INTRODUCTION

Nowadays, direct resin-based composites (RBCs) provide dental practitioners with esthetic restorations whose appearance and properties parallel those of natural teeth. For optimal clinical outcome, the selected RBC should match the shade and translucency of the surrounding natural teeth. A prerequisite to achieving optimal esthetics is to understand the optical and mechanical properties of both the natural teeth and the selected RBC.

Translucency is an intermediate state between complete opacity and complete transparency. Amongst the plethora of commercially available RBCs, translucency varied significantly within each brand according to shade designation; and for the same shade designation, translucency varied across the different brands. For the same brand and shade designation, the translucency of flowable RBC differed significantly from that of universal RBC. Both investigations have shown that the translucency of a RBC is influenced by its material composition.

Studies found that numerous factors affect the translucency of RBCs: refractive index mismatch, filler amount, and resin layer thickness. Translucency decreased as the difference in refractive indices between organic resin matrix and inorganic filler increased. Translucency also decreased when filler amount increased. As resin layer thickness diminished, translucency increased exponentially regardless of resin shade. In a multi-layer composite restoration, translucency was influenced by layer thickness and the proportion of dentin shade thickness to translucent shade thickness.

Aside from material composition and inherent optical properties, external factors such as aging and storage medium also affect the translucency of RBCs. Accelerated aging and environmental exposure such as daylight exposure decreased translucency, and so did storage/immersion in media such as salivary esterase, water, and whisky. A study found that translucent shades were more susceptible to discoloration than enamel shades.

Apart from exerting adverse influence on the optical properties of RBCs, aging also affected the mechanical properties of RBCs. Storage media such as water and artificial saliva produced the same effect on the physical properties (such as flexural strength and Vickers hardness) of RBCs, but alcohol resulted in more inferior performance. Some studies showed that storage duration had a more pronounced effect on the mechanical properties of RBCs than storage medium. Two reasons accounted for the different results among RBCs when they were subjected to the same storage condition: their chemical composition and structure. RBCs with a low resin matrix content showed low water sorption and high flexural strength and flexural modulus after storage in water or artificial saliva. Similarly, the chemical bonding between resin matrix and filler particles wielded a significant influence on the mechanical properties of RBCs after aging.

For studies on materials aging of translucent RBCs, the Translucent shade of Filtek Supreme XT (Filtek Supreme XT Translucent, 3M ESPE, St. Paul, MN, USA) seemed to be a popular material of choice. A quick survey of these studies revealed that different shades of Filtek Supreme XT yielded different performance characteristics. In one study, it was reported that
the water content of Filtek Supreme XT Translucent continued to increase throughout the 12-month water storage, whereas water uptake of the Body shade (Filtek Supreme XT Body) increased up to 3 months and then reached equilibrium. Filtek Supreme XT Translucent had a higher resin matrix content than Filtek Supreme XT Body, which might thus account for its higher volumetric shrinkage and water uptake.

Limited cure depth is a drawback of light-activated composites. The rate of cure of RBCs is affected by their shade and translucency, because these optical properties interfere with light transmission. For example, Filtek Supreme XT Translucent showed a higher rate of cure than the Dentin and Enamel shades. Other factors that affect the rate of cure of RBCs include the type of light curing unit used (with different light intensity outputs and spectral distributions), irradiation time, and distance from the curing light tip.

Generally, translucent shades of direct esthetic composites were less examined for their mechanical properties than for their optical properties. Therefore, the aim of this study was to investigate the effects of aging and irradiation time on the mechanical properties of a highly translucent RBC. Macro-mechanical properties examined were flexural strength and flexural modulus; micro-mechanical properties examined were Vickers hardness, indentation modulus, and creep. The influence of irradiation time on the rate of cure was also examined.

Three research hypotheses were tested in this study: (1) Aging by storage would not influence the mechanical properties of highly translucent RBC; (2) Irradiation time range between 5 and 40 s would not influence the mechanical properties of highly translucent RBC; (3) Rate of cure would not differ between the top and bottom surfaces of highly translucent RBC.

MATERIALS AND METHODS

The highly translucent RBC selected for this study was a nanohybrid composite of 45% translucency, IPS Empress Direct (Trans Opal shade, Ivoclar Vivadent, Schaan, Liechtenstein; Batch No. M62836). Its composition included dimethacrylate resin matrix, prepolymer and highly dispersed silicon dioxide.

Preparation of RBC specimens for mechanical properties testing

Composite material was filled into a steel mold with internal dimensions of 16×2×2 mm. Top and bottom surfaces of filled steel mold were covered with polyacrylate sheets and pressed with glass plates. Irradiation was performed using a LED curing light (Elipar Freelight 2, 3M ESPE, Seefeld, Germany; 1.261 mW/cm²) from the top and bottom of the specimens, as specified in ISO/DIN 4049:2009 standards. Irradiation times were 5, 10, 20, and 40 s with three light exposures each per side. Each light exposure overlapped one irradiated section no more than 1 mm of the diameter of the light guide to prevent multiple polymerization.

After removal from the mold, a total of 180 specimens were ground with 4,000-grit silicon carbide papers (Leco, Mönchengladbach, Germany) to remove any flash and excess. All specimens were then stored in 37°C distilled water for 24 h. Fifteen specimens per irradiation time were used as control. The remaining specimens (n=30 per irradiation time) were additionally aged by thermocycling for 5,000 times between 5 and 55°C with a 30-s dwell time each, and then stored for 4 weeks at 37°C in artificial saliva (n=15 per irradiation time) or in a 1:1 alcohol-water mixture (n=15 per irradiation time).

Evaluation of macro-mechanical properties

Flexural strength and flexural modulus were determined using a three-point-bending test (n=15 per irradiation time per storage condition). Test was carried out using a universal testing machine (Z 2.5, Zwick Roell, Ulm, Germany) with a three-point bend fixture, which had a span length of 12 mm between the supports and which was constructed according to the guidelines of NIST No. 4877.

During the test, specimens were immersed in distilled water at room temperature. Specimens were loaded at a crosshead speed of 0.5 mm/min until failure occurred. The universal testing machine measured the force during bending as a function of beam deflection. Flexural modulus was calculated from the slope of the linear part of the force-deflection curve.

Evaluation of micro-mechanical properties

Among the three-point bending test specimens, six fragments for each irradiation time and storage condition were randomly selected for Vickers hardness (HV), indentation modulus (E), and creep tests. These micro-mechanical properties were measured using a microhardness measuring system (Fischerscope H100C, Helmut Fischer GmbH, Sindelfingen, Germany) as prescribed in German standard DIN 50359-1:1997-10. Measurements were done on the top surfaces of fragment slabs (10 measurements per slab; 60 measurements for each irradiation time and storage condition).

Prior to testing, specimens were ground sequentially on 2,500- and 4,000-grit silicon carbide papers (Hermes, Hamburg, Germany) in a grinding system (EXAKT 400CS plate grinder equipped with AW 110 controller, EXAKT, Norderstedt, Germany). Test was performed by applying controlled force, with the test load increasing and decreasing at a constant speed between 0.4 and 500 mN. Load and penetration depth of the indenter were continuously measured during the loading-unloading hysteresis.

Universal hardness (HU) is defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements, a conversion factor between HU and HV was derived and implemented in the software, so that measurement results were presented in the more familiar HV units.

Indentation modulus, which matches a material’s modulus of elasticity, was determined from the slope of the tangent of the unloading curve at maximum load.
To measure creep, a constant load was applied for 5 s. Indentation creep was determined from the change in indentation depth whilst the applied load was maintained constant for 5 s.

For specimens stored in artificial saliva and alcohol, their fractured surfaces after three-point bending test were inspected and photographed using Fischerscope H100C at 40× magnification.

**Rate of cure measurement**

Rate of cure (RC) was examined using two different specimen geometries: one 2-mm-high increment measured in a white Teflon mold of 2 mm height and 3 mm diameter versus a 0.1-mm-thick composite film. Specimens were cured for 5, 10, 20, and 40 s by applying the light curing unit directly on specimen surface (n=6).

Using a Fourier transform infrared spectrometer with an attenuated total reflectance accessory (Nexus, Thermo Nicolet, Madison, USA), measurements were made in real-time (5-min measurement time, 2 spectra/s, 4 cm⁻¹ resolution). By placing the non-polymerized composite paste directly on the diamond attenuated total reflectance crystal, spectra at the bottom of both 0.1-mm-thick and 2-mm-thick specimens were thus recorded.

RC was calculated as the variation in peak height ratio of the absorbance intensities of methacrylate carbon double bond peak at 1,634 cm⁻¹ before curing and after 5, 10, 20, or 40 s of curing:

RC (%) = \left[ 1 - \frac{\text{Peak height after curing}}{\text{Peak height before curing}} \right] \times 100

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<tr>
<td></td>
<td>Storage condition</td>
<td>Storage condition</td>
<td>Storage condition</td>
</tr>
<tr>
<td>5</td>
<td>24 h water</td>
<td>4 w saliva</td>
<td>4 w alcohol</td>
</tr>
<tr>
<td>5</td>
<td>2.2⁴ C</td>
<td>2.8⁸ D,E</td>
<td>1.4¹ A</td>
</tr>
<tr>
<td>10</td>
<td>2.6⁴ B</td>
<td>2.9⁸ D,E</td>
<td>1.6¹¹ A,B</td>
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<td>20</td>
<td>3.1¹⁴ E,F</td>
<td>3.5¹⁸ G</td>
<td>1.8¹¹ B</td>
</tr>
<tr>
<td>40</td>
<td>3.3¹⁴ E,F</td>
<td>3.2¹⁸ F</td>
<td>1.7¹¹ A,B</td>
</tr>
</tbody>
</table>

Flexural modulus and flexural strength values are listed as mean values and standard deviations (in parentheses). Same superscript letters indicate no statistically significant differences (Tukey’s HSD test, α=0.05).

**Table 1** Effects of irradiation time and storage condition on macro-mechanical properties

**Fig. 1** Surfaces of fractured specimens after three-point bending test, according to irradiation time and storage medium (40× magnification).
Fig. 2 Weibull analysis of the flexural strengths exhibited under these storage conditions: (a) 24 h in water; (b) 4 weeks in saliva; (c) 4 weeks in alcohol.

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**Fig. 2** Weibull analysis of the flexural strengths exhibited under these storage conditions: (a) 24 h in water; (b) 4 weeks in saliva; (c) 4 weeks in alcohol.

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**Statistical analysis**
Results were statistically compared using one-way ANOVA and Tukey’s HSD post hoc test ($\alpha=0.05$). A multivariate analysis (general linear model with partial eta-squared statistics) tested the effects of irradiation time and storage condition on the macro- and micro-mechanical properties (SPSS Inc., Chicago, IL, USA; Version 18.0). Weibull analysis was additionally performed for flexural strength data.

**RESULTS**

**Effects on macro-mechanical properties**
Table 1 presents the effects of irradiation time and storage condition on the macro-mechanical properties of highly translucent RBC, namely, flexural strength and flexural modulus. Among the three storage conditions, control specimens stored in water for 24 h showed the highest flexural strengths for all irradiation times especially when irradiated for 20 and 40 s. For each irradiation time, flexural strength significantly decreased after aging, with a more pronounced decrease after alcohol aging.

Figure 1 shows the surfaces of aged specimens obtained after three-point bending test. The flaw patterns observed well correlated with the flexural strength results. For all specimens aged in alcohol or irradiated for only 5 s, they exhibited similar flaws. The shorter the irradiation time, the larger the flaws.

For specimens aged for 24 h in water or 4 weeks in saliva, their Weibull modulus $m$ values were pronouncedly lower at 5-s irradiation time compared to being irradiated for 10 s or longer (Fig. 2). For specimens aged for 4 weeks in alcohol, they showed lower $m$ values than the other storage conditions at 10-, 20-, and 40-s irradiation times.

For specimens aged for 24 h in water or 4 weeks in saliva, they exhibited their highest flexural modulus values when irradiated for 20 or 40 s (Table 1). Aging in alcohol resulted in the lowest flexural modulus value among the three storage conditions for each irradiation time.

**Effects on micro-mechanical properties**
Table 2 presents the effects of irradiation time and storage condition on the micro-mechanical properties of highly translucent RBC. When aged in saliva and irradiated for 20 s or longer, RBC specimens showed significantly improved values for indentation modulus, Vickers hardness, and creep than the control specimens aged for 24 h in water, which in turn showed significantly higher values than those aged in alcohol.

When irradiation time was reduced to less than 20 s, the values of indentation modulus and Vickers hardness also decreased across all the three storage conditions. For creep, storage in alcohol led to inferior results at all irradiation times.

**Effects on rate of cure**
Table 3 shows the rates of cure at top surface (simulated by 0.1-mm-thick RBC film) and bottom surface (2 mm depth) for all irradiation times. At each irradiation time (except 40 s), significantly higher rates of cure were observed at the bottom surface than at the top surface. When irradiation time was 40 s, both the top and bottom surfaces yielded their highest rates of cure, which were not significantly different from each other. Figure 3 shows that as irradiation time decreased, the rate of cure decreased at both top and bottom surfaces.

**Effects of irradiation time and storage condition on mechanical properties**
Table 4 presents the effects of storage condition and irradiation time on macro- and micro-mechanical properties. Storage condition was found to have a stronger influence on macro-mechanical properties, whereas irradiation time had a greater influence on micro-mechanical properties.
Table 2: Effects of irradiation time and storage condition on micro-mechanical properties

<table>
<thead>
<tr>
<th>Time [s]</th>
<th>E [GPa]</th>
<th>HV [N/mm²]</th>
<th>Creep [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Storage condition</td>
<td>Storage condition</td>
<td>Storage condition</td>
</tr>
<tr>
<td>24 h water</td>
<td>4 w saliva</td>
<td>4 w alcohol</td>
<td>24 h water</td>
</tr>
<tr>
<td>5</td>
<td>4.4&lt;sup&gt;A&lt;/sup&gt; (1.1)</td>
<td>5.4&lt;sup&gt;B&lt;/sup&gt; (0.4)</td>
<td>4.4&lt;sup&gt;A&lt;/sup&gt; (0.2)</td>
</tr>
<tr>
<td>10</td>
<td>5.2&lt;sup&gt;CD&lt;/sup&gt; (0.6)</td>
<td>5.8&lt;sup&gt;K&lt;/sup&gt; (0.5)</td>
<td>4.7&lt;sup&gt;AB&lt;/sup&gt; (0.2)</td>
</tr>
<tr>
<td>20</td>
<td>6.7&lt;sup&gt;F&lt;/sup&gt; (0.7)</td>
<td>7.1&lt;sup&gt;C&lt;/sup&gt; (0.3)</td>
<td>5.1&lt;sup&gt;C&lt;/sup&gt; (0.2)</td>
</tr>
<tr>
<td>40</td>
<td>6.1&lt;sup&gt;E&lt;/sup&gt; (0.5)</td>
<td>7.1&lt;sup&gt;C&lt;/sup&gt; (0.6)</td>
<td>5.0&lt;sup&gt;RC&lt;/sup&gt; (0.3)</td>
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</table>

Modulus of elasticity (E), Vickers hardness (HV), and creep values are listed as mean values and standard deviations (in parentheses). Same superscript letters indicate no statistically significant differences (Tukey’s HSD test, α=0.05).

Table 3: Rates of cure at 0.1-mm and 2-mm depths

| Time [s] | RC<sub>0.1mm</sub> [%] | RC<sub>2mm</sub> [%] |
|---------|----------------|----------------|----------------|
| 5 | 29.5<sup>A</sup> (2.3) | 34.2<sup>B</sup> (1.5) |
| 10 | 35.2<sup>B</sup> (4.5) | 39.1<sup>C</sup> (2.1) |
| 20 | 39.3<sup>C</sup> (3.3) | 44.8<sup>B</sup> (1.7) |
| 40 | 43.2<sup>D</sup> (4.1) | 45.1<sup>D</sup> (2.2) |

Rates of cure (RC) at 0.1-mm and 2-mm depths are listed as mean values and standard deviations (in parentheses). Same superscript letters indicate no statistically significant differences (Tukey’s HSD test, α=0.05).

Fig. 3: Variation of rate of cure within 5 min after light curing according to irradiation time (mean value of 6 measurements per irradiation time): (a) 0.1 mm; (b) 2 mm.


**DISCUSSION**

*Effects of material composition*

The RBC examined in this study, IPS Empress Direct of Trans Opal shade, was distinctly unique from the IPS Empress Direct range of products. Apart from having the highest degree of translucency (45%), it boasted of a different chemical composition. Typically, ytterbium trifluoride and aluminosilicate glass are used to make dental restorative materials radiopaque. Radiopacity of dental materials is important because it permits tooth-colored restorative materials to be differentiated from the natural tooth or caries on X-rays. However, a high level of radiopacity and translucency cannot be achieved at the same time. In IPS Empress Direct of Trans Opal shade, highly dispersed silicon dioxide and an organic prepolymer were used instead of a high ratio of barium-aluminum-fluorosilicate glass and ytterbium trifluoride.

For IPS Empress Direct of Trans Opal shade, silicon dioxide was not considered as inorganic filler. As a result, the amounts of dimethacrylates in the monomer matrix (17 wt%) and inorganic filler particles (60.5 wt%, 45 vol%) were lower than the other shades of IPS Empress Direct products (21 wt%, 75–79 wt% or 52–59 vol% respectively). Translucency increases as filler amount decreases. The lowest amount of inorganic filler particles in Trans Opal shade, thus logically accounted for its highest degree of translucency amongst the wide range of shades and various levels of translucency offered by IPS Empress Direct products.

Changes in material composition and optical properties affect the mechanical properties of RBCs. Our previous study showed that the mechanical properties of IPS Empress Direct Trans Opal were lower than those of Dentin shade. It was also shown that IPS Empress Direct Trans Opal had below-average macro-mechanical properties when compared to other nanohybrid RBCs; its mechanical properties were more comparable to those of microfilled RBCs, flowable RBCs and compomers.

The clinical indication for Trans Opal shade is limited to esthetic anterior restorations to create the effects of opalescence and translucency in natural enamel. Three-point bending test in this study revealed that the flexural strength of Trans Opal shade was less than 80 MPa, which is the minimum flexural strength limit of ISO 4049 for restorative materials claimed suitable for restorations involving outer occlusal surfaces. IPS Empress Direct products (21 wt%, 75–79 wt% or 52–59 vol% respectively) 25) were lower than those of IPS Empress Direct Trans Opal shade, which were distinctly unique from the IPS Empress Direct range of products. Apart from having the highest degree of translucency (45%), it boasted of a different chemical composition.

### Table 4: Effects of storage condition and irradiation time on mechanical properties

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Flexural modulus</th>
<th>Flexural strength</th>
<th>Weibull</th>
<th>E</th>
<th>HV</th>
<th>Creep</th>
</tr>
</thead>
<tbody>
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<td>Storage condition</td>
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<td>.865</td>
<td>.195</td>
<td>.356</td>
<td>.269</td>
<td>.164</td>
</tr>
<tr>
<td>Irradiation time</td>
<td>.050</td>
<td>.119</td>
<td>.606</td>
<td>.471</td>
<td>.528</td>
<td>.318</td>
</tr>
</tbody>
</table>

Effects of storage condition and irradiation time on macro- and micro-mechanical properties and Weibull modulus $m$. The higher the partial eta-squared values, the higher is the influence of the variable on the measured property.

*Effects of irradiation time*

In the present study, the manufacturer-recommended time to light-cure a 2-mm increment with a light intensity of at least 1,000 mW/cm² was 10 s. The light curing unit used in this study had an intensity of 1,261 mW/cm². Therefore, a 10-s irradiation time was presumably sufficient. However, results obtained for macro- and micro-mechanical properties and rate of cure (Tables 1–3) showed that irradiation for 20 s or more led to significantly better performance. The results of this study agreed with those of Rueggeberg *et al.*

1. Rueggeberg, et al.
the use of manufacturer-recommended irradiation times produced lower flexural strength and scraped composite thickness than did prolonged irradiation times.

**Resin layer thickness versus rate of cure**

At depths of 3.5-5.5 mm, it was reported that the microroughness values of light-cured translucent RBCs were about 80% of the surface values\(^{(20)}\). As the clinical indication of translucent RBCs is for anterior restorations to mimic the optical properties of natural teeth, as opposed to restoring deep cavities in posterior teeth, a resin layer thickness greater than 2 mm is rarely used. Therefore in this study, rate of cure was investigated only for the top surface and at 2-mm depth.

Irrespective of irradiation time, rate of cure at 2-mm depth was higher than on the top surface. This could be because an oxygen inhibition layer was present on the top surface, which was produced when RBC was polymerized in air. Shawkat et al.\(^{(36)}\) showed that a decrease in composite viscosity brought about by an increase in diluent monomer content in the composite matrix led to an increase in oxygen inhibition layer thickness. However, the oxygen inhibition layer thickness of RBCs ranged between 4 and 40 μm. Nonetheless, this phenomenon of increased hardness at intermediate subsurface depths compared with the hardness at small depths was discussed in numerous studies, such as one by Asmussen and Peutzfeldt\(^{(47)}\).

Several reasons were cited for the increasing degree of cure at subsurface depths. They included proximity to the source of heat in polymerization unit or the shrinkage of unbonded light-cured RBC towards the center\(^{(39)}\). The latter served as a preferred explanation for RBCs, which had a reduced filler content and higher shrinkage than universal RBCs\(^{(20)}\).

**Research hypotheses: acceptance or rejection**

The three research hypotheses of this study asserted that storage condition and irradiation time would not influence the mechanical properties of highly translucent RBC and that rate of cure at 2 mm depth would not differ from that on the top surface of RBC. These hypotheses were rejected based on the findings of this study.

**CONCLUSIONS**

Within the limitations of the present study, the following conclusions were drawn:

1. Alcohol aging significantly reduced the mechanical properties of highly translucent RBC.
2. Aging in artificial saliva for 4 weeks produced a positive effect on micro-mechanical properties.
3. Irradiation time was recommended to be at least 20 s to yield favorable mechanical properties.
4. Highly translucent RBC was not indicated for restorations involving outer occlusal surfaces.

**REFERENCES**