$). The specimens were covered with an acetate sheet and left at room temperature for 24 hours to allow the completion of polymerization. The disc specimens were then removed, trimmed excess, and placed in a tube containing 10 mL of deionized water. They were incubated at 37°C for 24 hours and 4 weeks. Then, the discs were

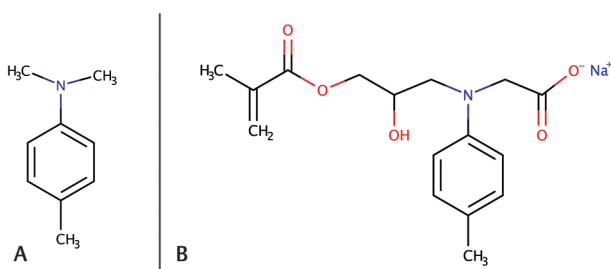


Fig. 1 Chemical structure of (A) DMPT (N, N-dimethyl-p-toluidine) and (B) NTGGMA (Na-N-tolylglycine glycidyl methacrylate).

Table 1 The composition of commercial materials

Materials	Composition	Lot no.	Suppliers
Unifast Trad (UF)	Powder: ethyl-methyl methacrylate monomer, polymethyl methacrylate, barbituric acid derivative, organic copper compound, pigments	1808271	GC Corporation; Tokyo, Japan
	Liquid: methyl methacrylate, N,N-dimethyl-p-toluidine trimethylolpropane, ethylene glycol dimethacrylate		
Luxacrown (LC)	Inorganic and organic filler, matrix of multifunctional acrylates	770658	DMG; Hamburg, Germany
Luxatemp (LT)	Polymethyl methacrylate, SiO ₂ , bisphenol A-glycidyl methacrylate, urethane dimethacrylate, other dimethacrylates	802411	DMG; Hamburg, Germany
Protemp 4 (PT)	Base: Ethoxylate bisphenol-A dimethacrylate, silane-treated amorphous silica, reaction product of 1,6-diisocyanatohexane with 2-[(2-methacryloyl) ethyl]6-hydroxyhexonate and 2-hydroxyethyl methacrylate	4249947	3M ESPE; St. Paul, MN, USA
	Catalyst: ethanol, 2,2'-[(1-methylethylidene)bis(4,1-phenyleneoxy)]bis-diace-tate, benzyl-phenyl-barbituric acid, silane-treated silica		

Note: The materials are either polymethyl methacrylate-based material (Unifast Trad) or bis-acryl-based materials (Luxacrown, Luxatemp, and Protemp 4). The ingredients of Luxacrown and Luxatemp in each catalyst or base pastes were not supplied by suppliers.

removed, blotted dry, and mounted in the ball-on-ring testing jig. BFS test was performed under the mechanical testing frame (AGSX, Shimadzu, Kyoto, Japan) using 500 N load cell with the crosshead speed of 1 mm/min. The BFS (Pa) of the specimen was obtained using the following equation¹⁶:

$$BFS = \frac{F}{d^2} \left\{ (1 + \hat{i}) \left[0.485 \ln \left(\frac{r}{d} \right) + 0.52 \right] + 0.48 \right\} \quad (2)$$

where F is the load at failure (N), d is the specimen's thickness (m), r is the radius of circular support (m), and \hat{i} is Poisson's ratio (0.3). Additionally, biaxial flexural modulus (BFM) was calculated using the following equation¹⁷:

$$BFM = \left(\frac{\Delta H}{\Delta W_c} \right) \times \left(\frac{\hat{a}_c d^2}{q^3} \right) \quad (3)$$

where $\Delta H/\Delta W_c$ is the rate of change of load with regards to central deflection or gradient of force versus displacement curve (N/m), \hat{a}_c is the center deflection junction (0.5024),¹⁸ and q is the ratio of support radius to the radius of disc. Additionally, the fracture surface of tested specimens at 4 weeks was investigated using a scanning electron microscope (SEM; JSM 7800F, JEOL Ltd., Tokyo, Japan).

Color Stability

The disc specimens ($n = 3$) were prepared and immersed in 10 mL of deionized water. The specimens were incubated at 37°C for 3 weeks. The CIELab coordinates of all specimens before and after immersion were measured using a dental spectrophotometer (Easysshade V; VITA Zahnfabrik, Baden-Württemberg, Germany). The spectrophotometer was calibrated according to the manufacturer's instructions prior to the measurement. The specimens were placed over the opaque white background. The illumination of the room was 850 lux which was measured by a light meter (LX1330B Light Meter; Dr. Meter Digital Illuminance, StellarNet Inc.; Florida, United States).¹⁹ The spectrophotometer probe tip was positioned perpendicular to the center of the specimens until the completion of measurement. The color coordinates

(CIE L^* , a^* , b^* , C^* , and h°) were then recorded. The L^* , a^* , and b^* parameters refer to value axis, red-green axis, and yellow-blue axis, respectively. In addition, C^* and h° were chroma and hue angle.

The measurement for each specimen was performed in triplicate. Color differences or color changes of the composites after immersion in deionized water was calculated using the CIEDE2000 (E_{00}) formula²⁰:

$$E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L} \right)^2 + \left(\frac{\Delta C'}{K_C S_C} \right)^2 + \left(\frac{\Delta H'}{K_H S_H} \right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C} \right) \left(\frac{\Delta H'}{K_H S_H} \right) \right]^{1/2} \quad (4)$$

where $\Delta L'$, $\Delta C'$, and $\Delta H'$ represent the changes in lightness, chroma, and hue, respectively. Furthermore, R_T is a rotation function related to the interaction between chroma and hue differences in the blue region. Additionally, S_L , S_C , and S_H are weighting functions and K_L , K_C , and K_H are correction terms for experimental conditions.

Statistical Analysis

Values reported in the current study are mean \pm standard deviation. The data were analyzed using Prism 9 (GraphPad Software; San Diego, California, United States). Normality of the data was tested using the Shapiro–Wilk test. For normally distributed data (MC, BFS, color stability), one-way analysis of variance followed by post hoc Tukey multiple comparison was employed. For non-normally distributed data (BFM), Kruskal–Wallis test followed by Dunn's multiple comparisons was used. Significance level was set at $p = 0.05$. Additionally, power analysis was performed using G*Power version 3.1.9.6 (University in Düsseldorf, Germany) which indicated that the sample used in each test gave power > 0.95 at $\alpha = 0.05$.

Results

Degree of Monomer Conversion

The highest MC was obtained from Unifast (UF) ($75.6 \pm 1.5\%$) (\blacktriangleright Fig. 2). D-temp ($57.4 \pm 1.3\%$) exhibited comparable MC to

N-temp ($59.0 \pm 1.3\%$) ($p = 0.8346$). The conversion of both D-temp and N-temp were similar to that of Luxacrown (LC) ($60.1 \pm 2.9\%$) ($p = 0.3676, 0.9626$). D-temp and N-temp however showed significantly higher MC than Luxatemp (LT) ($48.0 \pm 1.6\%$) ($p < 0.01$) and Prottemp (PT) ($48.1 \pm 3.4\%$) ($p < 0.01$).

Biaxial Flexural Strength and Biaxial Flexural Modulus

At 24 hours, the highest and lowest BFS were obtained from LT (214.1 ± 29.7 MPa) and UF (119.8 ± 13.6 MPa), respectively (►Fig. 3A). The BFS of D-temp (164.2 ± 18.1 MPa) was comparable to that of N-temp (168.6 ± 8.9 MPa) ($p = 4332$). The BFS of both D-temp and N-temp were significantly higher than that of UF ($p = 0.008, 0.002$) but comparable to that of PT (185.6 ± 19.0 MPa) ($p = 0.2783, 0.5315$) and LC (193.5 ± 17.2 MPa) ($p = 0.0540, 0.1452$). The BFS of D-temp and N-temp was reduced to 121.1 ± 31.2 and 143.2 ± 12.8 MPa after immersion in simulated body fluid for 4 weeks. The BFS of D-temp and N-temp at 4 weeks was significantly lower than that of PT (187.5 ± 26.5 MPa), LC ($173.6.5 \pm 23.8$ MPa), and LT (184.7 ± 24.0 MPa) ($p < 0.05$).

The highest and lowest BFM at 24 hours were obtained from N-temp (4.0 ± 0.2 GPa) and UF (1.3 ± 0.2 GPa), respectively (►Fig. 3B). BFM of N-temp was comparable to that

of D-temp (3.0 ± 0.4 GPa) ($p = 0.6267$) but was significantly higher than that of UF ($p < 0.01$), PT (2.0 ± 0.2 GPa) ($p = 0.0001$), and LC (2.6 ± 0.4 GPa) ($p = 0.0382$). After 4 weeks, the values of D-temp and N-temp were reduced to 2.5 ± 0.7 and 3.5 ± 0.4 GPa, respectively. The highest and lowest observed mean values were obtained from N-temp (3.5 ± 0.4 GPa) and UF (1.5 ± 0.5 GPa), respectively.

Fracture surfaces of the tested specimens at 4 weeks were examined under SEM (►Fig. 4). PT, LC, and LT showed smooth fractured surfaces. However, multiple voids with diameter of 20 to 50 μm in the bulk of materials were detected on the fracture surface of D-temp and N-temp.

Color Stability

The highest and lowest observed color difference (ΔE_{00}) were obtained from D-temp (2.69 ± 0.66) and UF (0.55 ± 0.17), respectively (►Fig. 5). ΔE_{00} of D-temp and N-temp (2.46 ± 0.78) were comparable ($p = 0.997$) but were significantly higher than that of UF ($p = 0.0124, 0.0266$). The color difference of UF was similar to that of PT (0.91 ± 0.25) ($p = 0.9800$), LC (1.16 ± 0.84) ($p = 0.8399$), and LT (0.98 ± 0.72) ($p = 0.9544$). Additionally, PT exhibited significantly lower color difference than D-temp ($p = 0.0396$).

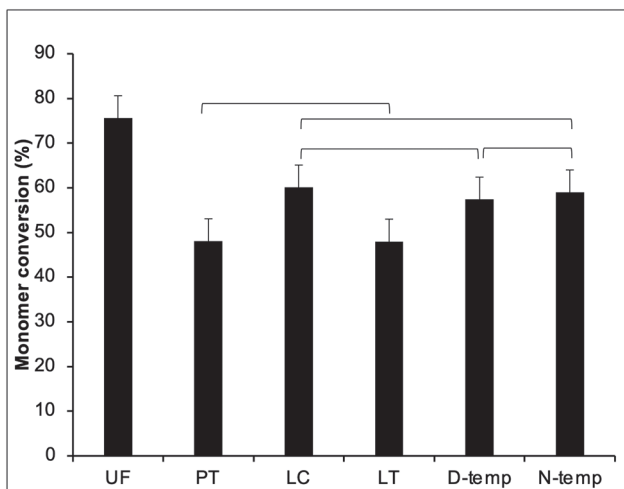


Fig. 2 Monomer conversion of Unifast (UF), Prottemp (PT), Luxacrown (LC), Luxatemp (LT), and experimental composites (D-temp and N-temp). Error bars are standard deviation (SD) ($n = 5$). Lines represent $p > 0.05$.

Discussion

Currently, one of the main chemical activators used in resin-based materials for provisional dental restorations is DMPT. However, the major concern of this monomer is its toxic effects. The aim of this preliminary study was therefore to investigate the effect of using different amine activators (DMPT or NTGGMA) on degree of MC, BFS, and color stability of the materials. The hypothesis was accepted as the use of DMPT or NTGGMA showed no detrimental effect to MC, BFS, and color stability of the materials. It should be noted that the current study is an *in vitro* study. Hence, the clinically relevant aspects should be carefully interpreted.

Degree of Monomer Conversion

High degree of MC of provisional restorations may help to ensure adequate physical and mechanical properties for the restorations.²¹ Additionally, it was expected that the high conversion could also reduce the risk of unreacted monomer

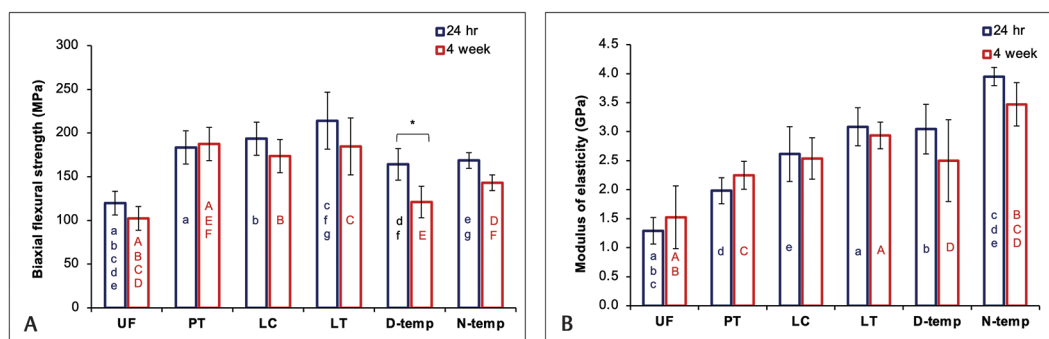


Fig. 3 (A) Biaxial flexural strength (BFS) and (B) biaxial flexural modulus (BFM) of Unifast after immersion in water for 24 hours and 4 weeks. Error bars are standard deviation (SD) ($n = 8$). Same lower-case and upper-case letters denoted $p < 0.05$ for the strength at 24 hours and 4 weeks, respectively. Stars (*) represent $p < 0.05$ for the strength of the same material.

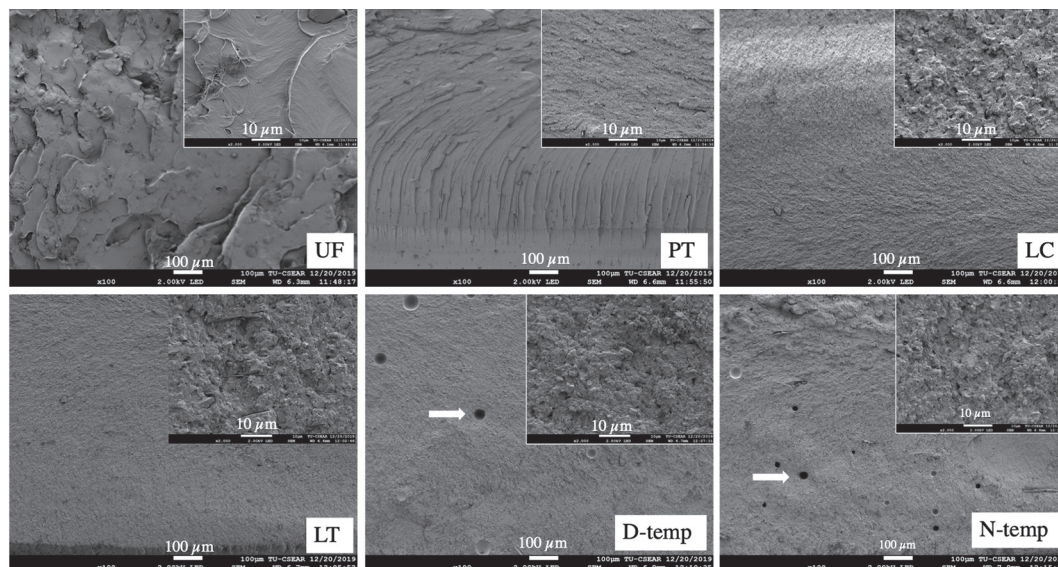


Fig. 4 Scanning electron microscope (SEM) images of fracture surface from Unifast (UF), Protemp (PT), Luxacrown (LC), Luxatemp (LT), and experimental composites (D-temp and N-temp). The scale bars represent 100 and 10 μm in length. Voids in the core of materials were observed with D-temp and N-temp (arrows).

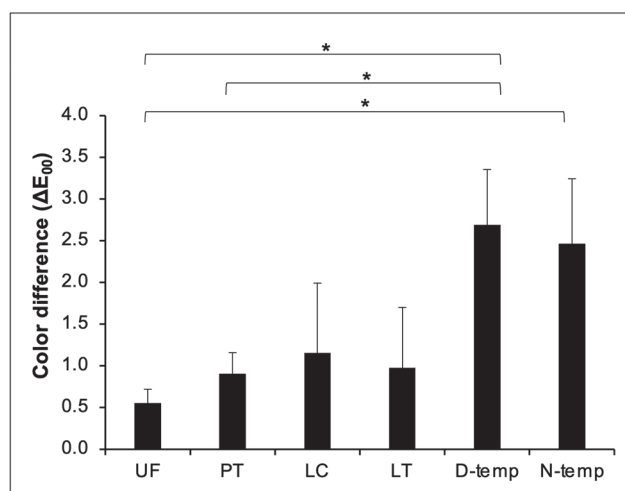


Fig. 5 Color difference (E_{00}) of Unifast (UF), Protemp (PT), Luxacrown (LC), Luxatemp (LT), and experimental composites (D-temp and N-temp) after immersion in deionized water for 3 weeks. Error bars are standard deviation (SD) ($n = 3$). Stars (*) represent $p < 0.05$.

release that may cause cytotoxic effects.²² It is known that monomer with low glass transition temperature (T_g) and high flexibility could contribute to high degree of MC of the polymer.^{17,23} The highest MC was detected with PMMA-based material (UF) which could be due to the use of low molecular weight methyl methacrylate monomer (molecular weight = 101 g/mol). However, high MC usually associates with high exothermic reaction which may affect dentin-pulp complex.²⁴ A study demonstrated that PMMA exhibited increase in temperature during setting by 4.2 to 11.6°C which was higher than that of bis-acryl composite (2.0–6.6°C).⁴

It should be mentioned that the concentration of DMPT (1 wt%) was lower than NTGGMA (2 wt%). The pilot study showed that using 2 wt% DMPT and 1 wt% NTGGMA enabled suitable handling characteristics. The MC of D-temp

and N-temp were higher than that of two commercial bis-acryl-based materials (PT and LT). The primary base monomer of D-temp and N-temp was UDMA. The T_g of UDMA (-38°C)²⁵ was lower than bis-GMA ($T_g = -10^\circ\text{C}$) which was the primary base monomer of bis-acryl-based materials (PT, LT). However, the actual composition of the monomers of commercial materials was not supplied from the manufacturers.

Biaxial Flexural Strength and Biaxial Flexural Modulus

The provisional restorations required adequate strength to ensure the survival upon the repeated chewing forces before the placement of definitive restoration.³ The required flexural strength from 3-point bending test for polymer-based crown material according to the BS ISO 10477–2018 was 50 MPa.²⁶ The current study employed BFS instead of 3-point bending test as stated in the standard. It is suggested that BFS test could give similar results to 3-point bending test but with more reproducibility.²⁷ The results from the current study suggested that flexural strength of the experimental composites should pass the standard even after aging in water for 4 weeks.

The strength of dimethacrylate-based composites (PT, LT, LC, D-temp, and N-temp) was higher than of the strength obtained from monomethacrylate-based materials (UF). This was in accordance with the previous studies.^{2,28} The lowest BFS and modulus was obtained from UF which could be due to the lack of reinforcing fillers in the material. Additionally, the linear structure of monomethacrylate polymer and the lack of cross-links between polymer chains of UF may result in the low rigidity and strength.² The 24-hour flexural strength of commercial materials in the current study was higher than that reported in the published studies (UF ~64–111 MPa,^{28–30} PT ~85–113 MPa,^{29,31} LT ~81.7 MPa⁵). The possible explanation could be due to the use of different protocol for specimen preparation. In the current study,

the specimens were left undisturbed in the metal circlip at room temperature for 24 hours prior to immersion. The delay of specimen removal may therefore allow polymerization reaction to continue, thus increasing the cross-links polymer network and strength of the materials.³² The highest modulus of elasticity was obtained from N-temp. This may be due to the increase in filler load of the experimental materials (69.7 wt%) which was higher than that of commercial materials (30.8–39.3 wt%).⁵ The high level of fillers may then increase the stiffness of the materials.³³ The high stiffness and rigidity of the experimental materials may be considered suitable for temporizing the long-span fixed dental prostheses.

The fracture surface of PT demonstrated smoother and more homogenous surface compared with other bis-acryl-based materials.³⁴ This may be due to the lower filler load (~30.8 wt%)⁵ or smaller filler diameter of PT compared with other materials. The bis-acryl-based materials and the experimental materials were mixed using the mixing tips which may decrease the risk of air bubbles incorporation.³⁵ Multiple voids were detected in the fracture surfaces of D-temp and N-temp (► Fig. 4). This could be due to the incorporation of air bubbles during the hand mixing of powder and liquid to produce initiator/activator pastes. In the future work, the mixed paste should be stored in a vacuum to help release air bubbles in the materials.

The increase in immersion time enabled materials to absorb water which led to polymer plasticization, which could reduce the physical/mechanical properties of the materials.³⁶ The use of NTGGMA which contained hydrophilic group (carboxyl group) showed no significance on the strength. It was expected that the high MC and the incorporation of salinized glass fillers may help maintain mechanical strength.² The experimental provisional materials however showed large decrease in strength after immersion for 4 weeks compared with commercial materials. The possible explanation could be that the experimental materials contained HEMA. The addition of HEMA was expected to promote wetting of the materials on the hydrophilic tooth surface. The hydrophilicity of HEMA may encourage water sorption and reduce strength of the materials.³⁷ However, the strength of the experimental provisional materials after 4-week immersion were still higher than that required by the ISO standard.

Color Stability

The degree of color changes of provisional materials was associated with various factors such as chemical properties of the materials, filler size, water sorption, the incorporation of air bubbles, and degree of cross-linking molecules.³⁸ It was proposed that perceptibility threshold which represent minimum color difference identifiable by viewer was when $\Delta E_{00} = 0.8$.³⁹ Additionally, the acceptability threshold (AT) which indicate the level of color difference that was acceptable by viewer is when $\Delta E_{00} = 1.8$. Hence, it is expected that the materials should exhibited color difference within those range. The ΔE_{00} of all experimental materials (0.6–1.2) were within the range of both perceptibility threshold and AT. The

high MC of experimental composites was expected to reduce color change of the materials. However, the color differences of both D-temp (2.7) and N-temp (2.5) were higher than AT level. This could be due to the use of hydrophilic HEMA polymer which may promote water sorption and affect color stability of the material.^{38,40}

Conclusion

Within the limitation of this study, the following conclusions can be drawn:

- The use of NTGGMA or DMPT in the current study showed no detrimental effect on MC, BFS, and color stability of the experimental provisional dental restorations.
- The MC and BFS of experimental materials were in the range of those observed with the commercial composite for provisional restorations. The strength was also higher than that required by the ISO standard.
- The color stability of experimental materials was lower than that of the commercial provisional materials.

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Conflict of Interest

None declared.

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