Effect of filler type and polishing on the discoloration of composite resin artificial teeth

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In this study, the effects of filler type and polishing on the discoloration of composite resin artificial teeth were examined.

Four types of experimental resins were prepared: one was a matrix resin, while the others were composite resins containing three different types of fillers (nano-sized silica filler with or without silanization, and prepolymerized filler). Specimens were immersed in distilled water, coffee, red wine, or curry. Color change after immersion was measured using a colorimeter. Color difference values (ΔE) and changes in translucency parameter (ΔTP) were statistically analyzed using three-way ANOVA and Tukey’s comparison.

On the influence of the polishing factor, statistically significant differences were neither observed in ΔE nor ΔTP between polished and non-polished tooth surfaces. On the contrary, the influences of filler type and discoloration medium, and their interaction thereof, were significant. With unsilanized filler, the ΔE value of composite resin artificial teeth was significantly increased.

Key words: Resin composite, Color difference, Artificial teeth

INTRODUCTION

When natural teeth are lost, a denture is essential for prosthodontic rehabilitation. It is composed of a denture base, artificial teeth, and retainers. In particular, artificial teeth play the roles of restoring masticatory efficiency, pronunciation function, and esthetic appeal. To date, acrylic resins and porcelains have been used for the fabrication of artificial teeth. However, neither type completely fulfils the requirements of an ideal prosthetic tooth.

Porcelain teeth show good masticatory efficiency and good resistance to discoloration. However, they do not bond efficiently to denture base resins. As a result, their interfaces are frequently stained. With acrylic resin teeth, they can potentially achieve chemical bonding with denture base resins and exhibit acceptable resistance to discoloration. However, acrylic resin teeth have poor wear resistance. For this reason, acrylic resin teeth have been modified to overcome this disadvantage by using different monomers coupled with the addition of fillers. Consequently, new types of modified acrylic resin teeth and composite resin teeth have emerged and are now commonly used in dental practice.

Commercially available composite resin artificial teeth vary in multiple aspects as follows: filler shape, filler amount, polymer type, and degree of crosslinking. Despite their excellent physical properties as artificial teeth, they have poor resistance to discoloration in comparison to other materials.

On discoloration, several discoloration media such as coffee, tea, red wine, curry powder, fuchsin, and cola have been used to evaluate discoloration by colorimetric measurements. The discoloration of composite resin artificial teeth in these studies has been discussed primarily with regard to the polishing method and denture base processing method used. However, the components of composite resin teeth that are responsible for discoloration have not been clearly identified and confirmed. Furthermore, the effects of polishing on discoloration are inconsistent among different studies.

In the present in vitro study, experimental composite resin artificial teeth were prepared and the color difference and change of translucency of these teeth were measured after exposure to different discoloration media. These colorimetric measurements were taken to elucidate the effects of filler type and polishing on the resistance of the prepared artificial teeth to discoloration. The hypothesis of the present study was that filler type and polishing affect the resistance to discoloration of composite resin teeth.
MATERIALS AND METHODS

Experimental composite resin teeth

An experimental heat-polymerizing resin was prepared based on the formulation of a commercial composite resin artificial tooth\(^{16,17}\). The materials used in the present study are listed in Table 1. The ratio of urethane dimethacrylate, triethylene glycol dimethacrylate, and neopentylglycol dimethacrylate was 75/10/15 by mass%, and 1.0 mass% of benzoyl peroxide was added to initiate polymerization\(^{16,18}\).

Three types of fillers were used to prepare the experimental composite resins: nano-sized silica without silanization (OX; average particle size was 40 nm), nano-sized silica with silanization (RX; average particle size was 40 nm), and crushed prepolymerized microfine silica with trimethylolpropane trimethacrylate (TMPT; particle size ranged from 50 \(\mu m\) to submicrons) used for a commercial brand of composite artificial denture teeth\(^{16}\). The amount of filler content in the experimental composite resins was 20 mass%. Unfilled matrix resin was used as a control.

Disk-shaped specimens of the experimental unfilled resin and three composite resins were prepared using a Teflon mold (20 mm in diameter and 2.5 mm in height), covered with a glass plate, and heat-polymerized in a heating oven (DRM420DA, Advantek, Japan) at 80°C for 2 hours and at 120°C for 30 minutes. Twenty-four specimens of each resin were thus prepared.

Polishing procedure

One side of the specimen surface facing the Teflon mold was polished with silicone carbide paper (#400 to #1500), followed by diamond slurry (2 \(\mu m\)) with a polishing cloth and an aluminum oxide slurry (0.3 \(\mu m\)) with a polishing machine (Dialap ML150P, Maruto, Tokyo, Japan). This side was named the polished side. The other side of the specimen surface facing the glass plate remained unpolished and was named the glass side. Specimen size was measured using a digital micrometer (MDC-25M, Mitutoyo, Yokohama, Japan; minimum reading: 1 \(\mu m\)). Specimen thickness was controlled at 2.0 ± 0.05 mm.

Each specimen was immersed in distilled water at 37°C for 24 hours using a brown glass vial (50 ml).

Colorimetric measurements

Before performing the colorimetric measurements, specimens from the vials were washed with distilled water, dried by shaking in air for 30 seconds, and gently wiped with tissue paper. The color values of the specimens were measured using a colorimeter (CR-13, Konica Minolta, Tokyo, Japan; diameter of measurement area: 8 mm) on white (CIE \(L^* = 88.57, a^* = -1.17, b^* = 2.83\)) and black (CIE \(L^* = 36.37, a^* = -0.73, b^* = -0.8\)) backgrounds. The CIE \(L^*a^*b^*\) value of each specimen was calculated from the mean of three measurements. The CIE \(L^*a^*b^*\) system is defined as a color space with coordinates for whiteness-blackness (\(L^*\)), redness-greenness (\(a^*\)), and yellowness-blueness (\(b^*\)).

Total color difference (\(\Delta E\)) is expressed by the following formula:

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

where \(\Delta L^*\), \(\Delta a^*\), and \(\Delta b^*\) are differences in the respective \(L^*\), \(a^*\), and \(b^*\) values of white background\(^{15}\).

The change in translucency of each composite resin tooth after immersion was determined using the change of translucency parameter (TP) according to the following formula:

\[
TP = [(I_{v^*} - I_{w^*})^2 + (aw^* - aw^*)^2 + (bw^* - bw^*)^2]^{1/2}
\]

Table 1  Materials used in the present study

<table>
<thead>
<tr>
<th>Material</th>
<th>Brand</th>
<th>Lot No.</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urethane dimethacrylate</td>
<td>UDMA</td>
<td>N/A</td>
<td>Nagami Chemical Industrial, Japan</td>
</tr>
<tr>
<td>Neopentyl glycol dimethacrylate</td>
<td>Purity 95.7%</td>
<td>09624CU</td>
<td>Aldrich, Germany</td>
</tr>
<tr>
<td>Triethylene glycol dimethacrylate</td>
<td>NK Ester-3G</td>
<td>0919R</td>
<td>Shin-Nakamura Chemical, Japan</td>
</tr>
<tr>
<td>Benzoyl peroxide</td>
<td>1st grade of SAJ</td>
<td>A8345</td>
<td>Sigma Aldrich, Japan</td>
</tr>
<tr>
<td>Fillers</td>
<td></td>
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</tr>
<tr>
<td>Unsilanized silica nanofiller</td>
<td>OX50</td>
<td>N/A</td>
<td>Nippon Aerosil, Japan</td>
</tr>
<tr>
<td>Silanized silica nanofiller</td>
<td>RX50</td>
<td>N/A</td>
<td>Nippon Aerosil, Japan</td>
</tr>
<tr>
<td>Silanized silica prepolymerized filler</td>
<td>TMPT filler</td>
<td>171222-1</td>
<td>Nissin, Japan</td>
</tr>
</tbody>
</table>

N/A: Not available
where the subscript “W” refers to the CIE L*a*b* value for each specimen on the white background, and the subscript “B” refers to the value for each specimen on the black background\textsuperscript{13-19}. Changes in translucency (ΔTP) before and after immersion in discoloration media were thus calculated.

**Discoloration media**

Four types of discoloration media were prepared: distilled water, red wine (French wine), coffee, and curry solution.

Coffee was prepared by dissolving 3.6 g of instant coffee (Nescafe Gold Blend, Nestle Japan) in 350 ml of distilled water. Curry solution was prepared by dissolving 4.0 g of curry powder (spicy curry powder, S&B Shokuhin Co. Ltd.) in 350 ml of distilled water.

Six specimens of each composite resin and unfilled resin were immersed in one of the above-mentioned discoloration media using a shaking incubator (37°C, 1 Hz frequency). Discoloration media were changed every day. Color measurements were performed after immersion for one day and one week.

**Scanning electron microscopy (SEM) observation**

Carbon-coated polished and glass surfaces of one specimen of each unfilled resin and composite resin were observed after 1-week immersion in distilled water with a scanning electron microscope (S-4500, Hitachi High-Technologies Corp., Tokyo, Japan).

**Statistical analysis**

Changes in color difference (ΔE) and translucency parameter (ΔTP) were evaluated using three-way ANOVA. The main factors selected were namely — filler type, polishing, and discoloration medium, and their interactions. Mean values were compared using Tukey’s multiple comparison analysis (\(\alpha=0.05\)).

**RESULTS**

Discoloration of the specimens after 1-week immersion in coffee, red wine, and curry solution was easily recognized by naked eye. However, no obvious differences could be observed among filler types as well as with and without polishing.

Changes in color difference (ΔE) and translucency (ΔTP) after immersion are summarized in Tables 2 and 3 respectively.

As seen in Table 2, the smallest ΔE value was observed for the polished surface of unfilled resin specimen in water, whereas the greatest was at the glass surface of OX composite specimen in curry solution. Three-way ANOVA revealed that filler type, discoloration medium, and the interaction between these two factors were statistically significant factors affecting ΔE, but not so for polishing and the remaining interactions. Figure 1 shows the effects of the significant factors on ΔE. Tukey’s comparison revealed that the ΔE value of OX composite was significantly greater than those of the other fillers, whereas the ΔE value of TMPT was significantly smaller than the other fillers. In the same vein, the ΔE value of curry solution was significantly greater than the other immersion media. Further on curry solution immersion, the ΔE value of OX composite was significantly greater than the

<table>
<thead>
<tr>
<th>Type of resin</th>
<th>Baseline (L*, a*, b*)</th>
<th>water</th>
<th>coffee</th>
<th>wine</th>
<th>curry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass surface</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unfilled</td>
<td>81.1, -2.0, 4.7</td>
<td>0.54  (0.18)</td>
<td>1.85 (0.32)</td>
<td>1.69 (0.32)</td>
<td>11.33 (2.69)</td>
</tr>
<tr>
<td>OX</td>
<td>74.7, -4.0, -0.3</td>
<td>1.05  (0.52)</td>
<td>2.90 (1.07)</td>
<td>2.69 (0.41)</td>
<td>18.64 (14.78)</td>
</tr>
<tr>
<td>RX</td>
<td>74.9, -3.7, -0.6</td>
<td>1.11  (0.84)</td>
<td>2.01 (0.68)</td>
<td>2.16 (0.27)</td>
<td>10.10 (2.05)</td>
</tr>
<tr>
<td>TMPT</td>
<td>78.8, -2.3, 9.6</td>
<td>1.33  (0.46)</td>
<td>1.14 (0.28)</td>
<td>1.62 (0.54)</td>
<td>4.81 (0.83)</td>
</tr>
<tr>
<td>Polished surface</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unfilled</td>
<td>81.2, -1.9, 4.8</td>
<td>0.48  (0.11)</td>
<td>1.90 (0.37)</td>
<td>1.59 (0.25)</td>
<td>10.94 (2.29)</td>
</tr>
<tr>
<td>OX</td>
<td>74.8, -4.1, 0.0</td>
<td>1.04  (0.35)</td>
<td>2.85 (0.98)</td>
<td>2.58 (0.31)</td>
<td>15.06 (5.62)</td>
</tr>
<tr>
<td>RX</td>
<td>74.9, -3.8, -0.3</td>
<td>1.20  (0.88)</td>
<td>2.02 (0.62)</td>
<td>2.44 (0.24)</td>
<td>10.51 (2.21)</td>
</tr>
<tr>
<td>TMPT</td>
<td>78.9, -2.3, 9.7</td>
<td>1.25  (0.37)</td>
<td>1.17 (0.23)</td>
<td>1.50 (0.42)</td>
<td>4.96 (1.44)</td>
</tr>
</tbody>
</table>

Table 2 Baseline and color difference (ΔE) after immersion

Unfilled: unfilled resin; OX: composite resin containing nano-sized silica filler without silanization; RX: composite resin containing nano-sized silica filler with silanization; TMPT: composite resin containing crushed prepolymerized filler.
Color difference ($\Delta E$) at 1 week after immersion in four discoloration media. The topmost figure indicates $\Delta E$ of each filler, the middle figure indicates $\Delta E$ of each discoloration medium, and the bottom figure indicates $\Delta E$ of each filler in each discoloration medium. Bars with the same letter indicate differences that were not statistically significant. 95% CI: confidential interval of 95%.

Changes in translucency parameter ($\Delta TP$) at 1 week after immersion in four discoloration media. The topmost figure indicates $\Delta TP$ of each filler, the middle figure indicates $\Delta TP$ of each discoloration medium, and the bottom figure indicates $\Delta TP$ of each filler in each discoloration medium. Bars with the same letter indicate differences that were not statistically significant. 95% CI: confidential interval of 95%.

Other fillers and the $\Delta E$ value of TMPT significantly smaller than the other fillers.

As seen in Table 3, the smallest $\Delta TP$ value was observed for the unfilled composite specimen in water, whereas the greatest was exhibited by OX composite specimen in curry solution. Three-way ANOVA revealed that filler type, discoloration medium, and their interaction were statistically significant factors, but not so for polishing and the other interactions. Figure 2 shows the effects of the significant factors on $\Delta TP$. Tukey’s comparison revealed that the $\Delta TP$ value of the curry solution was significantly greater than the other discoloration media. Further on curry solution immersion, the $\Delta TP$ value of OX composite was significantly greater than unfilled and TMPT specimens. On water immersion, the $\Delta TP$ value of unfilled resin was significantly lower than the others.

Figure 3 shows the typical SEM images of the unfilled resin and composite resins after immersion. There were no distinct differences among the glass surfaces of unfilled resin and composite resins.
<table>
<thead>
<tr>
<th>Type of resin</th>
<th>Baseline</th>
<th>water</th>
<th>coffee</th>
<th>wine</th>
<th>curry</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glass surface</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unfilled</td>
<td>49.09 (0.61)</td>
<td>-1.64 (0.90)</td>
<td>-0.85 (0.70)</td>
<td>-0.99 (0.56)</td>
<td>0.89 (0.64)</td>
</tr>
<tr>
<td>OX</td>
<td>18.81 (0.57)</td>
<td>0.09 (0.15)</td>
<td>-0.51 (0.32)</td>
<td>-0.75 (0.25)</td>
<td>2.61 (2.44)</td>
</tr>
<tr>
<td>RX</td>
<td>15.28 (0.45)</td>
<td>-0.01 (0.38)</td>
<td>-0.17 (0.32)</td>
<td>-0.64 (0.32)</td>
<td>1.63 (0.35)</td>
</tr>
<tr>
<td>TMPT</td>
<td>39.39 (0.34)</td>
<td>0.81 (1.00)</td>
<td>0.48 (0.71)</td>
<td>-0.00 (0.61)</td>
<td>-0.63 (3.35)</td>
</tr>
<tr>
<td>Polished surface</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unfilled</td>
<td>49.31 (0.55)</td>
<td>-1.41 (0.94)</td>
<td>-0.87 (0.34)</td>
<td>-1.18 (0.71)</td>
<td>0.50 (0.42)</td>
</tr>
<tr>
<td>OX</td>
<td>18.79 (0.47)</td>
<td>-0.10 (0.56)</td>
<td>-0.45 (0.28)</td>
<td>-0.70 (0.51)</td>
<td>2.38 (1.77)</td>
</tr>
<tr>
<td>RX</td>
<td>15.26 (0.36)</td>
<td>0.06 (0.29)</td>
<td>-0.17 (0.29)</td>
<td>-0.84 (0.18)</td>
<td>1.70 (0.58)</td>
</tr>
<tr>
<td>TMPT</td>
<td>39.48 (0.34)</td>
<td>0.58 (0.71)</td>
<td>0.17 (0.97)</td>
<td>-0.02 (0.64)</td>
<td>1.10 (0.64)</td>
</tr>
</tbody>
</table>

Unfilled: unfilled resin; OX: composite resin containing nano-sized silica filler without silanization; RX: composite resin containing nano-sized silica filler with silanization; TMPT: composite resin containing crushed prepolymerized filler.

![SEM images of the glass surfaces (G) and polished surfaces (P) of specimens after water immersion.](image)

**Fig. 3** SEM images of the glass surfaces (G) and polished surfaces (P) of specimens after water immersion. Unfilled: unfilled resin; OX: composite resin containing nano-sized silica filler without silanization; RX: composite resin containing nano-sized silica filler with silanization; TMPT: composite resin containing crushed prepolymerized filler.

However, exposure of fillers could be observed on the polished surface of TMPT.

**DISCUSSION**

Many types and brands of composite resin artificial teeth are commercially available in the market. For an unfilled resin, the composition used in the present study was identical to that of a previously reported commercial product. Urethane dimethacrylate-based matrix resin has been reported to exhibit a higher degree of discoloration in comparison to another matrix resin. For filled composite resins, the filler size and content of the enamel layer of commercial composite resin artificial teeth are reported to range from 80 µm to submicrons and from 5.9 to 42.9 mass% respectively. In particular, micro-silica filler and prepolymerized micro-silica filler are often used for composite artificial teeth because they render a smooth surface. Furthermore, to achieve good bonding between silica and resin, a silane coupling process efficiently accomplishes this
objective. Therefore, two types of micro-silica fillers with and without silanization were used in the present study. As for trimethylolpropane trimethylacrylate filler, it contains residual double bonds on the surface that chemically bond to the resin matrix through copolymerization. The resultant highly cross-linked structure then exhibits improved mechanical properties.\textsuperscript{12,16,19}

The surfaces of the experimental composite resins facing the glass after heat polymerization were considered to be polymer-rich. After polishing with a diamond slurry, exposure of fillers and the interfaces of filler and resin were expected. However, contrary to expectation, the surface textures of both glass and polished surfaces were almost the same (Fig. 3). Therefore, the effects of polishing were considered to be due to filler exposure, and not surface roughness.

On the discoloration media employed, commonly consumed dietary liquids were used in the present study to evaluate the discoloration of composite resin teeth in an in vitro setting.

On colorimetric differences determined using color difference ($\Delta E$) with CIE L*a*b* system, it is because this method is a widely accepted quantitative and objective evaluation of color differences for various dental materials. Color differences greater than approximately one CIE L*a*b* unit were perceivable for 50% of normal human observers under appropriate inspection conditions. However, the average color difference of 3.7 is suggested to be an acceptable color match when resin composite veneer restorations were judged with their adjacent or contralateral natural teeth.\textsuperscript{10} Independently, a color difference of less than 3.3 has been reported to be clinically acceptable.\textsuperscript{10} The $\Delta E$ values of composite resin artificial teeth after 1-week immersion in coffee have been reported to range from 0.58 to 2.7, and from 2.0 to 11.4 for composite resin restorations.\textsuperscript{2,4,7,10,11} In the present study, the obtained $\Delta E$ values were within the range of previously reported values.

The experimental composite resins used in the present study were likened to the enamel layer. For artificial teeth, one important characteristic that affect the natural appearance is translucency. In the present study, translucency of the prepared artificial teeth was assessed using the translucency parameter (TP).

A greater TP means a greater translucency and less opacity. However, TP is influenced by a number of factors: thickness of the measured material, color of background, and change of color. The colorimeter used for the present study measured a relatively superficial area of the specimen. As specimen thickness was 2.0 mm\textsuperscript{14}, color change of the opposite side was considered negligible. TPs of commercial composite resins after polymerization have been reported to range from 3 to 18. In the present study, the TPs of OX and RX composites were within this range, but those of TMPT composite and unfilled resin were more translucent. The TP is computed based on differences of L*, $a^*$, and $b^*$, and usually L* is the greatest determining factor on TP.\textsuperscript{20} In the present study, not only the TPs of OX and RX increased after curry immersion, but that $b^*$ also increased in comparison to $a^*$ and L*. Therefore, the changes in TPs of OX and RX did not indicate an increase in translucency, but a change in the chrome attribute in this study.

Regardless of the discoloration medium, the discoloration of experimental unfilled resin and composite resins was ranked as follows: OX > RX = unfilled > TMPT. This result suggested that the trimethylolpropane trimethylacrylate composite exhibited better resistance to discoloration than the urethane dimethacrylate composite. Discoloration occurs in the matrix resin, filler, and at their interface. If the filler were not silanized, then gaps might form at the interface due to matrix resin swelling by water uptake. Consequently, the OX composite showed greater discoloration as compared to the other composites and unfilled resin. As for the effect of TMPT filler, commercial composite resin artificial teeth using TMPT filler have been reported to exhibit better resistance to discoloration than that using an organic filler.\textsuperscript{9} TMPT filler consists of approximately 43 mass% content of micro-silica embedded in resin, which meant that the inorganic content of TMPT composite was smaller than the RX composite. Consequently, $\Delta E$ of TMPT composite was smaller than the other composites.

On the effect of polishing, studies have suggested that polished composite resin restorations exhibited better resistance to discoloration.\textsuperscript{7,8,11,12} However, the effects of polishing of composite resin artificial teeth on discoloration are not consistent. On one hand, polishing of composite resin artificial teeth has been reported to reduce discoloration.\textsuperscript{7} On the other hand, scratching of composite resin artificial teeth due to polishing has been reported to increase discoloration — which means that polishing should be avoided.\textsuperscript{12} In the present study, the effect of polishing on $\Delta E$ and $\Delta TP$ were not significant. Specifically, the $\Delta E$ values of unfilled resin and RX composite were almost the same, although that of OX composite was significantly higher. These results indicated that the effect of polishing was not a strong influential factor in discoloration, if the filler were silanized and if well-polished surfaces could be attained. It is noteworthy that artificial polymer teeth are prone to being starched or stained during the denture fabrication process. Therefore, polishing the artificial teeth is acceptable if the tooth surface were damaged.
On the effect of the discoloration medium, the curry solution showed the highest $\Delta E$ and $\Delta TP$ values regardless of the type of composite. As for the ranking on discoloration of the composites in each discoloration medium, they were almost the same. These results were in agreement with those of previous studies, whereby the same tendency was reported and with the curry solution resulting in the highest degree of discoloration\(^6\)\(^-\)\(^10\). Therefore, the curry solution is indeed a suitable discoloration medium for discoloration evaluation.

During clinical usage, the denture is exposed to temperature changes (due to dietary habits) or chemical challenges (such as denture cleaners). These conditions must be taken into account as they also contribute to the discoloration of artificial composite resin teeth. Further studies are therefore needed to clarify the effects of this array of clinical factors in the oral cavity.

CONCLUSIONS

Within the limitations of the present study, the results showed that composite resin artificial teeth showed better discoloration resistance with silanized filler, while polishing did not influence the discoloration of composite resin artificial teeth.

ACKNOWLEDGEMENTS

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