Influence of different curing modes on flexural properties, fracture toughness, and wear behavior of dual-cure provisional resin-based composites

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We investigated the influence of different curing modes on the mechanical properties and wear behavior of dual-cure provisional resin-based composites (DCPRs). Three DCPRs and a self-curing bis-acryl provisional resin-based composite were used. Flexural strength (σf), elastic modulus (E), resilience (R), and fracture toughness (KIC) were measured. The specimens were fabricated with and without light irradiation, stored in distilled water at 37°C for 24 h, and subjected to 5,000 or 10,000 thermal cycles. For sliding impact wear testing, 12 specimens were prepared with and without light irradiation. The maximum facet depth and volume loss were determined using a noncontact profilometer. Some of the mechanical properties and wear behavior of DCPRs are affected by light irradiation. This study indicated that proper light irradiation is important in polymerization process of the DCPRs to enhance the wear resistance and some mechanical properties.

Keywords: Dual-cure provisional resin-based composites, Flexural properties, Fracture toughness, Wear behavior

INTRODUCTION

Provisional restorations play an important role in indirect restorative procedures such as maintaining masticatory function, esthetics, and vocal quality in the period between preparing teeth and cementing final restorations8-10. In addition, provisional restorations maintain the spacing of teeth, seal prepared teeth, and protect the margins of prepared teeth. Provisional restorations can also be used to identify final restorations that meet patients’ esthetic and functional requirements8-10. Therefore, despite advances in digital dentistry, provisional restorations remain useful for designing final restorations.

In some patients, provisional restorations are expected to function for an extended period until treatment is completed. When treating parafunctional disorders, severe periodontal disease including with dental implant therapy, or advanced tooth wear requiring reconstruction of large areas of the dental arches, long-term stability of provisional restorations under intraoral conditions is essential11-13. However, after long service under intraoral conditions, restorations often degrade, necessitating replacement or repair. A primary cause of damage requiring replacement or repair of a provisional restoration is fracturing in the body and margin, the treatment of which is associated with additional costs and time11-13.

Although clinical trials with long observation periods measure the real-world durability of restorative materials, these studies are time-consuming, expensive, and subject to significant biases. Conversely, understanding the characteristics of materials through standardized laboratory investigations facilitates the selection of optimal materials for clinical use. A systematic review of clinical trials on the mechanical parameters of resin composites and their relationships with fracturing and wear identified a weak negative correlation between fracture toughness (KIC) and clinical fractures, and a statistically significant correlation between flexural strength (σf) and clinical wear behavior14-16. Therefore, laboratory investigations of the mechanical properties of resin-based materials can yield useful information on their degradation in vivo.

Recently, bis-acryl-based provisional resin-based composites containing inorganic fillers for the fabrication of provisional restorations have been introduced that exhibit superior mechanical properties and easy handling characteristics8-10. Several of these new products offer dual-curing modes, enabling the on-demand polymerization of restorations17. However, some reports have claimed that polymerization initiated by self-curing alone results in poorer mechanical properties than polymerization with light irradiation18-20. Our previous report on dual-cure provisional resin-based composites (DCPRs) indicated that lower σf values were obtained without light irradiation than with light irradiation21. When fabricating provisional restorations with DCPRs either at the chair side or in a dental laboratory, impression materials are often used as an index. In this technique, the provisional resin paste is mixed, and placed in the tissue surface of the index, and reseated on the prepared diagnostic casts or prepared teeth in the mouth. In some cases, light irradiation cannot be performed on the provisional resin paste immediately, resulting in delayed polymerization. In the worst case scenario, light irradiation is not performed and clinicians rely on self-cure mode. Therefore, light irradiation may be a crucial factor affecting the long-term durability of DCPRs. However, little is known about
the differences in the mechanical and wear properties of DCPRs polymerized using different curing modes with and without light irradiation.

In this study, we examined the influence of curing mode under artificial aging on flexural properties and $K_{IC}$ of DCPRs. We also evaluated wear behavior under different curing modes. The null hypotheses to be tested were that curing mode would have no effect under artificial aging in the mechanical properties or wear behavior of DCPRs.

MATERIALS AND METHODS

Study materials

The DCPRs used in this study were: Tempsmart (TS; GC, Tokyo, Japan); Luxatemp Automix Solar (LX; DMG Chemisch-Pharmazeutische Fabrik, Hamburg, Germany); and Integrity Multi•Cure (IG; DENTSPLY Sirona, Milford, DE, USA). These materials cover much of the range of different types of resin monomer, different types of filler distribution, filler size, and shape in commercially available DCPRs. A self-curing bis-acryl provisional resin, Protemp Plus (PP; 3M Oral Care, St. Paul, MN, USA), was employed as a comparison material (Table 1). To avoid any influence of the reported non-uniformity of light-emitting diode curing units 17,18), a halogen-quartz-tungsten curing unit (Optilux 501, SDS Kerr, Danbury, CT, USA) was used, and the light irradiance (average 600 mW/cm²) of the curing unit was checked using a dental radiometer (model 100, SDS Kerr).

Flexural strength test

Following the International Organization for Standardization 4049 specifications 19), the flexural properties of the provisional resin-based composites were tested. According to the manufacturers’ instructions, each provisional resin paste was mixed and placed into a stainless-steel split mold with the dimensions 25×2×2 mm. The mold was then positioned on a glass slide. DCPRs were evaluated with (DC: dual cure mode) or without (SC: self cure mode) light irradiation. To examine the dual-cure mode, resin paste was placed into the mold, the middle third of the specimen was irradiated for 30 s, and the remaining thirds were irradiated for 30 s each. After the hardened specimen was removed from the mold, all six sides were wet polished with 1200-grit silicon carbide paper (Fuji Star type DDC, Sankyo Rikagaku, Saitama, Japan). The prepared specimens were stored in distilled water at 37°C for 24 h under dark conditions, and then subjected to 5,000, or 10,000 thermal cycles (TCs) between 5 and 60°C with a dwell time of 30 s. Baseline specimens were stored in distilled water at 37°C under dark conditions for 24 h before conducting the $\sigma_F$ test.

After the storage period, 12 specimens per test group were subjected to the three-point bending $\sigma_F$ test (span length=20 mm) using a universal testing machine (model 5500R, Instron, Canton, MA, USA) at a cross-head speed of 1 mm/min until the breaking of the specimen. The $\sigma_F$ and elastic modulus ($E$) were determined from the stress–strain curve using computer software (Bluehill version 2.5, Instron) linked to the testing instrument. In addition, the modulus of resilience ($R$) was calculated using the equation: $R=\sigma_F^2/2E$.

Fracture toughness measurements

To obtain the $K_{IC}$ values (MPa•m$^{1/2}$) of four provisional resin-based composites, the single-edge notched-beam

| Code | Provisional resins (Shade: Lot No.) | Main components | Inorganic filler content (wt%) | Manufacturer |
|------|------------------------------------|----------------|-------------------------------|--------------|
| TS   | Tempsmart (A2: 1505171)            | Bis-GMA, UDMA, dimethacrylate, silane treated amorphous silica, photo initiator, pigment | (24.4%)* | GC (Tokyo, Japan) |
| LX   | Luxatemp Automix Solar (A2: 715884) | Multifunctional methacrylate, glass filler, catalyst, stabilizer, additives | (39.3%)* | DMG (Hamburg, Germany) |
| IG   | Integrity Multi Cure (A2: 140808)   | Bis-acrylate, multifunctional methacrylate, barium boroaluminosilicate glass filler, catalyst, photoinitiator, stabilizer | (37.5%)* | Dentsply Sirona (Milford, DE, USA) |
| PP   | Protemp Plus (A2: Base 498865), (A2: Catalyst 495535) | Dimethacrylate (BISEMA 6), silane treated silica, ethanolate, 2,2'-[1-methylethylidene)-bis(4,1-phenylenoxy)] bisethyl diacetate, benzyl-phenyl-barbituric acid | (30.8%)* | 3M Oral Care (St. Paul, MN, USA) |

Bis-GMA: bisphenol-A-glycidyl methacrylate; UDMA: urethane dimethacrylate

*Inorganic filler contents from previous report 12
(SEN) - type three-point bending test was used. The procedure for making specimens for the \( K_{IC} \) test was similar to that used for the \( \sigma_f \) test except in terms of specimen configuration. The mixed resin paste was compacted into a stainless-steel split mold with the dimensions 25 x 5 x 2.5 mm, and a knife edge was used to make a notch at the center of one long edge of the specimen (0.5 mm wide and 1.5 mm deep). The mold was then positioned on a glass slide. Light irradiation and polishing of the surfaces were conducted in the same manner as for the \( \sigma_f \) test.

The prepared specimens were stored in distilled water at 37°C for 24 h under dark conditions, and then subjected to 5,000 or 10,000 TCs as for the \( \sigma_f \) test. Baseline specimens were stored in distilled water at 37°C under dark conditions for 24 h before the \( K_{IC} \) test. After the storage time, 12 specimens per test group were subjected to the three-point bending test (span length=20 mm) using a universal testing machine (model 5500R, Instron) at a cross-head speed of 1 mm/min until the breaking of the specimen. Before measurements, a new razor blade was used under hand pressure to create a sharp crack approximately 0.5 mm in the notch.

\[ K_{IC} = \frac{F}{B} \cdot \frac{S}{W} \cdot f(c/W) \]

\[ f(c/W) = 2.9(c/W)^{1/2} - 4.6(c/W)^{3/2} + 21.8(c/W)^{5/2} - 37.6(c/W)^{7/2} + 4.8(c/W)^{9/2}, \]

where \( F \) is peak load, \( S \) is span length (the distance between the supports; 20 mm), \( B \) is specimen thickness (2.5 mm), \( W \) is specimen height (5 mm), and \( c \) is crack length. Crack length was measured and recorded for each specimen using a confocal laser scanning microscope (VK-9710, Keyence, Osaka, Japan) and built-in software (VK-Analyzer; Keyence) at 40× magnification.

### Sliding impact simulated wear test

Twelve specimens of each provisional resin were used to determine wear behavior using a sliding impact wear testing machine (K655-06, Tokyo Giken, Tokyo, Japan). The provisional resin-based composites were mixed and placed into a cylindrical mold (6 mm in diameter, 2 mm high) made of polytetrafluoroethylene. The specimens were prepared with or without light irradiation. To examine the dual-cure mode, resin paste was compacted into the mold and light-irradiated for 30 s. One flat surface of each specimen was polished using a sequence of silicon carbide papers of up to 2,000 grit (Fuji Star Type DDC), and then the specimens were stored under dark conditions for 24 h in distilled water at 37°C.

Before wear testing, the specimens were attached to the center of a custom fixture fabricated from cold-cure acrylic resin (Tray Resin II, Shofu, Kyoto, Japan) with a small amount of model-repair glue (Zapit, Dental Ventures of America, Corona, CA, USA). The antagonist for sliding impact wear-simulation was a stainless-steel ball bearing (radius=2.4 mm) mounted inside a collet assembly. The simulator had a plastic water bath that constantly provided 37°C distilled water, and four custom fixtures were mounted inside the bath. During the wear-simulation test, the antagonists directly impacted the specimens from above with a maximum force of 50 N at a rate of 0.5 Hz and then slid horizontally for a distance of 2 mm. Each specimen was subjected to 10,000 cycles of sliding impact motion.

### Wear measurements

After the wear-simulation tests, the specimens were ultrasonically cleaned in distilled water for 3 min and then profiled using the confocal laser scanning microscope (VK-9710) and built-in software (VK-Analyzer). The maximum depth (MD: µm) and volume loss (VL: mm³) of the wear facets were determined for the three DCPRs in both the dual- and self-cure modes and for the self-curing provisional resin PP.

### Scanning electron microscopy observations

Cured provisional resin specimens were polished to a high gloss with abrasive discs (Fuji Star Type DDC) followed by a series of diamond pastes down to a particle size of 0.25 µm (DP-Paste, Struers, Ballerup, Denmark). The polished surfaces were then subjected to argon ion beam etching (IIS-200ER, Elionix, Tokyo, Japan) for 40 s, with the ion beam directed perpendicular to the polished surface (accelerating voltage=1 kV; ion current density=0.4 mA/cm²). The surfaces were then coated with a thin film of gold in a Quick Coater vacuum evaporator (Type SC-701, Sanyu Denchi, Tokyo, Japan). Observations were conducted using a scanning electron microscope (SEM; FE-8000, Elionix) at an operating voltage of 10 kV and magnification of 10,000×.

SEM examinations were conducted on representative wear facets of the four provisional resin-based composites. The specimens were randomly selected after wear measurements, and evaporation coating of samples was performed in the same manner as the polished specimens. The coated surfaces were visualized using SEM with an operating voltage of 10 kV and magnifications of 50× and 2,500×.

### Statistical analysis

Because of their homogeneity of variance (Bartlett’s test) and normal distribution (Kolmogorov-Smirnov test), the data for each material were subjected to analysis of variance (ANOVA) followed by Tukey’s honestly significant difference (HSD) test at a significance level of 0.05. Two-way ANOVA followed by Tukey’s HSD test (\( \alpha=0.05 \)) was used for the analysis of the \( \sigma_f, E, \) and \( K_{IC} \) values of the provisional resin-based composites. MD and VL values after the simulated wear test were analyzed using one-way ANOVA followed by Tukey’s HSD test (\( \alpha=0.05 \)). To understand the inter-relationships between the tested parameters, the Pearson product-moment correlation coefficient was used for pairwise comparisons. Statistical analyses were performed using a software system (Sigma Plot version 13.0, SPSS, Chicago, IL, USA).
RESULTS

Flexural properties

The flexural properties ($\sigma_F$, $E$, and $R$) of the DCPRs are shown in Tables 2, 3, and 4. Two-way ANOVA revealed that the factors of the tested material (DCPRs under different curing modes and PP) and number of TCs significantly influenced $\sigma_F$ values ($p<0.001$), and that the interaction between the two factors was significant ($p<0.001$). Regarding the $E$ of the provisional resin-based composites tested, two-way ANOVA revealed that the factors of the tested material and number of TCs significantly influenced $E$ values ($p<0.05$), and that the interaction between the two factors was significant ($p<0.001$). For resilience, two-way ANOVA revealed that the factors of the tested material and number of TCs significantly influenced $R$ values ($p<0.01$), and that the interaction between the two factors was significant ($p<0.001$), as for the other parameters.

Regardless of aging condition, the dual-cure group

Table 2 Influence of thermal cycling on flexural strength

| Material | Baseline | TC 5,000 | TC 10,000 |
|----------|----------|----------|-----------|
| TS DC    | 116.7 (4.5)abA | 107.7 (6.4)bB | 108.2 (4.0)aB |
| TS SC    | 102.0 (5.1)dA | 99.3 (6.8)bcA | 97.6 (4.4)ba |
| LX DC    | 91.4 (4.6)bB | 104.0 (9.6)cA | 88.9 (7.3)cB |
| LX SC    | 80.5 (5.2)dA | 81.4 (7.5)dA | 78.2 (8.3)dB |
| IG DC    | 121.3 (6.9)cA | 118.3 (4.4)aA | 104.1 (7.5)abB |
| IG SC    | 113.0 (4.2)bcA | 110.3 (2.5)aA | 102.9 (5.0)abB |
| PP       | 107.8 (5.4)cdA | 107.5 (4.3)bcA | 101.2 (5.5)abB |

Unit: MPa. Values in parentheses indicate standard deviation.
DC: Dual cure mode, SC: Self cure mode
Same lower case letter in vertical columns indicates no difference at 5% significance level.
Same capital letter in horizontal rows indicates no difference at 5% significance level.

Table 3 Influence of thermal cycling on elastic modulus

| Material | Baseline | TC 5,000 | TC 10,000 |
|----------|----------|----------|-----------|
| TS DC    | 3.7 (0.3)aAB | 3.8 (0.2)aA | 3.5 (0.2)ab |
| TS SC    | 3.5 (0.2)aA | 3.6 (0.3)abA | 3.4 (0.2)abA |
| LX DC    | 2.4 (0.2)cA | 2.9 (0.1)cB | 2.4 (0.1)cA |
| LX SC    | 1.9 (0.1)dA | 1.8 (0.1)dA | 2.0 (0.1)dA |
| IG DC    | 3.4 (0.3)abA | 3.6 (0.3)abA | 3.6 (0.2)aA |
| IG SC    | 3.0 (0.2)bA | 3.2 (0.3)bcA | 3.1 (0.3)cA |
| PP       | 3.3 (0.3)abA | 3.5 (0.2)abA | 3.2 (0.4)abA |

Unit: GPa. Values in parentheses indicate standard deviation.
DC: Dual cure mode, SC: Self cure mode
Same lower case letter in vertical columns indicates no difference at 5% significance level.
Same capital letter in horizontal rows indicates no difference at 5% significance level.

Table 4 Influence of thermal cycling on resilience

| Material | Baseline | TC 5,000 | TC 10,000 |
|----------|----------|----------|-----------|
| TS DC    | 1.85 (0.20)bA | 1.62 (0.26)abA | 1.65 (0.15)aA |
| TS SC    | 1.51 (0.13)cA | 1.41 (0.19)bA | 1.42 (0.13)aA |
| LX DC    | 1.78 (0.18)bcA | 1.94 (0.37)aA | 1.68 (0.37)aA |
| LX SC    | 1.70 (0.31)bcA | 1.82 (0.34)aA | 1.53 (0.36)aA |
| IG DC    | 2.22 (0.34)aA | 1.98 (0.22)aA | 1.57 (0.24)abB |
| IG SC    | 2.17 (0.18)aA | 1.90 (0.17)aB | 1.52 (0.20)cA |
| PP       | 1.80 (0.14)bcA | 1.66 (0.24)abA | 1.67 (0.23)abA |

Unit: MJ/mm³. Values in parentheses indicate standard deviation.
DC: Dual cure mode, SC: Self cure mode
Same lower case letter in vertical columns indicates no difference at 5% significance level.
Same capital letter in horizontal rows indicates no difference at 5% significance level.
exhibited a higher $\sigma_F$ value than the self-cure group. In particular, all DCPRs in the baseline group showed a significantly higher $\sigma_F$ value with dual-curing than with self-curing. Most of the provisional resin-based composites showed a decreased $\sigma_F$ value with increasing numbers of TCs, and significantly lower $\sigma_F$ values were evident at 10,000 TCs for dual-cured TS, dual-cured IG, self-cured IG, and PP compared with the baseline group.

The dual-cure group tended to show a higher $E$ value than the self-cure group, regardless of aging period. Most of the provisional resin-based composites showed higher $E$ values at 5,000 TCs than at baseline. However, most of the provisional resin-based composites tested showed lower $E$ values at 10,000 TCs than at 5,000 TCs.

The dual-cure group tended to show a higher $R$ value than the self-cure group, regardless of aging period, as for the other flexural parameters. Most tested materials tended to show decreased $R$ values with increased aging periods. In particular, IG at TC 10,000 showed significantly lower $R$ values than at the baseline, in both curing modes. Although there were significant differences in $R$ values among the tested materials in the baseline groups, no significant differences were found at TC 10,000.

Fracture toughness

The results of $K_{IC}$ testing are shown in Table 5. Two-way ANOVA revealed that the factors of the tested material and number of TCs significantly influenced $K_{IC}$ values ($p<0.001$), and that the interaction between the two factors was significant ($p<0.001$).

There were no significant differences in $K_{IC}$ irrespective of aging period between the dual- and self-cure groups, except for with LX relative to baseline. Under all aging conditions, although there was no significant difference in $K_{IC}$ between PP and TS, TS showed significantly higher $K_{IC}$ values than the other DCPRs regardless of curing mode. There were three possible trends in $K_{IC}$ as the number of TCs increased, all of which were observed. That is, regardless of curing mode, there was no change in $K_{IC}$ for TS, $K_{IC}$ decreased with increasing TCs for IG, and $K_{IC}$ tended to increase with increasing TCs for LX and PP.

Sliding impact wear-simulation

The facet wear depth (MD) and volumetric loss (VL) values of the provisional resin-based composites are shown in Table 6. The rank order of all groups was the same as that for MD and VL. IG in dual-cure mode showed significantly lower MD and VL values than the other provisional resin-based composites. Conversely, LX in self-cure mode and PP showed significantly

Table 5 Influence of thermal cycling on fracture toughness

|                  | Baseline | TC 5,000 | TC 10,000 |
|------------------|----------|----------|-----------|
| TS DC            | 2.53 (0.20)aa | 2.57 (0.11)aa | 2.56 (0.11)aa |
| TS SC            | 2.58 (0.07)aa | 2.56 (0.14)aa | 2.56 (0.09)aa |
| LX DC            | 1.86 (0.28)cA | 1.94 (0.12)bA | 1.93 (0.14)bA |
| LX SC            | 1.61 (0.06)dB | 1.87 (0.20)bA | 1.91 (0.17)bA |
| IG DC            | 2.26 (0.16)bA | 1.96 (0.18)bB | 1.67 (0.21)cC |
| IG SC            | 2.28 (0.11)bA | 2.02 (0.28)bB | 1.51 (0.15)cC |
| PP               | 2.40 (0.20)abB | 2.65 (0.22)aA | 2.68 (0.18)aA |

Unit: MPa $\cdot$ m$^{1/2}$. Values in parentheses indicate standard deviation.

DC: Dual cure mode, SC: Self cure mode

Same lower case letter in vertical columns indicates no difference at 5% significance level.

Table 6 Maximum facet depth ($\mu$m) and volume loss (mm$^3$) after sliding-impact wear test

|                  | Maximum depth ($\mu$m) | Volume loss (mm$^3$) |
|------------------|------------------------|----------------------|
| TS DC            | 57.9 (4.1)c             | 0.0392 (0.004)c      |
| TS SC            | 66.9 (6.1)c             | 0.0475 (0.008)c      |
| LX DC            | 75.8 (8.5)b             | 0.0560 (0.008)b      |
| LX SC            | 92.9 (6.7)a             | 0.0786 (0.009)a      |
| IG DC            | 39.5 (4.9)d             | 0.0227 (0.004)d      |
| IG SC            | 65.5 (4.9)c             | 0.0422 (0.005)c      |
| PP               | 91.8 (8.2)a             | 0.0748 (0.007)a      |

Values in parentheses indicate standard deviation.

DC: Dual cure mode, SC: Self cure mode

Same lower case letter in vertical columns indicates no difference at 5% significance level.
higher MD and VL values than the other resins. When comparing wear behavior among the provisional resin-based composites in dual- and self-cure modes, significant differences were evident for both MD and VL, except for with TS.

**Inter-relationships between the tested parameters**

The inter-relationships as measured by the Pearson product-moment correlation coefficient ($r$) and $p$ values between the parameters tested in this study are shown in Table 7. In addition, representative inter-relationships between the tested parameters are presented in Figs. 1 and 2. Strong positive correlations were observed between MD vs. VL ($r=0.991$, $p<0.001$, Fig. 1), $E$ vs. $K_{IC}$ ($r=0.969$, $p<0.001$, Fig. 2), $\sigma_F$ vs. $E$ ($r=0.865$, $p<0.05$), and $\sigma_F$ vs. $K_{IC}$ ($r=0.771$, $p<0.05$). In addition, strong negative correlations were observed between $\sigma_F$ vs. MD and $\sigma_F$ vs. VL ($r=-0.755$, $p<0.05$ and $r=-0.769$, $p<0.05$). However, weak or no correlations were observed between the other parameters.

**Scanning electron microscopy observations**

SEM images of the four materials tested after argon-ion etching are presented in Fig. 3. Although each provisional resin exhibited differences in filler size, shape, and distribution, the tested materials were divisible into two

### Table 7  Interrelationships between the tested parameters

| Correlation coefficient ($r$) | $p$ value |
|-------------------------------|-----------|
| $\sigma_F$ vs. $E$            | 0.865     | 0.012 |
| $\sigma_F$ vs. $R$            | 0.640     | 0.121 |
| $\sigma_F$ vs. $K_{IC}$       | 0.771     | 0.042 |
| $\sigma_F$ vs. MD             | -0.755    | 0.044 |
| $\sigma_F$ vs. VL             | -0.769    | 0.043 |
| $E$ vs. $R$                   | 0.169     | 0.717 |
| $E$ vs. $K_{IC}$              | 0.969     | $<0.001$ |
| $E$ vs. MD                    | -0.600    | 0.154 |
| $E$ vs. VL                    | -0.611    | 0.145 |
| $R$ vs. $K_{IC}$              | 0.036     | 0.939 |
| $R$ vs. MD                    | -0.579    | 0.173 |
| $R$ vs. VL                    | -0.590    | 0.164 |
| $K_{IC}$ vs. MD               | -0.457    | 0.303 |
| $K_{IC}$ vs. VL               | -0.490    | 0.264 |
| MD vs. VL                     | 0.991     | $<0.001$ |

$\sigma_F$: flexural strength, $E$: elastic modulus, $R$: resilience, $K_{IC}$: fracture toughness, MD: maximum depth, VL: volume loss

**Fig. 1**  The inter-relationship between elastic modulus and fracture toughness.

- $E$: elastic modulus, $K_{IC}$: fracture toughness, TS: Tempsmart, LX: Luxatem Automix Solar, IG: Integrity Multi Cure, PP: Protemp Plus, DC: dual cure mode, SC: self cure mode

**Fig. 2**  The inter-relationship between maximum depth and volume loss.

- MD: maximum depth, VL: volume loss, TS: Tempsmart, LX: Luxatem Automix Solar, IG: Integrity Multi Cure, PP: Protemp Plus, DC: dual cure mode, SC: self cure mode
groups. TS and PP employ nanosized, almost spherical particles (Figs. 3A and D), whereas LX and IG employ irregular glass filler particles (Figs. 3B and C). Distinct aggregates of nanofiller particles were observed in TS but not in PP. Although 0.02–2.5-µm irregular glass filler particles were observed in LX (Fig. 3B), the typical filler size in IG was noticeably smaller than that of LX, at approximately 0.02–1 µm (Fig. 3C).

Representative SEM images of the wear facets after sliding impact wear testing are presented in Fig. 4. The morphologic appearance of the wear facets was material-, curing mode-, and location-dependent. In general, the wear facets of DCPRs in self-cure mode tended to have larger facets, rougher surfaces, and more cracks than those of DCPRs in dual-cure mode, regardless of the material. In addition, the center of the wear facet exhibited a rougher surface than the edge. Regarding material differences, TS and PP exhibited a similar wear pattern with smooth surfaces, in which it was difficult to observe evidence of plucking of the fillers (Figs. 4A and B). However, IG and LX in self-cure mode exhibited a similar wear pattern with rough surfaces (Figs. 4D and F). The surface texture of LX was noticeably rougher than that of the other DCPRs, and some plucking of the irregularly shaped filler particles was evident (Figs. 4C and D).

**DISCUSSION**

The $\sigma_F$ test simultaneously measures different types of stress, including tensile and compressive stresses [21], and is relatively easy to conduct using standard methods for the strength testing of dental materials. In this study, the $\sigma_F$ of DCPRs after TCs were higher in dual-cure mode than in self-cure mode, regardless of aging period. Most of the provisional resin-based composites showed decreased values with an increased number of TCs. Shibasaki et al. investigated the initial polymerization behavior of these DCPRs using ultrasonic measurement, and reported that all of the DCPRs exhibit a lower polymerization rate with self-curing than with dual-curing [12]. Lower polymerization of DCPRs may lead to higher water absorption, resulting in a lower $\sigma_F$ during the course of TCs.

The aging process during TCs is mainly caused by thermal stress and water absorption. In particular, thermal stress due to discrepancies in thermal expansion rates between the inorganic filler and resin matrix is a critical factor, because defects and cracks caused by thermal stress induce the percolation and breakdown of poorly polymerized oligomers within provisional resin-based composites [21,22]. A previous study measuring the coefficient of thermal expansion of bis-acryl provisional resin-based composites revealed that their rates were
two to three times higher than those of direct resin composites. In addition, water uptake into the polymer network induces swelling, which is associated with reduction of the intermolecular forces between polymer chains. A higher coefficient of thermal expansion and water uptake over time may lead to accelerated degradation of bis-acryl provisional resin-based composites after TCs.

$E$ is considered related to elastic deformation under high forces. Therefore, it is necessary to understand this property to predict the deformation of provisional restorations exposed to occlusal forces under intraoral conditions. Similar to the obtained $\sigma_F$ values, the $E$ values of the DCPRs after TCs were higher in dual-cure mode than in self-cure mode, regardless of aging period. In addition, a statistically significant positive correlation was observed between $E$ vs. $\sigma_F$ for the materials tested. However, when the flexural properties of the provisional resin-based composites tested were compared with those of eight different types of direct resin composite in a previous study, although four of the eight resin composites showed almost identical $\sigma_F$ values, their $E$ values were two or three times higher than those of the provisional resin-based composites in this study. Therefore, to enhance the handling properties of provisional resin-based composites by maintaining their flexibility and plasticity after initial polymerization, bis-acryl resin-based restorations, some bis-acryl provisional resin-based composites contain a plasticizer. The plasticizer is thought not to chemically bond to the monomer network, and may influence post-cure polymerization and degradation processes during TCs. TS does not contain any plasticizer, which may explain its relatively stable $K_{IC}$ values relative to the other DCPRs.

Regarding the artificial aging in $K_{IC}$ during TCs, three possible trends existed. Regardless of curing mode, there were no changes in $K_{IC}$ for TS, $K_{IC}$ decreased with increasing TCs for IG, and $K_{IC}$ tended to increase with increasing TCs for LX and PP. These trends presumably result from the different compositions of the provisional resin-based composites. In particular, to maintain flexibility and plasticity during the fabrication of provisional restorations, some bis-acryl provisional resin-based composites contain a plasticizer. The plasticizer may be related to the nanosized filler particles employed in TS and PP. An important factor for $K_{IC}$ improvement is reduction of the interparticle space, which serves as buffer to the soft polymer matrix. Also, the integration of nanosized particles reportedly increases the $K_{IC}$ values of resinous materials by strengthening the interface between the particles and polymer matrix. In addition, the presence of nanoparticles induces a pinning effect due to crack deviation, in which a crack begins from one pole of the nanoparticle, moves across the interface, and passes around the nanoparticle.

Although the three-body contact simulated wear model can provide information on the influence of simulated food media on the wear behavior of restorations during mastication, their relative contribution remains...
poorly understood. It is difficult to standardize media mixtures, including their viscosities and compositions, during the entire wear-simulation process. The simulated media may form an embedded layer on the surface of the tested material that is not observed with food particles under normal intraoral conditions\textsuperscript{[57]}. In contrast, the two-body contact wear method is advantageous because it involves a simplified model and eliminates variability through its lack of media. This wear method may directly illuminate the inter-relationships between the mechanical property data obtained and wear outcomes. In sliding impact wear tests in this study, MD and VL were material-dependent, similarly to mechanical properties. In particular, IG in dual-cure mode showed significantly lower wear values than the other materials tested. Conversely, LX in self-cure mode and PP showed significantly higher wear values than the other resins. When comparing the wear behavior of DCPRs in dual- and self-cure modes, self-cure mode yielded higher wear values than dual-cure mode, regardless of material. Therefore, the second null hypothesis to be tested, that curing mode would have no effect on the wear behavior of DCPRs, was rejected.

The relationship between the mechanical properties and wear resistance of resin-based materials has been investigated extensively\textsuperscript{[8,11,13,36,39]} In this study, $K_{IC}$ vs. wear, $R$ vs. wear, and $E$ vs. wear exhibited moderate correlations, whereas $\sigma_F$ showed a strong correlation with wear parameters. A systematic review of clinical trials investigating the laboratory measured mechanical properties of posterior resin composites and their relationship with fracturing and wear concluded that, although there was no correlation between $K_{IC}$ and clinical wear, $\sigma_F$ showed a moderate correlation with clinical wear\textsuperscript{[8]}. Conversely, although $\sigma_F$ was not correlated with clinical material fracturing, $K_{IC}$ had a weak correlation with clinical fracture rates\textsuperscript{[8]}. Our laboratory study is consistent with these conclusions.

There is little evidence of a relationship between clinical wear and the degree of conversion of the polymer matrix of resin composites\textsuperscript{[57]}. Although the DCPRs tested are similar in composition to direct resin composites, they include a larger proportion of resin matrix than direct resin composites. Therefore, the initial degree of conversion of bis-acryl provisional resin-based composites plays an important role in their resistance to biomechanical stress under intraoral conditions. Ultrasonic measurements of the polymerization process under different curing modes revealed that DCPRs in dual-cure mode showed a significantly higher ultrasonic velocity than DCPRs in self-cure mode\textsuperscript{[12]}. In general, although ultrasonic measurement is unable to detect the degree of conversion directly, an increased ultrasonic velocity indicates an increased $E^\text{090}$. Therefore, it is possible to speculate that dual-cure mode induces a higher degree of conversion of the resin matrices of DCPRs than self-cure mode.

The clinical implication of this study is that adequate light irradiation is necessary to reinforce the wear resistance and flexural properties of DCPRs. In order to prevent chipping and bulk fracturing of provisional restorations, it is important to not only select an appropriate provisional resin with a higher $K_{IC}$, but also take into account the details of clinical cases in which such resins have been used, including the location, type of restoration, and intended duration of use. In addition, further research is needed to investigate the influence of light irradiation conditions and timing on the mechanical properties of DCPRs.

**CONCLUSION**

In this study, we showed that DCPRs in dual-cure mode exhibited superior flexural properties and wear resistance to those in self-cure mode. In contrast, curing modes did not influence $K_{IC}$, and changes in $K_{IC}$ under TCs were material-dependent. SEM images of provisional resin-based composites showed different filler sizes, shapes, and distributions, but the resin composites fell into two groups: those with nanosized, near-spherical particles and those with irregular glass particles. Although the correlations between $K_{IC}$ vs. wear, $R$ vs. wear, and $E$ vs. wear were not significant, $\sigma_F$ exhibited a strong correlation with wear parameters.

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**REFERENCES**

1) Patras M, Naka O, Doukoudakis S, Pissiotis A. Management of provisional restorations' deficiencies: a literature review. J Esthet Restor Dent 2012; 24: 26-38.
2) Magne P, Magne M, Belser U. The diagnostic template: A key element to the comprehensive esthetic treatment concept. Int J Periodont Restor Dent 1996; 16: 560-569.
3) Luthardt RG, Stößel M, Hinz M, Vollandt R. Clinical performance and periodontal outcome of temporary crowns and fixed partial dentures: A randomized clinical trial. J Prosthet Dent 2000; 83: 32-39.
4) Burns DR, Beck DA, Nelson SK. A review of selected dental literature on contemporary provisional fixed prostodontic treatment: Report of the committee on research in fixed prosthodontics of the academy of fixed prosthodontics. J Prosthet Dent 2003; 90: 474-497.
5) Fondriest JF. Using provisional restorations to improve results in complex aesthetic restorative cases. Pract Proced Aesthet Dent 2006; 18: 217-224.
6) Kerby RF, Knobloch LA, Sharples S, Peregrina A. Mechanical properties of urethane and bis-acryl interium resin materials. J Prosthet Dent 2013; 110: 21-28.
7) Suting HJ, Kleverlaan C, Werner A, Feilzer AJ, Raghoebear GM, Meijer HJ. Occlusal wear of provisional implant-supported restorations. Clin Implant Dent Relat Res 2015; 17: 179-185.
8) Heintze SD, Ilie N, Hickel R, Reis A, Loguerio A, Rousson V. Laboratory mechanical parameters of composite resins and their relation to fractures and wear in clinical trials—A systematic review. Dent Mater 2017; 33: e101-e114.

9) Rosentritt M, Behr M, Lang R, Handel G. Flexural properties of prosthetic provisional polymers. Eur J Prosthodont Restor Dent 2004; 12: 75-79.

10) Akova T, Ozkumur A, Uysal H. Effect of food-simulating liquids on the mechanical properties of provisional restorative materials. Dent Mater 2006; 22: 1130-1134.

11) Takamizawa T, Barkmeier WW, Tsujimoto A, Scheidel DD, Erickson RL, Latta MA, et al. Mechanical properties and simulated wear of provisional materials. Oper Dent 2015; 40: 603-613.

12) Shibasaki S, Takamizawa T, Suzuki T, Nojiri K, Taujimoto A, Barkmeier WW, et al. Influence of different curing-modes on polymerization behavior and mechanical properties of dual-cured provisional resins. Oper Dent 2017; 42: 526-536.

13) Ooska S, Miyazaki M, Rikuta A, Moore BK. Influence of polymerization mode of dual-polymerization resin direct core foundation systems on bond strengths to bovine dentin. J Prosthodont Dent 2004; 92: 239-244.

14) Cekic-Nagas I, Ergun G, Vallitu PK, Lassila LVJ. Influence of polymerization mode on degree of conversion and micropush-out bond strength of resin core systems using different adhesive systems. Dent Mater J 2008; 27: 376-385.

15) Pereira SG, Fulgêncio R, Nunes TG, Toledano M, Osorio R, Carvalho RM. Effect of curing protocol on the polymerization of dual-cured resin cements. Dent Mater 2010; 26: 710-718.

16) Moosavi H, Hariri I, Sadr A, Thitthaweerat S, Tagami J. Effects of curing mode and moisture on nanoindentation mechanical properties and bonding of self-adhesive resin cement to pulp chamber floor. Dent Mater 2013; 29: 708-717.

17) Michaud PL, Price RBT, Labrie D, Rueggeberg FA, Sullivan B. Localised irradiance distribution found in dental light curing units. J Dent 2014; 42: 129-139.

18) Price RBT, Rueggeberg FA, Labrie D, Felix CM. Irradiance uniformity and irradiation from dental light curing units. J Esthet Restor Dent 2010; 22: 86-103.

19) International Organization for Standardization, ISO 4049:2009. Dentistry-Polymer-based restorative materials. International Organization for Standardization, 2009; Geneva, Switzerland.

20) Mese A, Palamarra JEA, Bagheri R, Fani M, Burrow MF. Fracture toughness of seven resin composites evaluated by three methods of I fracture toughness (KIC). Dent Mater J 2016; 35: 893-899.

21) De Munck J, Van Landuyt KL, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: Methods and results. J Dent Res 2005; 84: 118-132.

22) Powers JM, Wataha JC. Dental Materials: Properties and Manipulation Chapter 2 Properties of Materials, 10th ed. Missouri: Mosby, Elsevier Inc., USA, 2013. P. 14-25.

23) Shibasaki S, Takamizawa T, Nojiri K, Imai A, Tsujimoto A, Suzuki S, et al. Polymerization behaviour and mechanical properties of high-viscosity bulk-fill and low-shrinkage resin composites. Oper Dent 2017; 42: E177-E187.

24) Ferracane JL, Hopkin JK, Condon JR. Properties of heat-treated composites after aging in water. Dent Mater 1995; 11: 354-358.

25) Balkenhol M, Köhler H, Orbach K, Wöstmann B. Fracture toughness of cross-linked and non-cross-linked temporary crown and fixed partial denture materials. Dent Mater 2009; 25: 917-928.

26) Bettencourt AF, Neves CB, de Almeida MS, Pinheiro LM, Oliveira SA, Lopes LP, et al. Biodegradation of acrylic based resins: A review. Dent Mater 2010; 26: e171-e180.

27) Ferracane-JL. Resin-based composite performance: Are there some things we can’t predict? Dent Mater 2013; 29: 51-58.

28) Asmussen E, Peutzfeldt A. Influence of UEDMA, BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. Dent Mater 1998; 14: 51-56.

29) Ormäghi BP, Meier MM, Lohbauer U, Braga RR. Fracture toughness and cyclic fatigue resistance of resin composites with different filler size distributions. Dent Mater 2014; 30: 742-751.

30) Bonilla ED, Yashar M, Caputo AA. Fracture toughness of nine flowable resin composites. J Prosthodont 2003; 89: 261-267.

31) Abdulmohsen B, Parker S, Braden M, Patel MP. A study to investigate and compare the physicomechanical properties of experimental and commercial temporary crown and bridge materials. Dent Mater 2016; 32: 200-210.

32) Ferracane-JL. Resin composite—State of the art. Dent Mater 2011; 27: 29-38.

33) Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthodont 2002; 87: 642-649.

34) Ilie N, Hickel R, Valeanu AS, Huth KC. Fracture toughness of dental restorative materials. Clin Oral Investig 2012; 16: 489-498.

35) Topouzi M, Kontonasaki E, Bikiaris D, Papadopoulou L, Paraskevopoulos KM, Koidis P. Reinforcement of a PMMA resin for interim fixed prostheses with silica nanoparticles. J Mech Behav Biomed Mater 2017; 69: 213-222.

36) Hsieh TH, Lin C, Masania K, Lee JS, Taylor AC, Sprenger S. The toughness of epoxy polymers and fibre composites modified with rubber micro particles and silica nanoparticles. J Mater Sci 2010; 45: 1193-1210.

37) Heintze SD. How to qualify and validate wear simulation devices and methods. Dent Mater 2006; 22: 712-734.

38) Sumino N, Tsubota K, Takamizawa T, Shiratsuchi K, Miyazaki M, Latta MA. A critical review of the wear and flexural characteristics of flowable resin composites for posterior lesions. Acta Odontol Scand 2013; 71: 820-827.

39) Furuichi T, Takamizawa T, Tsujimoto A, Miyazaki M, Barkmeier WW, Latta MA. Mechanical properties and sliding-impact wear resistance of self-adhesive resin cements. Oper Dent 2016; 41: E83-E92.

40) Takubo C, Yasuda G, Murayama R, Ogura Y, Tonegawa M, Kurokawa H, et al. Fracture toughness of cross-linked and non-cross-linked temporary crown and fixed partial denture materials. Dent Mater 2009; 25: 917-928.

41) Bettencourt AF, Neves CB, de Almeida MS, Pinheiro LM, Oliveira SA, Lopes LP, et al. Biodegradation of acrylic based resins: A review. Dent Mater 2010; 26: e171-e180.

42) Ferracane-JL. Resin-based composite performance: Are there some things we can’t predict? Dent Mater 2013; 29: 51-58.

43) Asmussen E, Peutzfeldt A. Influence of UEDMA, BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. Dent Mater 1998; 14: 51-56.

44) Ormäghi BP, Meier MM, Lohbauer U, Braga RR. Fracture toughness and cyclic fatigue resistance of resin composites with different filler size distributions. Dent Mater 2014; 30: 742-751.

45) Bonilla ED, Yashar M, Caputo AA. Fracture toughness of nine flowable resin composites. J Prosthodont 2003; 89: 261-267.

46) Abdulmohsen B, Parker S, Braden M, Patel MP. A study to investigate and compare the physicomechanical properties of experimental and commercial temporary crown and bridge materials. Dent Mater 2016; 32: 200-210.

47) Ferracane-JL. Resin composite—State of the art. Dent Mater 2011; 27: 29-38.

48) Kim KH, Ong Jl, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthodont 2002; 87: 642-649.

49) Ilie N, Hickel R, Valeanu AS, Huth KC. Fracture toughness of dental restorative materials. Clin Oral Investig 2012; 16: 489-498.

50) Topouzi M, Kontonasaki E, Bikiaris D, Papadopoulou L, Paraskevopoulos KM, Koidis P. Biocompatibility of a PMMA resin for interim fixed prostheses with silica nanoparticles. J Mech Behav Biomed Mater 2017; 69: 213-222.

51) Hsieh TH, Lin C, Masania K, Lee JS, Taylor AC, Sprenger S. The toughness of epoxy polymers and fibre composites modified with rubber micro particles and silica nanoparticles. J Mater Sci 2010; 45: 1193-1210.

52) Heintze SD. How to qualify and validate wear simulation devices and methods. Dent Mater 2006; 22: 712-734.

53) Sumino N, Tsubota K, Takamizawa T, Shiratsuchi K, Miyazaki M, Latta MA. Comparison of the wear and flexural characteristics of flowable resin composites for posterior lesions. Acta Odontol Scand 2013; 71: 820-827.

54) Furuichi T, Takamizawa T, Tsujimoto A, Miyazaki M, Barkmeier WW, Latta MA. Mechanical properties and sliding-impact wear resistance of self-adhesive resin cements. Oper Dent 2016; 41: E83-E92.