치의과학박사 학위논문

Color stability and wear behavior of interim restorative materials for digital dentistry

디지털 치과용 임시 수복 재료의
마모 저항성 및 색 안정성에 관한 연구

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서울대학교 대학원

치의과학과 치과보철학 전공

GERELMAA MYAGMAR

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지도교수 한중석

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게렐마의 박사 학위논문을 인준함

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Abstract

Color stability and wear behavior of interim restorative materials for digital dentistry

GERELMAA MYAGMAR

Program in Prosthodontics, Department of Dental Science, The Graduate School, Seoul National University

(Directed by Professor Jung-Suk Han, DDS, MS, PhD)

Objectives. Evidence regarding the optical and mechanical properties of digitally fabricated interim restorations is lacking. In this study, the following two experiments were conducted. Experiment I investigated the wear resistance and surface roughness of three interim resin materials, which were subjected to chewing simulation. Experiment II evaluated whether different mouth rinses could affect the color and surface roughness of milled and printed interim restorations after simulated oral rinsing.

Materials and Methods. For Experiment I, three interim materials were evaluated: (1) three-dimensional (3D) printed (digital light processing type), (2) computer-aided design/computer-aided manufacturing (CAD/CAM) milled, and (3) conventional polymethyl methacrylate interim resin materials. A total of 48 substrate specimens were prepared. The specimens were divided into two subgroups and subjected to 30,000 or 60,000 cycles of chewing simulation (n=8). The wear volume loss and surface roughness of the materials were compared. Statistical analysis was performed using one-way analysis of variance and Tukey's post hoc test (α=.05). For Experiment II, disc-shaped specimens (N=180; 15×2 mm) were fabricated using conventional, milled, and printed resin materials. All resin specimens were divided into three different groups according to the rinsing material: distilled water, whitening mouth rinse, and conventional mouth rinse. The specimens were further allocated into short- and long-term groups, and oral rinsing simulation was performed (n=10). The color differences (CIEDE2000, ΔE_{00}) between the baseline and each time point were determined by using a spectrophotometer. The surface roughness of the tested specimens was measured using a confocal laser scanning microscope. Kruskal–Wallis tests with nonparametric pairwise comparisons were used to analyze the data (α=.05).

Results. According to the results of Experiment I, the mean±standard deviation value (SD) volume losses (mm^3) against the metal antagonist after 60,000 cycles were 0.10±0.01 for the 3D printed resin, 0.21±0.02 for the milled resin, and 0.44±0.01 for the conventional resin, respectively. Statistically significant differences among volume losses were found in the order of 3D printed, milled, and conventional interim materials (p<.001). After 60,000 cycles of simulated chewing, the mean surface roughness (Ra; µm) values for 3D printed, milled and conventional materials were 0.59±0.06, 1.27±0.49, and 1.64±0.44, respectively. A significant difference was found in the Ra value between 3D printed and conventional materials (p=.01). Experiment II showed the following results. On simulation of a 6-month use of the mouth rinse, the color change in the milled resin was not different than that in the conventional resin (all p>.334), but the printed resin showed a significantly greater color change (all p<.007). The greatest color change was observed when a conventional mouth rinse was used. However, all color changes were below the perceptible threshold. When daily rinsing for 14 years was simulated, all resin groups showed a perceptible color change when conventional mouth rinse was used, and the printed resin showed the greatest ΔE_{00} of 2.24±0.2. In both short-term and long-term simulations, the printed resin rinsed with the conventional mouth rinse showed significantly greater roughness than that rinsed with distilled water (all p<.009). **Conclusion.** The interim restorative materials for additive and subtractive manufacturing digital technologies exhibited less wear volume loss than the conventional interim resin. The 3D printed interim restorative material showed a smoother surface than the conventional interim material after simulated chewing. Printed resin showed lower color stability than the conventional resin, and the color change in it was greatest on using the conventional mouth rinse. However, after simulating 6 months of daily mouth rinse use, all the tested resin materials exhibited imperceptible color change and clinically acceptable surface roughness. Additively and subtractively manufactured interim resin materials can be selected for general interim restoration periods.

Keywords: Color stability; Computer-aided design; Dental restoration wear; Surface properties; Temporary dental restoration; Three-dimensional printing

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Contents

I. Introduction

Interim dental restorations play an important role in maintaining the patient's masticatory function and oral condition and in simulating outcomes of the definitive prosthesis.[1] Interim fixed dental prostheses are often used for an extended period in cases of implant-supported restorations and extensive prosthetic rehabilitation.[2, 3] In particular, when restoring a large edentulous area, it may be necessary to retain the interim restoration for a longer duration in the oral cavity.[4] This would enable the evaluation of the newly established intermaxillary relationship and its compatibility with the surrounding neuromuscular system before placing the definitive prosthesis.[1] Hence, the interim restorative materials should have adequate functionality, biocompatibility, surface characteristics, and good esthetic properties.

Resin-based materials, such as polymethyl methacrylate, are traditionally used interim restorative materials with advantages such as low cost, ease of oral adjustment, repairability, and esthetics.[5] For long-term provisionalization, the interim restoration should have high wear resistance and mechanical strength, biocompatibility, and an esthetic appearance.[6, 7] The existing resin materials used to fabricate interim restorations can be classified according to their chemical composition: autopolymerizing polymethyl methacrylate (PMMA), polyvinyl methacrylate, polyethylene methacrylate, bis-acryl, urethane methacrylate, and microfilled resin.[1] Owing to the complex environment of the oral cavity, several factors should be considered when selecting an appropriate material for a provisional restoration, including provisional timing, longevity, and ease of fabrication.[8] Although conventional self-polymerizing PMMA is often selected in routine clinical practice, it exhibits a high rate of shrinkage and heat generation during polymerization[6] and poor mechanical characteristics.[9]

With the development of computer-aided design and computer-aided manufacturing (CAD/CAM) technologies, interim prostheses can be fabricated using additive 3-dimensional (3D) printing and subtractive milling methods.[10] These digital dental technologies have the advantage of lower labor costs, can simplify fabrication processes, improve accuracy, reduce processing time [11], and fewer human errors as compared with manual fabrication. Subtractive interim restoration materials include prefabricated acrylic blocks, which are polymerized under optimal high temperatures and pressures.[12] The resin blocks for milling have been reported to possess a constant mechanical quality.[13] Although the milling technique has been used for a longer time in dentistry and is more familiar to dentists and dental technicians, subtractive milling has several disadvantages over 3D printing, such as wastage of milling burs and restorative materials and difficulty in producing complex shapes.[14] On the contrary, the additive manufacturing method can be used to precisely fabricate the desired complex shape by projecting light beams while minimizing unnecessary material waste, but there are doubts regarding the optical and mechanical properties of the 3-dimensionally (3D) printed materials that undergo chairside polymerization.[15, 16]

The color and surface stability of interim restorations are essential to maintain long-term esthetics and prevent plaque accumulation in the oral environment.[16, 17] The color and surface of tooth-colored restorations are reportedly affected by many extrinsic factors, such as acid, colorants, alcohol in food and beverage, and cigarette smoking.[18-27] Oral hygiene procedures, such as toothbrushing and oral rinsing, can also cause discoloration and surface deterioration of restorations due to periodical application throughout life.[28-33] In particular, since interim restorations are generally maintained during the healing process after surgical procedures such as tooth extraction or implant placement, they are often used in combination with mouth rinses. Thus, interim restorations should be stable and not easily be discolored or damaged due to the use of mouth rinses.

The color stability, and surface roughness of newly developed interim materials induced by various beverages and foods has been extensively studied.[20, 25, 27] However, there is a lack of evidence regarding the color stability and surface properties of interim resin materials for digital dentistry after daily use simulation of oral rinsing. Furthermore, the mechanical and surface characteristics of the novel interim materials after long-term use are still unclear.[34] Therefore, the first experiment (Experiment I) of this study aimed to compare the wear resistance and surface roughness of 3D printed, CAD/CAM milled, and conventionally fabricated interim restorative materials. In addition, the second experiment (Experiment II) aimed to investigate the effects of oral rinsing solutions on the color stability and surface roughness of additively and subtractively manufactured interim resin materials. The null hypothesis of Experiment I was that there is no difference in the wear amount and surface roughness between the tested interim materials after simulated chewing. The null hypothesis of Experiment II was that the color and surface roughness of the interim resin materials remain unaffected after rinsing over various periods.

II. Materials and Methods

II-1. Experiment I. Wear of 3D printed and CAD/CAM milled interim resin materials after chewing simulation

II-1.1. Specimens preparation

Three different types of interim resin materials were evaluated in this study: a 3D printed resin (NextDent C&B; NextDent BV), a PMMA-based CAD/CAM milled material (PMMA Disk; Yamahachi Dental Mfg, Co), and a conventional self-cured PMMA resin (Jet Tooth Shade; Lang Dental Manufacturing Co, Inc) (Table 1.1). To fabricate the 3D printed and milled specimens (n=8), rectangular parallelepipeds (15×10×10 mm; width × length × height) were designed using the Fusion 360 CAD software (Autodesk, Mill Valley, CA, USA), and the design files were exported in the standard tessellation language (STL) format.

The specimens of the printed resin were manufactured using a DLP- type 3D printer (NextDent 5100, 3D Systems, Rock Hill, SC, USA) with 405 nm ultraviolet light. The specimens were printed at a build angle of 0°[35], wherein the side to be tested was made parallel to the build platform. The thickness of each printing layer was set to 100 µm, and the support structure was attached to the bottom side of the specimens. It has been shown that accuracy of the printed specimens with different thicknesses have the highest accuracy at 0° orientation (degree), and significantly low error. The peak stress is also reportedly high in prints with a 100 µm layer thickness[15], After the 3D printing process, the monomer remaining on the surface of the specimen was washed for 20 min with 90% isopropyl alcohol using a cleaning system (FH-WA-01, Formlabs, USA). Then, the specimens were subjected to a post-curing process for 30 min using a post-curing machine (3D Systems LC-3DPrint Box; NextDent, Soesterberg, Netherlands). After post-curing, the support structure used for printing was removed.

To fabricate the milled specimens, CAM software program (HyperDENT® version 8.1; FOLLOW-ME! Technology GmbH, Munich, Germany) was utilized, and the PMMA resin disc (Yamahachi PMMA Disk, Yamahachi Dental Manufacturing) was machined. A 5-axis milling machine (ARUM 5X-400; Doowon ID Co., Ltd., Daejeon, Korea) was used for the milled specimens.

For the conventional interim resin specimens, a silicone mold was fabricated and self-cured resin (Jet™, Lang Dental Manufacturing) was poured into it. The mixing a powder-to-liquid ratio was 100:52, according to the manufacturer's recommendations. Subsequently, the mixture in the mold was covered with a glass slide, and cured in a pot containing water at a pressure of 0.21 MPa. Before the wear test, all specimens were dried at 37°C for 1 day. After that, the produced specimens were polished on both sides using 600- and 1200-grit silicon carbide paper on a rotary machine (Buehler Metaserv 2000, Buehler, Germany) with water cooling. Sixteen specimens were fabricated for each interim restorative material.

The metal antagonist was designed using CAD software (Autodesk Inventor 3D CAD, Autodesk, Mill Valley, CA, USA) with a hemisphere radius of 1.5 mm[36], because the radius of individual human cusps range between 0.6 mm and 2.4 mm.[37, 38] Then, the designed metal antagonist was additively manufactured with Cobalt-Chrome powder (EOS Cobalt Chrome SP2; EOS GmbH, Krailling, Germany) using a metal 3D printer (EOSINT M270, EOS GmbH, Krailling, Germany). The metal abrader surface to be tested was polished in one direction with a 1200-grit brown rubber point (Brownie® Polisher PC2; SHOFU, Kyoto, Japan) with water cooling at approximately 10,000 rpm.

II-1.2. Chewing simulation

A chewing simulator (CS-4.8, SD Mechatronik, Feldkirchen-Westerham, Germany) was used to conduct the wear test. The metal cast antagonist specimens were placed in the upper holders, and the resin substrate specimens were randomly placed in the lower specimen holders (Figure 1.1). The chambers in the machine simulated the simultaneous vertical and horizontal movements of the thermodynamic conditions. The chewing cycle of the abrader was set to have a 5 mm vertical descending movement and a 2 mm horizontal movement, followed by an ascending movement with the recovery of its original position. A vertical load of 5 kg was applied during sliding motion, which is comparable to 49 N of chewing force. During the wear simulation, the specimens were subjected to thermocycling in distilled water with heat circulation at 5–55°C using a heating/cooling system with a programmable logic controller. The specimens of each material were divided into two subgroups, and abraded for 30,000 and 60,000 cycles, which was considered to be equivalent to approximately 1.5 and 3 months of chewing, respectively (n=8).[39]

II-1.3. Wear volume loss

The abraded specimens were steam-cleaned and air-dried to remove any dirt prior to digital wear analysis. The specimens were scanned using a multiline blue LED light scanner (D1000, 3Shape Copenhagen, Denmark) with an accuracy of 5/8 μm (ISO

12836). The acquired images were imported into the universal reverse engineering software (Geomagic Control X 2018 version 1.2, 3D Systems, Rock Hill, SC, USA). The wear losses (mm^3) of the resin specimens were calculated as the difference in the volume before and after wear testing using the software.

II-1.4. Surface roughness

The impact of chewing simulation on the surface roughness of the materials was evaluated before and after simulated chewing. Four representative specimens were randomly chosen from each group, and a confocal laser scanning microscope (LSM 800 MAT, Zeiss, Jena, Germany) was used to analyze their tested surfaces. Laser excitation at 405 nm with the C Epiplan-APOCHROMAT 209/0.7 (Zeiss, Jena, Germany) was used to obtain images. For each representative specimen, three different sites were pictured. The surface roughness of the worn area was measured using the arithmetic mean deviation of the surface roughness (Ra). Overall, 12 Ra values were collected for each group. All assessments were performed according to the ISO 4287 standards.

II-1.5. Scanning electron microscopy

To evaluate the surface morphology of the specimen after chewing simulation, a representative specimen was selected for each group. A thin coating with platinum was applied to the worn surface using a sputter coater (Quorum Q150T-S, Quorum Technologies, West Sussex, UK). The wear patterns on the surface of the specimen were examined using a scanning electron microscope (SEM) (Apreo S;

ThermoFisher Scientific, Waltham MA, USA) at magnifications of ×200 and ×1000 with 10 keV.

II-1.6. Statistical analysis

The mean and standard deviation (SD) of the test parameters were calculated using statistical software (IBM SPSS version 25.0; IBM Corp., Chicago, IL, USA). Tests for normality and equality of variances were applied. The statistical significance of the mean difference of each parameter was evaluated at a significance level of 5% using one-way analysis of variance (ANOVA) and Tukey's post-hoc test for three different resins. The paired *t*-test was used to compare the mean volume loss of each resin between the two different thermocycles.

II-2. Experiment II. Color and surface stability of additively and subtractively manufactured interim restorative materials against mouth rinses

II-2.1. Specimen preparation

Three commercially available interim restorative acrylic resin materials were selected for this study (Table 1.1): a conventional resin (Jet Tooth Shade, A3 shade; Lang Dental Manufacturing Co, Inc), milled resin (PMMA Disk, A3 shade; Yamahachi Dental Mfg, Co), and printed resin (NextDent C&B, A3.5 shade; NextDent BV). The specimens were designed as a disc (15 mm in diameter and 2 mm in thickness). To fabricate specimens for the conventional resin group, a silicone mold was prepared and poured with the conventional resin mixed according to the manufacturer's recommendations. For the digitally fabricated specimens, the disc-shaped specimen was designed by using CAD software (Fusion 360; Autodesk Inc), and the Standard Tessellation Language file of the dataset was exported and used for specimen fabrication. The milled resin specimens were fabricated with a CAD-CAM milling acrylic resin material (PMMA Disk, A3 shade; Yamahachi Dental Mfg, Co) by using a 5-axis precision milling machine (5X-400; Arum Dentistry Co, Ltd). The printed resin specimens were additively manufactured with NextDent C&B material (A3.5 shade; NextDent BV) and a digital light-processing-type 3D printer (NextDent 5100; NextDent BV). The specimens were printed at a 50 µm layer thickness and 90° build orientation, cleaned with isopropyl alcohol, and post-cured in a UV light box (LC-3DPrint Box; NextDent BV). A total of 180 specimens were fabricated, with 60 specimens of each resin material. Both sides of all the specimens were polished with a series of silicon carbide of 600 and 1200 grit on a rotary machine (Metasery 2000; Buehler) with water cooling. Subsequently, each specimen was ultrasonically cleaned in distilled water for 5 min, and the cleaned specimens were stored at 37 °C for 24 hours.

II-2.2. Mouth rinsing simulation

For the oral rinsing simulation, a whitening mouth rinse (Listerine Healthy White; Johnson & Johnson) and a conventional mouth rinse (Listerine Cool Mint; Johnson & Johnson) from the same manufacturer were selected (Table 1.1). Distilled water was used as control. The interim resin specimens were divided into three groups and assigned to each liquid, and the specimens were further divided into two subgroups for short-term and long-term oral rinsing simulations (Fig. 2.1; n=10). The oral rinsing procedure was simulated in a shaking incubator (DS-210SF; Daewon Science

Inc) at a constant speed of 39 rpm and temperature of 37 °C. For the short-term group, daily use of the mouth rinse for 1 month, 3 months, and 6 months, which are close to the usual period of interim restoration use, were simulated. More extensive use periods of 2, 6, and 14 years, were simulated in the long-term group to study the properties of the interim materials. It has been reported that continuous exposure to a mouth rinse for 1 hour is equivalent to daily use of mouth rinse for 1 month (1 minute twice daily).[31, 40] Thus, the oral rinsing simulation was conducted for 1, 3, and 6 hours for the short-term usage simulation and 24, 72, and 168 hours for the long-term simulation.

II-2.3. Color measurements

A spectrophotometer (CM-700d; Konica Minolta, Inc) was used to analyze color differences. Before and after each period of oral rinsing simulation, the International Commission on Illumination (CIE) *L**, *a**, and *b** color coordinates of each specimen were measured. Three readings were obtained for each specimen, and their mean values were documented for analysis. The color measurements at each time point were compared with the color coordinates at baseline, and the color differences (ΔE_{00}) were calculated using the following CIEDE2000 color difference formula.[41]

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)},$$

where *L'*, *C'*, and *H'* indicate lightness, chroma, and hue, respectively; and R_T and *S* refer to the rotation and weighting functions, respectively. The correction terms for the experimental conditions, k_L, k_C, and k_H, were set to 1.[41]

II-2.4. Surface roughness

After the mouth rinsing simulation and color assessment, six representative samples were randomly chosen from each group, and the roughness of the material surface was examined by using a confocal laser scanning microscope (LSM 800 MAT; Zeiss Co Ltd) according to the ISO 4287 standards.

II-2.5. Scanning electron microscopy

Another representative specimen from each group was selected and evaluated by using a SEM (Apreo S; Thermo Fisher Scientific Solutions LLC). Specimens were sputter-coated with platinum (Q150T-S; Quorum Technologies Inc.) and examined at ×5000 with 10 keV.

II-2.6. Statistical analysis

Data analysis was performed using a statistical software program (IBM SPSS v25.0, IBM Corp., Chicago, IL, USA). The Shapiro–Wilk test was used to identify the normality of the data. As the data did not show a normal distribution, the Kruskal-Wallis test and pairwise Mann-Whitney U tests were used to analyze them. The significance level was set to 0.05, and the *p*-values were adjusted using Bonferroni correction for multiple tests.

III. Results

III-1. Experiment I. Wear of 3D printed and CAD/CAM milled interim resin materials after chewing simulation

The wear volume loss of the substrate specimens after two different chewing cycles are presented in Figure 1.2. The mean±SD volume loss (mm^3) after 30,000 and 60,000 cycles were 0.08±0.09 and 0.10±0.01 for the 3D printed resin, 0.06±0.01 and 0.21±0.02 for the milled resin, and 0.11±0.01 and 0.44±0.01 for the conventional resin, respectively. A significant difference in the wear volume loss was shown among the interim materials (p<.001). The wear volume loss of the 3D printed resin was lower than that of the milled and conventional resins for both cycles (p<.001). A significant difference between the loss amounts after 30,000 and 60,000 cycles was found in each resin group.

The mean±SD Ra values (µm) before (baseline) and after the wear tests at 30,000 and 60,000 cycles were 0.48±0.06 and 0.59±0.06 for the 3D printed resin, 0.88±0.05 and 1.27±0.49 for the milled resin, and 0.92±0.09 and 1.64±0.44 for the conventional resin. Statistically significant differences were found in Ra of the different interim resin materials and two different cycles (Table 1.2).

SEM images of the abraded surfaces of the specimens after wear tests are shown in Figure 1.3 (original magnification: ×200, ×1000). All three resin types exhibited compressed and crushed features. Crack line were observed when the metal abrader was applied for 60,000 cycles (see ×1000 images).

III-2. Experiment II. Color and surface stability of additively and subtractively manufactured interim restorative materials against mouth rinses

Figures 2.2 and 2.3 shows the color difference values comparing the color coordinates after the oral rinsing simulation with those at the baseline. When comparing the resin groups after the short-term simulation, the printed resin groups showed significantly greater color changes than the milled resin groups following use of the whitening and conventional mouth rinses (all $p<.001$). There was no significant difference between the milled and conventional resins in all cases when short-term simulations were performed (all $p>.094$). The median (interquartile range (IQR)) values of color change when the printed resin was rinsed with the conventional mouth rinse were 0.80 (0.2), 0.86 (0.13), and 0.82 (0.13) after 1, 3, and 6 hours of rinsing, respectively. These values were significantly higher than those obtained when the resin was rinsed with distilled water (all $p<.005$).

Similarly, following long-term simulation, the printed resin groups showed significantly greater color changes than the conventional resin group for all types of mouthwash (all $p<.001$). The printed resin showed a significantly greater color change than the conventional resin, even when rinsed only with distilled water (all $p<.007$). However, when the milled resin was rinsed with distilled water, the change was not significantly different than that in the conventional resin (all $p>.025$). The color change induced by the whitening mouthwash in the printed resin was not significantly different from that induced by distilled water (all $p>.057$). However, the printed resin groups showed significantly greater color changes when washed with the conventional mouth rinse (all $p<.015$), and the median (IQR) value of the largest color change was 2.24 (0.2) after 168 hours of rinsing. At this time, the printed resin had significantly lower L^* and a^* values than when it was washed with distilled water (all $p<.005$). This indicates that the specimens became darker and greener (Table 2.1).

Table 2.2 lists the surface roughness values after the short- and long-term simulations. When the printed resin groups were rinsed with conventional mouth rinse, significantly greater surface roughness was observed following both short-term and long-term simulations than when washed with distilled water (all $p<.009$). For the milled resin, there was no significant difference in surface roughness among the mouth rinses and distilled water after the short-term simulation ($p=.073$). However, after the long-term simulation of the milled resin groups, the surface roughness on using mouth rinses was significantly greater than when distilled water was used (all $p<.004$). Figure 2.4 shows scanning electron microscopy images at ×5000 after the short-term rinse simulation. No noticeable differences were observed in the surface morphologies of the groups.

IV. Discussion

IV-1. Experiment I. Wear of 3D printed and CAD/CAM milled interim resin materials after chewing simulation

This in vitro study investigated the wear behavior and surface roughness of three different interim restorative materials at two different time intervals, and the null hypothesis of this study was rejected. The results of the study showed that there was a significant difference in wear resistance and surface roughness among the tested interim materials. The wear volume loss of the 3D printed and milled resins was less than that of the conventional resin. The 3D printed group showed a smoother surface than that of the conventional PMMA group after simulated chewing.

In the present study, interim resin materials fabricated using digital dental technologies, including 3D printing and milling, showed significantly less volume of wear than the conventional resin material, after a simulated period of 1.5 and 3 months of clinical chewing. Rayyan et al.[12] have reported that CAD/CAM milled PMMA resin showed a lower percentage of weight loss due to wear than autopolymerizing conventional PMMA interim resin after subjecting the materials to 2 million cycles of a load of 40 N. Stawarczyk et al.[42] have also reported that CAD/CAM milled resin materials exhibit less wear rates than conventional manually polymerized interim resin materials. These results are in accordance with the findings of the current study.[12, 42] CAD/CAM milled resin materials are industrially polymerized under optimum manufactured conditions and thus, considered to exhibit better mechanical properties than conventional resin materials.[12]

Park et al.[35] also compared the wear resistance of printed interim resin and conventional PMMA resin.[35] In the study[35], the printed resin did not show a significantly different amount in wear volume loss compared to the conventional interim resin after 30,000 cycles of chewing simulation. Similar to our study, the previous study investigated the same printed resin material; however, the 3D printer and post-curing machine used in the previous study are different from those used in the current study. These differences in equipment might have resulted in disparate results between the previous study and the present study. Another recent study reported that printed PMMA denture teeth exhibited a statistically lower depth of wear compared to the prefabricated PMMA resin denture teeth after 200,000 cycles of simulated chewing.[43] Based on the results of these previous studies[35, 43], and this present study, printed resin materials are considered to have equivalent or superior wear resistance compared to the conventional PMMA materials.[35, 43]

In this study, printed interim resin showed a significantly lower Ra value than the conventional interim resin before and after simulated chewing. Previous studies have also reported that PMMA resin has a rougher surface than printed resin.[27, 44] Furthermore, all the tested groups in this study showed increased surface roughness after masticatory simulation compared to the baseline. For both digitally and conventionally fabricated interim restorative materials, material wear leads to a rougher surface and promotes more plaque accumulation on the worn surfaces.[45] The rough surface on the interim restoration could induce bacterial adhesion and dental biofilm formation, resulting in adverse effects on periodontal health.[46] In the present study, the mean Ra values of tested materials after 30,000 and 60,000 cycles were higher than the previously reported plaque accumulation threshold of 0.2 µm[45] and a tongue detectable surface roughness threshold of 0.25-0.5 µm.[47] However, since both 3D printed resin and milled resin showed similar or smoother surfaces compared to the conventionally used PMMA resin, it is considered that this printed and milled materials can be used for fabricating interim restorations in clinical practice. The printed resin materials have also been reported to have different surface roughness depending on the type of material[48] and the printing orientation (degree)[49], thus, this should also be considered while selecting the material.

The strength of this study is that it simulated chewing for a period similar to that of actual interim restoration use in clinical practice. Interim restorations are usually used for approximately 1.5 months for a simple crown restoration. However, for multiple units of prosthesis, or if the treatment also includes additional root canal treatment, periodontal surgery, or implant surgery, the interim restorations often need to be used for more than 3 months. Thus, in this study, the changes after 1.5 and 3 months were studied by subjecting the materials to 30,000 and 60,000 cycles of chewing simulation, respectively.[39] To the best of our knowledge, this is the first study to evaluate the wear resistance after 3 months of using interim restorations fabricated with additive manufacturing digital technologies.

In this study, although the setting of the chewing simulator was as similar to the clinical conditions as possible, it still has the limitations of in vitro design. To facilitate the evaluation of the wear volume loss of the material itself, specimens in the shape of a rectangular parallelepiped rather than a crown shape were used in this study. The results may be different for teeth. Furthermore, factors that influence wear include the physical properties of enamel[50, 51], parafunctional habits, eating habits, and the type of antagonist material used.[51-57] In the oral cavity, the wear process is promoted by mechanical, thermal, and chemical stimuli.[58, 59] Therefore, further clinical studies are needed to confirm whether the results of this study are clinically consistent.

IV-2. Experiment II. Color and surface stability of additively and subtractively manufactured interim restorative materials against mouth rinses

This study investigated the influence of mouth rinse use on the color and surface of digitally fabricated interim restorative materials. Based on the findings of the present study, the use of mouth rinses had a significant effect on the color and surface roughness of interim restorative resin materials; thus, the null hypothesis of the present study was rejected.

In this study, all the tested interim resin materials generally showed greater color changes as the rinsing time increased. However, when short-term use of mouthwash was simulated, all color change values (ΔE_{00}) were within the previously reported perceptible threshold of 1.30.[60] This means that the color changes induced by the use of mouth rinses were significantly greater for the printed resin than for the conventional resin, however, these changes were not perceptible following use of less than 6 months. In addition, when long-term use was simulated, 6-year and 14-year use of conventional mouthwash showed perceptible color changes in all three interim resin materials. However, except for the printed resin group that underwent simulation of 14-year-use of conventional mouth rinse, all color change values were within the clinically acceptable thresholds of 2.25.[60]

This study showed that the 3D printed resin had lower color stability than milled resin and conventional resin, when rinsed with distilled water as well as mouth rinses.

This is consistent with the results of previous studies, according to which, the color stability of additively processed resin materials was lower than that of other resin materials.[16, 25] Scotti et al. reported that the color stability of printed resin was inferior to that of nanoparticle composite resin and bis-acrylic interim resin.[16] Shin et al. studied the color stability against curry and beverages and reported that additively manufactured resins showed lower color stability than various subtractively manufactured resins.[25] In this study, the milled groups generally showed relatively high color stability, similar to that of conventional resin groups. Bitencourt et al. have also reported that milled resin showed high color stability against various acidic/stainable beverages compared to other resin materials.[20] Various factors, such as degree of conversion, type of initiator, particle distribution, and water absorption have been reported to affect the color stability of resin-based restorative materials.[16, 22] Milled resin materials have an excellent degree of conversion because they are polymerized at the manufacturer's facilities. Thus, the differences in color stability may have occurred due to the relatively low degree of conversion due to the chairside curing of printed resin materials.[24] In addition, since the DLP-type 3D printer used in this study adopts the layer-by-layer manufacturing method, the printed resin materials have microstructures on their surface. The presence of these microstructures may make those materials susceptible to staining.

In the present study, mouth rinses changed the color of the interim restorative materials to a greater extent than distilled water, which is consistent with the results of previous studies reporting the effect of mouth rinses on the color of various restorative materials.[28, 31-33] The conventional mouth rinse caused a greater color change than the whitening mouth rinse, which may have resulted from the difference in the ingredients of the two rinsing solutions. The conventional mouth rinse had a blue-green color, unlike the colorless whitening mouthwash. This coloring component may have affected the color tone of the interim resin material. In this study, the printed resin showed an increase in the green component (-*a**) after rinsing with the conventional mouthwash. The alcohol content of the mouthwash may have also induced discoloration by softening the resin matrix.[32, 33] The conventional mouthwash contains 21.6% ethanol, which has a higher alcohol content than 14.58% of the whitening mouthwash. In addition, the conventional mouthwash with a pH of 3.93 is more acidic than the whitening mouthwash having a pH of 6.56.[31] This high acidity may induce a color change.[20, 21] Previous studies have reported that applying a light-polymerizing surface protective coating material increases the color stability of the interim restorative resins.[27, 61] To prevent unintentional discoloration caused by these components of a mouthwash, the application of surface coating materials on the cameo surface of interim restorations may be considered.

The results of the present study revealed that the surface roughness of all materials after the short-term simulation was near or slightly higher than the plaque retention threshold of 0.2 μm. As the printed resin showed a significantly larger color change when rinsed with the conventional mouthwash than when rinsed with distilled water, the surface roughness was also significantly greater. This may also be due to the high alcohol content and strong acidity of the conventional mouthwash.[31] Previous studies have reported that the surface of resin-based materials became rougher when immersed in a solution with high alcohol content or high acidity.[19, 26] However, in this study, the median value of the surface roughness of all groups did not exceed 0.6 μm after the long-term as well as the short-term simulation, which was much lower than the clinically acceptable threshold of 10 μm.[62] Moreover, there was no noticeable difference between the surface of the distilled water and mouth rinse groups when observed at a magnification of 5000×.

The strength of this study is that oral rinsing was simulated on various interim restorative materials for different time periods. Interim restorations are used in different stages of dental implant and prosthetic rehabilitation, usually for periods of less than 6 months. Therefore, in this study, apart from long-term simulation, the use of mouthwash for 1, 3, and 6 months, similar to the actual interim restoration period, was also simulated. Despite these strengths, this study had several limitations owing to its in vitro design. Changes in the color and surface roughness of the restorative material may vary depending on the masticatory condition, food preference, and the presence of saliva.[31] In addition, since the use of mouthwash is intermittent in an actual clinical situation and affects only the cameo surface of the interim restorations, the effect may have been aggravated in this experiment, in which continuous rinsing was applied to both sides of the specimen.[18] Therefore, further clinical researches are required to confirm that the results of the present study are consistent with those in a clinical situation.

V. Conclusions

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Printed resin and CAD/CAM milled resin showed greater wear resistance than conventional interim resin after simulation of clinical chewing period equivalent to a duration of 1.5 and 3 months.
2. Worn printed resin specimens showed a smoother surface than the conventional interim resin after chewing simulation.
3. The printed resin groups generally showed a greater color change than the milled and conventional resin groups.
4. The conventional mouth rinse influenced the printed resin the most, making it darker and greener with increased surface roughness.
5. After simulating 6 months of oral rinsing, all color changes were within the perceptible threshold, and the surface roughness values were within the clinically acceptable range. Thus, interim restorations fabricated using additive and subtractive manufacturing techniques may be used in conjunction with mouth rinses during the interim restorative period.

The additively and subtractively manufactured interim resin materials would be clinically acceptable as an interim restorative material for general interim restoration periods.

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Tables

Table 1.1. Type of resin materials tested in this study

Type	Product	Manufacturer	Component*	Lot
Printed resin	NextDent C&B	NextDent BV		XK 133N02
Milled resin	PMMA Disk	Yamahachi Dental Mfg, Co		PA03
Conventional resin	Jet Tooth Shade	Lang Dental Manufacturing Co, Inc		(Powder) 1430-19HC (Liquid) 1404-20AC
Abrader	EOS CobaltChrome SP2	EOS GmbH		H131501
Whitening mouth rinse	Listerine Healthy White	Johnson & Johnson	Active ingredients: Sodium fluoride 0.02% (0.01% w/v fluoride ion). Inactive ingredients: Water, Alcohol (14.58%), Sorbitol solution, Hydrogen peroxide, PEG-40 hydrogenated castor oil, Flavors, Poloxamer 407, Thymol, Menthol, Citric acid, Sodium Benzoate, Eucalyptol, Sodium saccharin, Phosphoric acid, Disodium phosphate, Sucralose	6122463
Conventional mouth rinse	Listerine Cool Mint	Johnson & Johnson	Active ingredients: Eucalyptol 0.092%, Menthol 0.042%, Methyl salicylate 0.060%, Thymol 0.064%. Inactive ingredients: Water, Alcohol (21.6%), Sorbitol solution, Flavoring, Poloxamer 407, Benzoic acid, Sodium saccharin, Sodium benzoate, FD&C Green No. 3	6107624

*According to the manufacturer’s information

Table 1.2. Mean ±standard deviation of surface roughness (Ra; µm) values for tested interim restorative materials

	Baseline	**30,000 cycles**	**60,000 cycles**
Printed resin	0.13 ± 0.01^{Aa}	0.48 ± 0.07^{Ab}	0.59 ± 0.06^{Ac}
Milled resin	0.19 ± 0.03^{Ba}	0.88 ± 0.05^{Bb}	1.27 ± 0.49^{ABb}
Conventional resin	0.26 ± 0.02^{Ca}	0.92 ± 0.10^{Bb}	1.64 ± 0.44^{Bc}

Same superscript letters indicate no statistically significant differences. Uppercase letters for each column, lowercase letters for each row.

Table 2.1. Median and Interquartile range (IQR) of CIE *L**, *a**, *b** values of tested interim resin materials after oral rinsing simulation

Rinsing time	Color parameters	Groups	Distilled water	Whitening mouth rinse	Conventional mouth rinse
Short-term (6 hours)	*L**	Conventional resin	72.72 (0.50)^{Ca}	74.57 (0.36)^{Cb}	72.43 (0.35)^{Ba}
		Milled resin	65.13 (0.20)^{Ab}	65.05 (0.20)^{Ab}	63.43 (0.45)^{Aa}
		Printed resin	72.21 (0.24)^{Ba}	72.57 (0.38)^{Bb}	72.18 (0.27)^{Ba}
	*a**	Conventional resin	1.21 (0.12)^{Cb}	1.58 (0.21)^{Cc}	0.75 (0.12)^{Ca}
		Milled resin	-0.68 (0.05)^{Ac}	-0.84 (0.11)^{Ab}	-1.44 (0.10)^{Aa}
		Printed resin	0.36 (0.14)^{Ba}	0.52 (0.21)^{Bb}	0.50 (0.06)^{Bb}
	*b**	Conventional resin	12.36 (0.20)^{Bab}	12.55 (0.18)^{Ab}	12.34 (0.40)^{Ba}
		Milled resin	11.79 (0.49)^{Ab}	12.12 (0.22)^{Ab}	10.55 (0.73)^{Aa}
		Printed resin	19.44 (0.56)^{Ca}	19.26 (0.49)^{Ba}	19.31 (0.87)^{Ca}
Long-term (168 hours)	*L**	Conventional resin	72.36 (0.37)^{Bb}	74.22 (0.33)^{Bc}	71.24 (0.30)^{Ba}
		Milled resin	64.86 (0.48)^{Ac}	64.14 (0.21)^{Ab}	63.54 (0.22)^{Aa}
		Printed resin	73.07 (1.76)^{Bb}	74.20 (0.30)^{Bb}	70.70 (0.48)^{Ba}
	*a**	Conventional resin	1.18 (0.16)^{Ca}	1.74 (0.09)^{Cb}	0.95 (0.13)^{Ca}
		Milled resin	-1.08 (0.10)^{Aa}	-0.68 (0.07)^{Ac}	-0.87 (0.21)^{Ab}
		Printed resin	0.66 (0.11)^{Bb}	0.84 (0.29)^{Bb}	-0.50 (0.16)^{Ba}
	*b**	Conventional resin	12.93 (0.48)^{Aa}	12.89 (0.27)^{Ba}	13.20 (0.74)^{Aa}
		Milled resin	12.51 (0.67)^{Ab}	11.37 (0.42)^{Aa}	12.26 (0.83)^{Ab}
		Printed resin	18.78 (2.41)^{Bb}	17.06 (0.50)^{Ca}	19.39 (0.55)^{Bb}

Bonferroni correction for multiple tests: A<B<C, a<b<c. Uppercase letters for columns, lowercase letters for rows

Table 2.2. Median and interquartile range (IQR) of surface roughness (Ra; µm) values for tested interim restorative materials after oral rinsing simulation

Rinsing	Groups	Distilled water	Whitening mouth rinse	Conventional mouth rinse
Short-term (6 hours)	**Conventional resin**	0.34 (0.04)^{Bb}	0.24 (0.04)^{Ba}	0.23 (0.10)^{Aa}
	Milled resin	0.17 (0.02)^{Aa}	0.18 (0.04)^{Aa}	0.20 (0.02)^{Aa}
	Printed resin	0.19 (0.07)^{Aa}	0.24 (0.06)^{Bab}	0.25 (0.04)^{Ab}
Long-term (168 hours)	**Conventional resin**	0.37 (0.15)^{ABa}	0.53 (0.02)^{Bb}	0.48 (0.08)^{ABab}
	Milled resin	0.27 (0.08)^{Aa}	0.44 (0.06)^{Ab}	0.43 (0.06)^{Ab}
	Printed resin	0.43 (0.05)^{Ba}	0.49 (0.05)^{ABab}	0.59 (0.04)^{Bb}

Bonferroni correction for multiple tests: A<B<C, a<b<c. Uppercase letters for columns, lowercase letters for rows

Figures

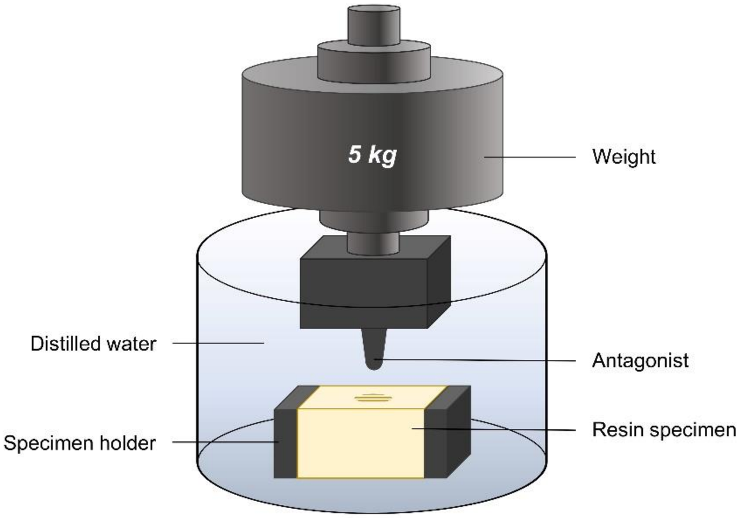

Figure 1.1. Schematic drawing of chewing simulation. Metal cast antagonist specimens were embedded in the upper holders. Resin substrate specimens were randomly embedded in the lower specimen holders.

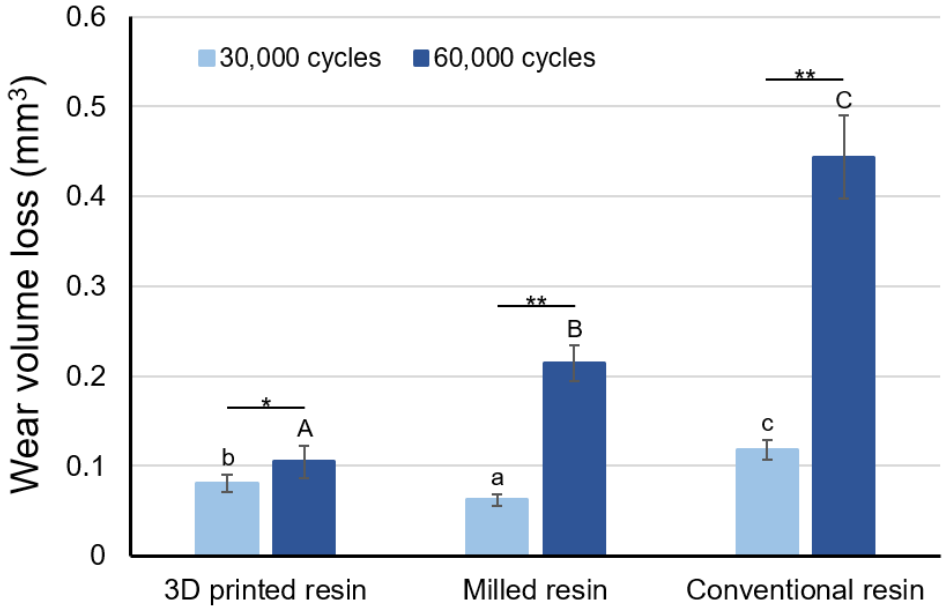

Figure 1.2. Wear volume loss (mean±standard deviation) after 30,000 and 60,000 cycles of chewing simulation. Same letters indicate no statistically significant differences. Lowers letters indicated for 30,000 cycles, uppercase letters for 60,000 cycles. $^{*}p < .05$, $^{**}p < .001$.

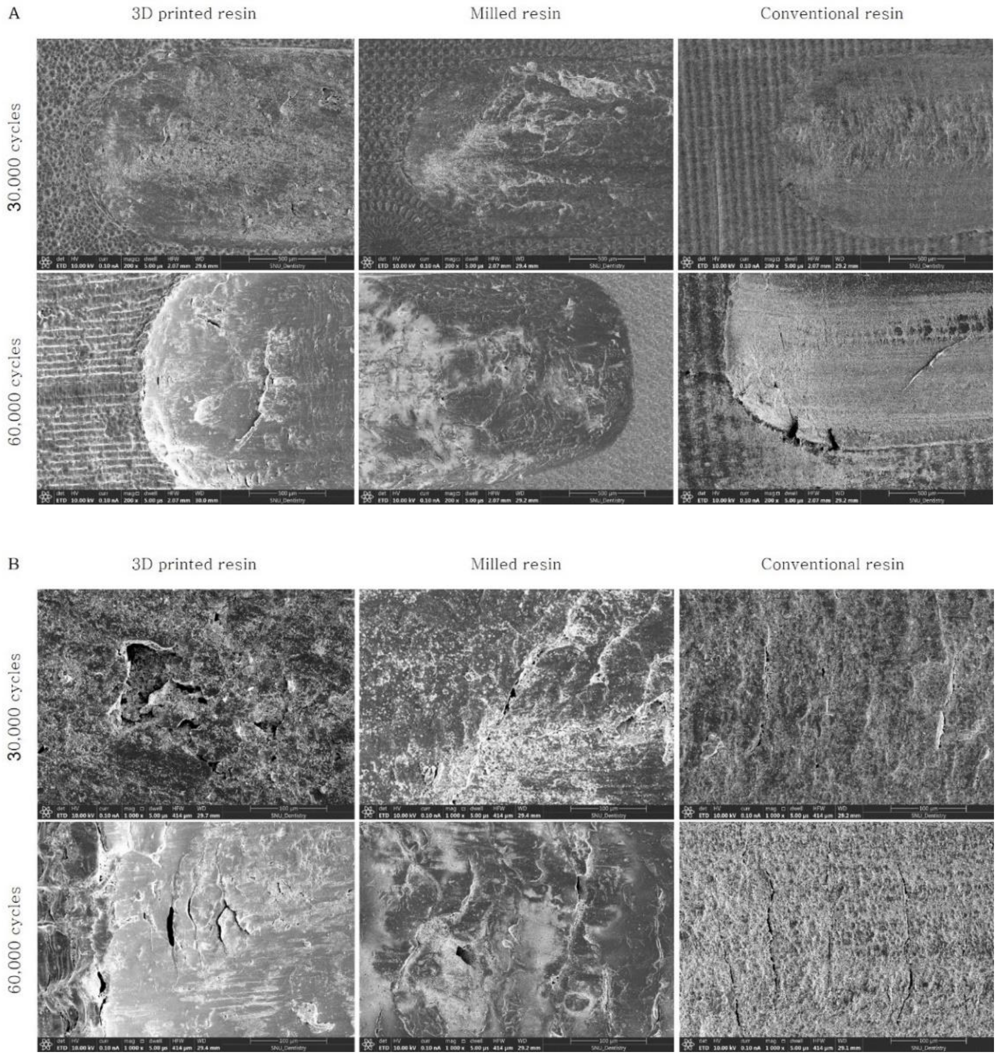

Figure 1.3. Scanning electron microscope images of the worn resin surfaces after 30,000 and 60,000 cycles of chewing simulation. Crack lines are shown on the surfaces of specimens that simulated chewing for 60, 000 cycles. A) Original magnification ×200, B) Original magnification ×1000.

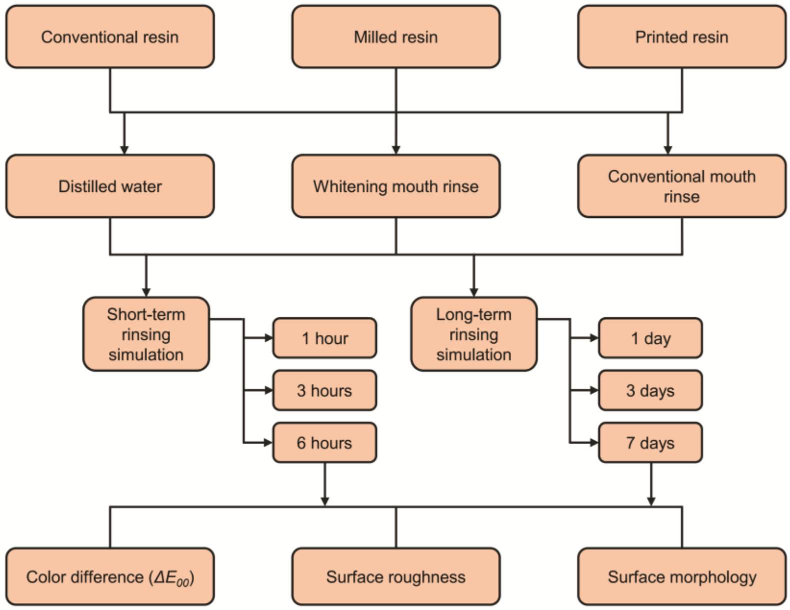

Figure 2.1. Experimental workflow of this study

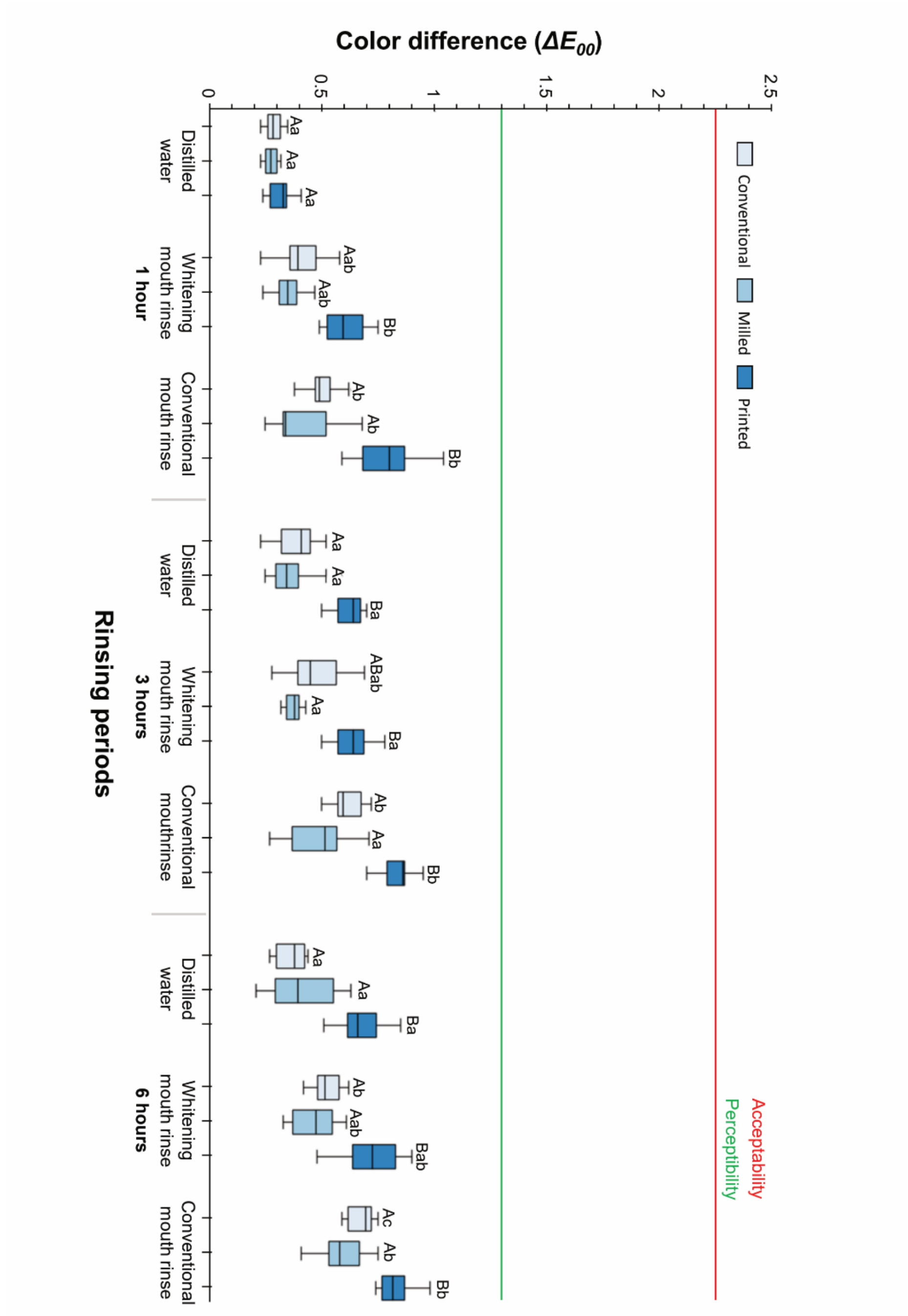

Figure 2.2. Color changes (ΔE_{00}) after short-term mouth rinsing simulation. The oral rinsing experiment was conducted for 1 to 168 hours to simulate 1 month to 14 years of daily mouth rinsing. Bonferroni correction for multiple tests: A<B<C, a<b<c. Different uppercase letters denote significant differences among different materials for same type of solution. Different lowercase letters denote significant differences among same type of materials for different solutions.

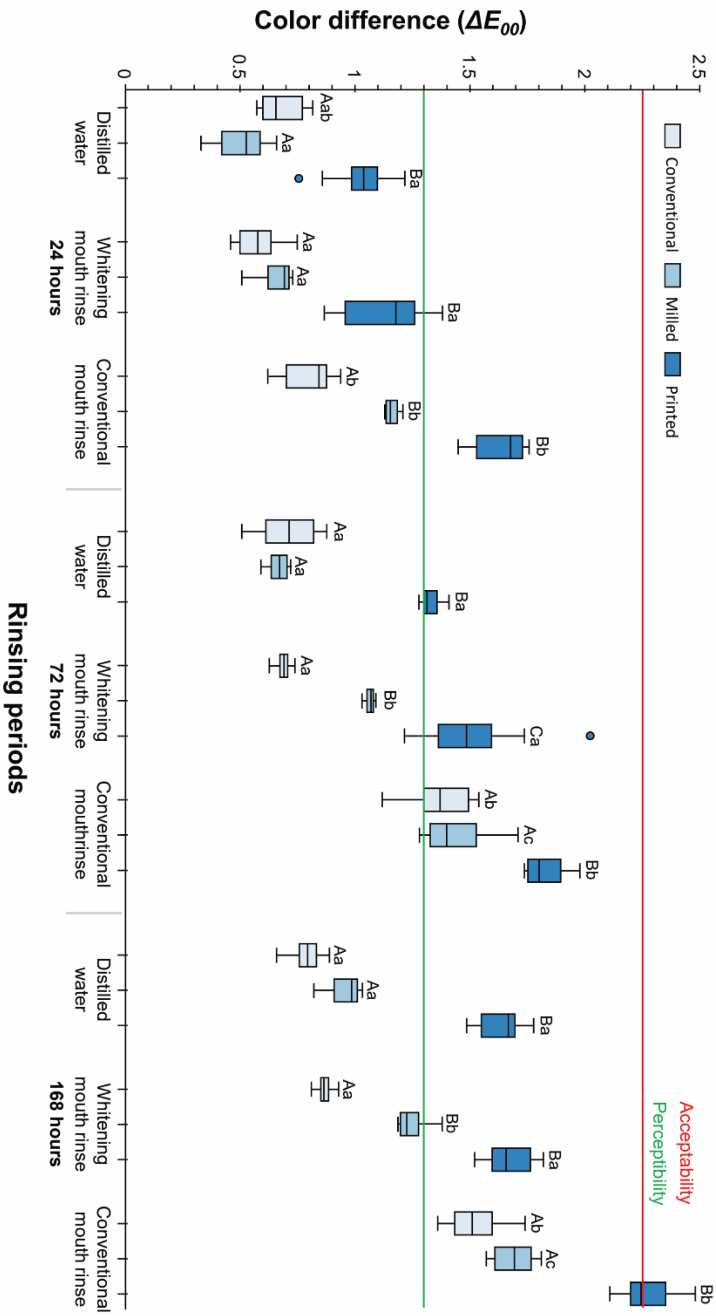

Figure 2.3. Color changes (ΔE_{00}) after long-term mouth rinsing simulation. The oral rinsing experiment was conducted for 1 to 168 hours to simulate 1 month to 14 years of daily mouth rinsing. Bonferroni correction for multiple tests: A<B<C, a<b<c. Different uppercase letters denote significant differences among different materials for same type of solution. Different lowercase letters denote significant differences among same type of materials for different solutions.

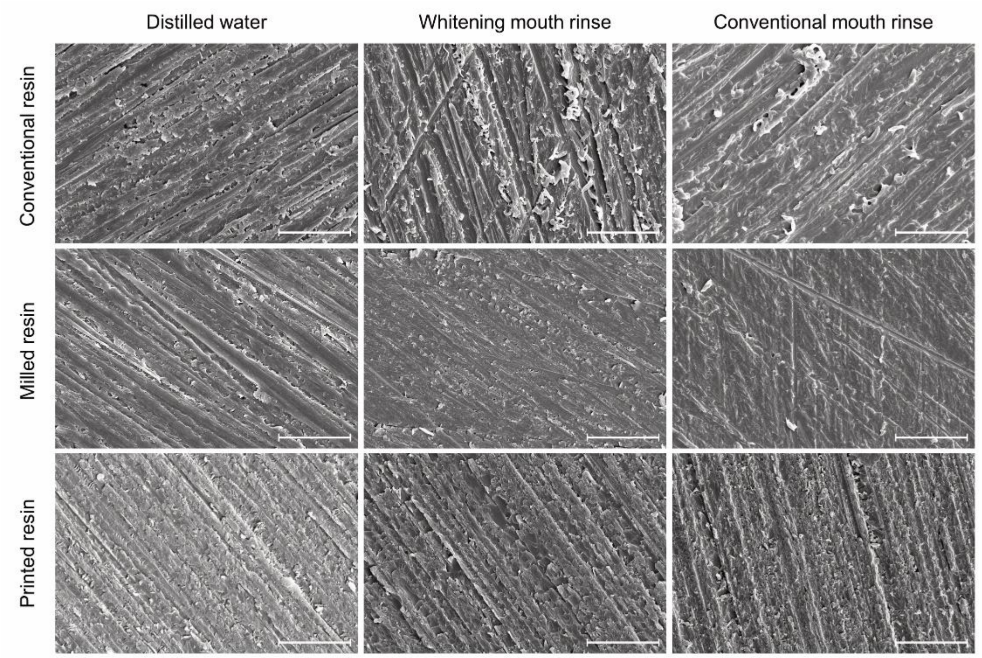

Figure 2.4. Scanning electron microscopy images of representative specimens after 6 hours of short-term oral rinsing simulation (scale bars, 20 μm)

국문 초록

디지털 치과용 임시 수복 재료의 색 안정성 및 마모 저항성에 관한 연구

GERELMAA MYAGMAR
서울대학교 대학원 치의과학과 치과보철학 전공
(지도교수 한 중 석)

연구목적: 최근의 디지털 제작 방식 활용의 확대는 치과 수복 분야에 상당한 영향을 미쳤다. 그러나 적층 가공법을 이용한 치과용 임시 수복 재료의 광학적 및 기계적 특성에 관련된 연구는 많지 않다. 따라서 본 연구의 목적은 디지털 치과용 임시 수복 재료들의 광학적 및 기계적 특성을 평가하는 것이었다. 실험 I에서는 3D 프린팅용 레진, CAD/CAM 밀링용 레진, 종래형 임시 수복용 레진의 마모에 따른 마모량과 표면 변화를 비교 평가하였다. 실험 II는 구강세정제 사용에 따른 디지털 임시 수복 레진과 기존의 임시 수복용 레진 재료의 색 안정성 및 표면 변화를 비교 평가하였다.

연구방법: 실험 I을 위하여 3D 프린팅용 레진, 밀링용 레진, 종래형 PMMA 레진을 이용해 총 48개의 직육면체 모양 시편을 제작하였다. 각 재료의 시편들을 다시 두 그룹으로 나누어 30,000회 또는 60,000회의 저작 시뮬레이션을 수행하였다(n=8). 각각의 재료의 마모량과 표면거칠기를 비교분석하였다. 통계분석은 일원배치 분산분석을 시행하였다(α=.05). 실험 II를 위하

여, 실험 I에서 사용한 것과 동일한 세가지 종류의 임시 수복용 레진으로 디스크 모양의 시편을 그룹당 10개씩 제작하였다(n=10). 제작한 세가지 종류의 시편들을 증류수(대조군), 미백기능강화 구강세정액, 종래형 구강세정액으로 나누어 단기간(1, 3, 6시간) 및 장기간(24, 72, 168시간)으로 세정하였다. 세정 전 후에 각각의 시편의 색조를 분광광도계를 사용하여 측정하였고, 색 변화량(ΔE_{00})을 계산하였다. 표면거칠기, 표면 형태도 함께 평가하였다. 통계 분석은 Shapiro−Wilk 정규성 검정 후, Kruskall−Wallis 비모수 분석법을 사용하였다(α=.05).

연구결과: 실험 I의 결과, 60,000 사이클 후의 마모량은 종래형 레진의 마모량이 0.44±0.01 mm^{3}으로 가장 컸으며, 밀링용 레진 0.21±0.02 mm^3, 3D 프린팅용 레진 0.10±0.01 mm^{3}순으로 작았다. 종래형 레진 군의 마모량은 밀링 군과 3D 프린팅 군보다 유의하게 컸다(p<.001). 60,000 사이클 후 표면거칠기(Ra)는 3D 프린팅 군이 0.59±0.06 μm으로 가장 작았고, 밀링 및 종래형 군에서 각각 1.27±0.49 μm, 1.64±0.44 μm순으로 컸다. 마모 후 3D 프린팅 및 종래형 군의 표면거칠기에서 통계적으로 유의한 차이가 관찰되었다(p=.01). 실험 II의 결과, 6시간 동안 세정하였을 때는, 밀링용 레진의 색조 변화는 종래형 레진의 색조 변화와 유의한 차이가 없었지만 (p>.334), 프린팅용 레진은 유의하게 큰 색조 변화를 보였다(p<.007). 종래형 구강세정제를 사용할 때 가장 큰 색조 변화가 관찰되었으나, 모든 색조 변화는 인지 가능 역치 미만이었다. 168시간 동안 세정하였을 때는, 모든 레진 그룹에서 종래형 구강세정제를 사용했을 때 인지 가능한 색조 변화를 보였고, 프린팅용 레진은 2.24±0.2로 가장 큰 ΔE_{00}을 나타냈다. 단기 및 장기 시뮬레이션 모두에서 종래형 구강세정제로 세정된 프린팅용 레진이 증류수로 세정된 것에 비해 보다 큰 표면거칠기를 보였다(p<.009).

결론: 적층 및 절삭 제작된 디지털 치과용 임시 수복 재료들은 마모 후 종래형 레진과 비교하여 적은 마모량을 보였다. 구강세정제 적용 후 모든 임시 수

복 재료들에서 색 변화가 관찰되었으나, 6개월 이내의 임상 사용을 시뮬레이션 한 경우, 해당 색 변화값들은 인지 가능 역치에 비해 낮았다. 본 연구의 결과는 디지털 방식으로 제작하는 적층 및 절삭 가공용 임시 수복 레진이 통상적인 임시 수복 기간 동안의 임시 수복물 재료로 기능할 수 있을 것임을 시사한다.

주요어: 3차원 프린팅; 색 안정성; 수복물 마모; 임시 수복물; 컴퓨터-기반 디자인; 표면 특성
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