# Micro-tensile bond strength between 3D printing resin and conventional provisional resin with different surface treatments and fracture toughness of combined resins with different thickness

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# Micro-tensile bond strength between 3D printing resin and conventional provisional resin with different surface treatments and fracture toughness of combined resins with different thickness

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This certifies that the Master's thesis of

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2021년 12월 장진영 올림

# Table of Contents

# List of Figures

# List of Tables

## Abstract

# Micro-tensile bond strength between 3D printing resin and conventional provisional resin with different surface treatments and fracture toughness of combined resins with different thickness

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## 1. Objectives

The objectives of this study were to compare the micro-tensile bond strength between 3D printing resin with different surface treatments and conventional provisional resin and to compare the fracture toughness when both resins are combined with different thickness.

## 2. Materials and Methods

3D printing resin blocks (RAYDENT C&B) with different surface treatments (Group NT: no surface treatment, Group ML: monomer liquid application for 60 seconds, Group CB: bond of Clearfil SE Bond application) were combined with conventional provisional resin (Tokuso Curefast). 20 beams (1 mm x 1 mm x 10 mm) per group were made and subjected to the micro-tensile bond strength test.

15 specimens (25 mm x 2 mm x 1.5 mm) per group with different composition (Group 1: 1.5-mm-thick conventional provisional resin, Group 2: 0.5-mm-thick 3D printing resin with 1.0-mm-thick conventional provisional resin, Group 3: 1.0-mm-thick 3D printing resin with 0.5-mm-thick conventional provisional resin, Group 4: 1.5-mm-thick 3D printing resin) were made. The 3-point bending test was performed for fracture toughness.

## 3. Results

For the mean micro-tensile bond strength, Group ML had significantly higher value than Group NT and Group CB ($p < 0.05$). For the mean fracture toughness, Group 3 and Group 4 showed significantly higher values than Group 1 and Group 2 ($p < 0.05$).

## 4. Conclusion

When provisional crowns made with 3D printing resin are relined by conventional provisional resin, pretreating the inside surface of the crowns with monomer liquid and ensuring the thickness of 3D printing resin would be recommended clinically.

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**Keywords**: conventional provisional resin; fracture toughness; micro-tensile bond strength; 3D printing resin

# I. Introduction

CAD/CAM (computer-aided-design/computer-aided-manufacturing) technology is currently widely used in dental laboratories and chairside. The milling technique, which cuts block-shaped materials to create a designed shape using a milling machine, is often considered as a synonym of CAD/CAM technology. However, it only refers to subtractive manufacturing among CAD/CAM technologies [1]. On the other hand, there is a three-dimensional (3D) printing technique that corresponds to additive manufacturing among CAD/CAM technologies. In the 3D printing technology, after a computer decomposes designed modules into many sections, then the 3D printer makes three-dimensional shape of the printed object by building up the layers based on this data. The major difference between the two CAD/CAM techniques is that in subtractive manufacturing, there is waste of excess materials while in additive manufacturing, there is no loss of materials [2]. Furthermore, there is limitation in making complex shapes in subtractive manufacturing because of its axis and size of the milling tool [3], while more complex geometrical structures can be produced in additive manufacturing [2].

There are various types of 3D printing methods, and they can be classified into liquid-based-, powder-based-, and solid-based methods according to the materials added. In the field of dentistry, the liquid-based 3D printer which uses liquid photopolymer resin as the material is mostly used. This includes stereolithography (SLA), digital light processing (DLP), liquid crystal display (LCD) and other printing technologies [4]. The SLA, which is the earliest 3D printing method, uses an ultraviolet (UV) layer to trace out cross-sections of the object. The UV laser solidifies the photo-curable liquid resin filled in a vat [5]. Both DLP and LCD are similar to SLA in that they are based on vat-polymerization technology, but they differ from the SLA in the light source used for polymerization [6]. DLP uses a digital micromirror device (DMD) to reflect the light and polymerize each layer [5, 6],

whereas LCD uses an UV light generated from an array of LEDs shining through an LCD monitor [7]. LCD 3D printers are inexpensive relative to other technologies, so it can be a low budget alternative prototyping solution [4, 7].

Provisional crowns are necessary in that they protect the prepared teeth from physical, chemical and thermal injuries and provide positional stability and occlusal function of teeth [8]. Conventional provisional resin which is used for fabrication of provisional restorations includes those based on monomethacrylates or acrylic resin and those based on dimethacrylates or bis-acryl/composite resin [9]. Autopolymerizing polymethyl methacrylate (PMMA) which is based on acrylic resin was introduced around 1940 and is frequently used materials for provisional restorations [10].

Provisional crowns are conventionally fabricated in direct, indirect, or indirect-direct method [11]. In direct method, provisional crowns are made on prepared teeth intraorally. Thus, direct method is technique sensitive as the quality of restoration totally depends on the operator's skills. On the other hand, in indirect method, provisional crowns are made on the stone model of prepared teeth following an impression after tooth preparation. However, it is less commonly used, because patients need to be seated at the chairside until the overall procedures are done [12]. More often, an indirect-direct method is another possible way [11]. In this method, a hollowed shell of a provisional crown is prefabricated from a mock preparation of the tooth, and then it is relined intraorally on the prepared tooth at chairside [12].

Recently, 3D printing technique can be used to fabricate provisional crowns both in indirect method and indirect-direct method. It can shorten the production time and save labor with the sufficient precision and quality of provisional crowns [13]. Several studies showed that 3D printing resin used as a material for provisional restorations has good enough mechanical properties compared to conventional provisional resin [14-16]. Also, provisional crowns fabricated by 3D printers have good marginal integrity and internal fit [17, 18]. However, they might need of modification at chairside by relining conventional

provisional resin for correction because marginal fit is relatively inaccurate at a certain location like subgingival finish line [19]. Also, in indirect-direct method, provisional crowns are prefabricated by 3D printers as a form of shell and then they are relined with conventional provisional resin after the tooth preparation to get internal fit.

It is important that relined conventional provisional resin combines well with the 3D printing resin to form a durable bond. Some in vitro studies analyzed the bond strength between 3D printing resin and conventional provisional resin [20-22], but there are still limited data about it.

Meanwhile, the thickness of the relined resin varies according to the thickness of the shell. Within a limited thickness, the strength can vary depending on which material has more portion. It is also important that provisional crowns fabricated by such a way have mechanical properties to avoid fracture. However, there is little information about the fracture toughness when 3D printing resin and temporary provisional resin are combined each other with different thickness.

Therefore, the objectives of this study were 1) to compare the micro-tensile bond strength between 3D printing resin and conventional provisional resin according to the different surface treatments of 3D printing resin, and 2) to compare the fracture toughness when both resins with different thickness are combined in the same total thickness. The null hypotheses to be tested are that 1) different surface treatments of 3D printing resin do not affect the micro-tensile bond strength between 3D printing resin and conventional provisional resin, and 2) different thickness of both 3D printing resin and conventional provisional resin does not affect the fracture toughness of combined two resins.

# II. Materials & Methods

## 1. Micro-tensile bond strength test

The materials used in this study are summarized in Table 1.

**Table 1**. Materials used in this study.

| Materials | Manufacturers | Composition | Lot number |
|---|---|---|---|
| **RAYDENT C&B** | RAY Co., Seongnam, Korea | Monomer based acrylic esters:<br>Low molecular weight Urethane Acrylate Polymer (proprietary)<br>(octahydro-4,7-methano-1H-indenediyl)bis(methylene)bismethacrylate<br>2-Hydroxyethyl Methacrylate<br>2,4,6-trimethylbenzoyl-diphenyl phosphine oxide<br>1,6-Hexanediol diacrylate<br>Color pigments (proprietary)<br>4-Methoxyphenol | RCB21132B |
| **Tokuso CureFast** | Tokuyama Dental Corp., Tokyo, Japan | Powder:<br>Polymethyl methacrylate,<br>Benzoyl peroxide,<br>Di-isobutyl azonitrile Dibutyl phthalate | 836M |
| | | Liquid:<br>Methyl methacrylate, | 316M |

| | | | |
|---|---|---|---|
| | | Hydroquinone,<br>N, N-dimethyl-para-toluidine,<br>Butyl or Octyl methacrylate Glycol dimethacrylate | |
| **Clearfil SE Bond** | Kuraray Noritake Dental Inc., Sakazu, Japan | * Primer not used<br>Bond:<br>2-Hydroxyethyl methacrylate (HEMA),<br>10-Methacryloyloxydecyl dihydrogen phosphate (MDP),<br>Bisphenol A diglycidylmethacrylate (Bis-GMA)<br>Hydrophobic dimethacrylate<br>dl-Camphorquinone<br>N,N-diethanol-p-toluidine<br>Silanated colloidal silica | 6Q0565 |

Three 3D printing resin blocks (15 mm in width, 15 mm in length, 5 mm in thickness) were made using the 3D printer (RAYDENT Studio, Ray Co., Seongnam, Korea) by LCD method with 3D printing resin (RAYDENT C&B, Ray Co., Seongnam, Korea). The monomer remaining on the surface of the blocks was washed off using water. Then they were post-cured for 10 minutes using post-curing unit (RPC 500, Ray Co., Seongnam, Korea) with a 395-nm UV light with a intensity of 120 $\mu Wcm^{-2}$ and internal temperature of 60 °C.

After storage of the blocks in the air at room temperature for 7 days, surfaces of each block were ground for 5 times in one direction using 800-grit silicon carbide (SiC) paper (Silicon Carbide Water Proof Abrasive Paper Electro Coated, Daesung Abrasive Co., Seoul, Korea). After that, blocks were cleaned in distilled water for 5 minutes using an ultrasonic device (U-SONIC, Uil Ultra Sonic Co., Ansan, Korea). Then, the blocks were placed on

clean paper towels and dried with oil-free compressed air. The blocks were randomly divided into three groups (n=1) according to the different surface treatments of the 3D printing resin. The surface treatments method of each group are as follows.

(1) Group NT (control): No additional surface treatment

(2) Group ML: The monomer liquid (Tokuso Curefast, Tokuyama Dental Corp., Tokyo, Japan) was applied to the surface of the 3D printing resin block with a microbrush by gently rubbing it for 5 seconds. Then, the block was left for 60 seconds and the surface remained wet.

(3) Group CB: Without application of primer of Clearfil SE Bond, only bond of Clearfil SE Bond (Kuraray Noritake Dental Inc., Sakazu, Japan) was applied to the surface of the 3D printing resin block with a microbrush by gently rubbing it for 5 seconds. Then, to make the bonding agent thinner, oil-free compressed air was blown for 30 seconds at a distance about 10 cm to the block. After that, light curing was carried out for 10 seconds using LED light-curing unit (Elipar Deep Cure-L, 3 M ESPE, St. Paul, MN) at a distance about 1 cm with 850 mW/cm$^2$.

Afterwards, each block was put into the silicone mold to be layered by autopolymerizing acrylic resin (Tokuso Curefast, Tokuyama Dental Corp., Tokyo, Japan) above.

In order to make the size of the layering PMMA resin the same as the size of the 3D printing resin, the inner space of the mold was made to have a rectangular parallelepiped shape with a width of 15 mm, a length of 15 mm, and a thickness of 10 mm.

According to the manufacturer's instructions, the powder and liquid of relining temporary resin were mixed by hand using a plastic spatula, injected into a silicone mold, and left at room temperature for 10 minutes to polymerize. Then, the block in which the two resins were combined was separated from the silicone mold.

Then, the block was attached to a transparent acrylic plate (15 mm in width, 15 mm

in length, 20 mm in thickness) using cyanoacrylate glue (Loctite 401, Henkel, Germany) and then fixed to a low-speed diamond cutter (RB 205 Metsaw-LS, R&B Corp., Daegu, Korea)

Afterwards, the blocks were serially sectioned into 1-mm-thick slices with a diamond saw (C3B-150-TMN-SOLID, R&B Corp., Daegu, Korea) rotating at 500 rpm under water cooling and then sectioned again at a right angle. Finally, the blocks were sectioned into beams (1 mm x 1 mm x 10 mm) with a cross sectional bonded area of approximately 1 $mm^2$. After that, the beams were carefully separated from the acrylic plate using a tweezer. The cross-sectional area for each specimen was calculated by measuring the width and length of specimens using an electrical digital caliper (Model CD-15CP, Mitutoyo Corp., Kawasaki, Japan). Finally, a total of 60 specimens (20 beams per group) were made, as shown in Figure 1. Then they were put in a microtube, soaked in distilled water, and stored in an incubator (C-INDF3, Chang shin scientific Co., Seoul, Korea) at 36 °C for 24 hours.

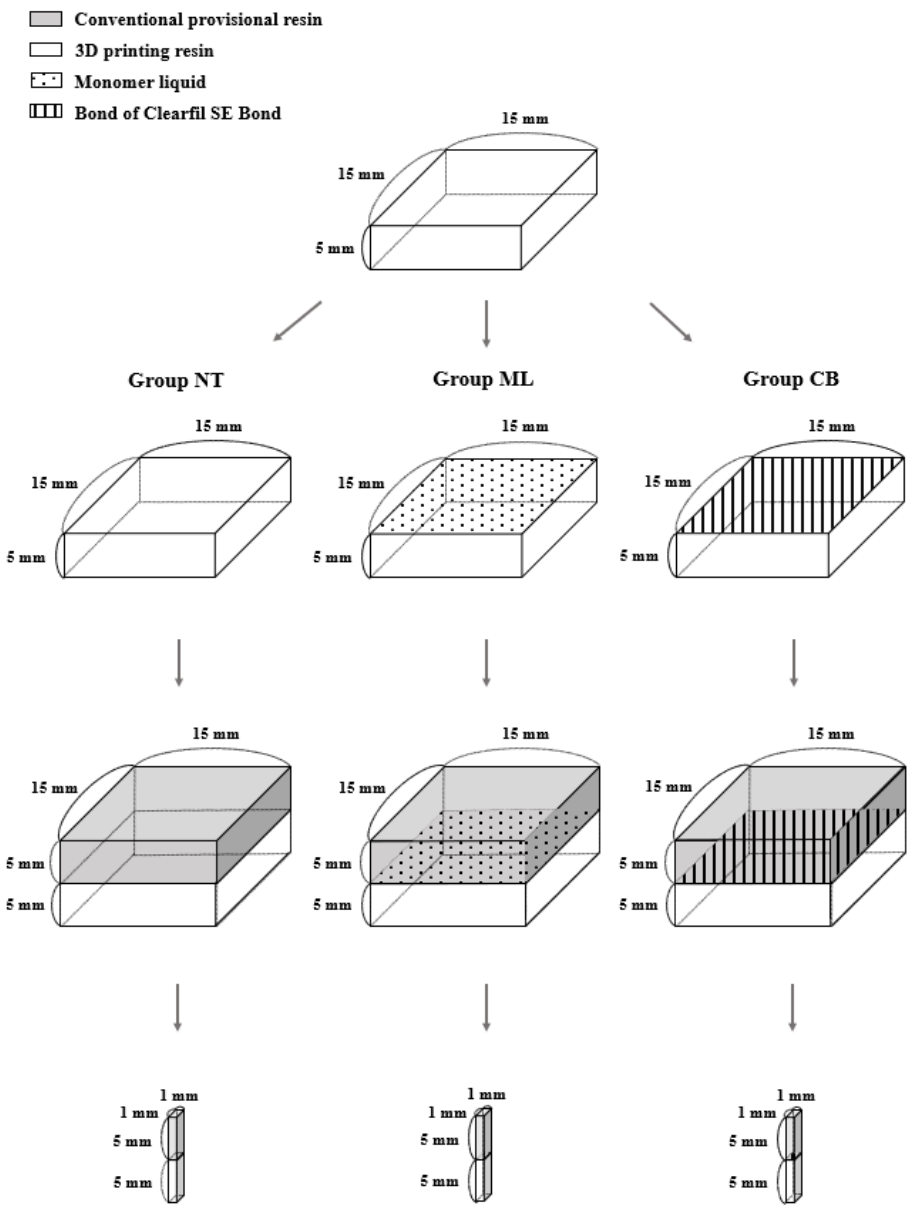


**Figure 1.** Diagram of the micro-tensile bond strength test specimens. The bonding surface was treated with different methods; no treatment, monomer liquid, bond of Clearfil SE Bond.

Then, for the micro-tensile bond strength test, the specimens were fixed on a jig with cyanoacrylate glue (Zapit, Super Glue Corp., Ontario, U.S.A), and then using a universal testing machine (EZ-test, Shimadzu Corp., Kyoto, Japan) a tensile force was applied to the specimen at a cross head speed of 1 mm/min.

Trapezium X software (version 1.5.1, Shimadzu Corp., Kyoto, Japan) was used to record the load (N) over time until fracture of the specimen occurred. The stress value (MPa) obtained by dividing the peak load (N) recorded at fracture by the cross-sectional area ($mm^2$) of the specimen was used as micro-tensile bond strength (MPa).

After testing, all specimen pairs available were observed under the dental operating microscope (S100 OPMI pico, Carl Zeiss Meditec AG, Jena, Germany) at a magnification of 21.25 to determine the mode of failure. Failure modes were classified into five categories as

(a) Adhesive failure: In Group NT and Group ML, failure observed between 3D printing resin and conventional provisional resin. In Group CB, failure observed between the bonding agent and either side of the resin (3D printing resin or conventional provisional resin)

(b) Cohesive failure within 3D printing resin

(c) Cohesive failure within conventional provisional resin

(d) Cohesive failure within the bonding agent: This category is only for Group CB

(e) Mixed failure: failure including both adhesive and cohesive failure.

The results were expressed in distribution of failure mode for tested groups.

## 2. Fracture toughness test

For the fracture toughness test, rectangular parallelepiped specimens (25mm in width, 2mm in length, 1.5mm in thickness) were made. 3D printing resin and conventional provisional resin material used in the study is the same as in the micro-tensile bond strength test (Table 1.). Groups were divided according to the thickness of 3D printing resin and conventional provisional resin combined each other in the total thickness of 1.5mm (Figure 2.).

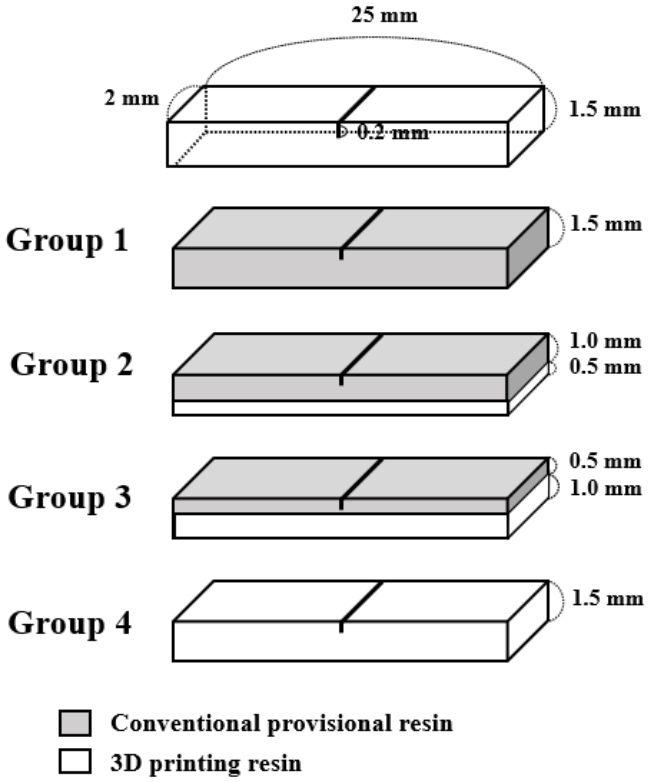


**Figure 2.** Diagram of the fracture toughness test specimens.

(1) Group 1: specimens consist of 1.5-mm-thick conventional provisional resin

(2) Group 2: specimens consist of 0.5-mm-thick 3D printing resin combined with 1.0-mm-thick conventional provisional resin

(3) Group 3: specimens consist of 1.0-mm-thick 3D printing resin combined with 0.5-mm-thick conventional provisional resin

(4) Group 4: specimens consist of 1.5-mm-thick 3D printing resin

Each fifteen 0.5-, 1.0-, 1.5-mm-thick 3D printing resin beams were made using a LCD 3D printer with a photocurable resin (RAYDENT C&B). The monomer remaining on the surface of the blocks was washed off using water. Then they were post-cured for 10 minutes using post-curing unit (RPC 500) with 395-nm UV light with an intensity of 120 $\mu Wcm^{-2}$ and internal temperature of 60 °C.

The beams were stored in the air at room temperature for 7 days before specimen fabrication and there was no additional surface treatment on the beams.

In order to reduce errors in making specimens, a customized metal mold having the inside space with a width of 25 mm, a length of 2 mm, and a thickness of 1.5 mm was made. The metal mold was made to be attached and detached. A 0.2-mm-deep groove line was made at the center of the mold so that a notch could be formed in the specimens by positioning the blade in this groove.

The specimen preparation procedures for each group (n=15) were as follows.

*Group 1*

For easy separation from the specimen, a small amount of Vaseline (White Petrolatum, Firson, Cheonan, Korea) was applied to the metal mold with a microbrush, and then a mylar strip was placed under the mold. After that, according to the manufacturer's instructions, the powder and liquid of relining temporary resin were mixed by hand using a plastic spatula, and then poured into the empty space inside the metal mold. After that, a mylar

strip was placed on the top of the mold, covered with slide glass, and pressed with finger pressure to make the conventional provisional resin level at the top of the mold. Before the PMMA resin was completely polymerized, the slide glass and mylar strip were removed and the resin excess outside the frame was removed. Then the blade (Dorco procut, Dorco living vina Co., Seoul, Korea) was placed in the mold groove and therefore a notch could be formed at the top surface of the conventional provisional resin. After leaving the resin for 10 minutes to polymerize, the blade was removed and the specimen was carefully separated from the mold. Irregular thin excess was removed by grinding with 800-grit silicon carbide (SiC) paper (Silicon Carbide Water Proof Abrasive Paper Electro Coated, Daesung Abrasive Co., Seoul, Korea) attached to the specimen.

*Group 2 and Group 3*

After applying the Vaseline to the metal mold in the same way as in the Group 1, 0.5-mm-thick 3D printing resin specimen (Group 2) or 1.0-mm-thick 3D printing resin specimen (Group 3) was placed into the mold. After that, the powder and liquid of relining temporary resin were mixed in the same way as in the Group 1, and then poured into the space above the 3D printing resin specimen inside the metal mold. Therefore, in Group 2, the 1.0-mm-thick provisional resin was lined above the 0.5-mm-thick 3D printing resin, and in Group 3, the 0.5-mm-thick provisional resin was lined above the 1.0-mm-thick 3D printing resin. Following procedures including notch formation are the same as in the Group 1.

*Group 4*

A 1.5-mm-thick 3D printing resin was placed in the metal mold. Then, the blade was carefully moved several times along the straight groove in the mold over the top surface of the 3D printing resin, so that a 0.2-mm-deep notch could be formed on the top surface of the 3D printing resin.

After specimen preparation, the width and the thickness of the specimens were measured using an electrical digital caliper (Model CD-15CP, Mitutoyo Corp., Kawasaki,

Japan). A total of 15 specimens per group were prepared and then stored in the air at room temperature for 24 hours.

For the fracture toughness test, the specimen was placed on the test jig with the notch side facing the floor. Then, the 3-point bending test was performed at a crosshead speed of 1 mm/min using a universal testing machine (EZ-test, Shimadzu Corp., Kyoto, Japan). Trapezium X software (version 1.5.1, Shimadzu Corp., Kyoto, Japan) was used to measure the load (N) over time until fracture of the specimen occurred, and a stress-strain graph was obtained. The peak load (N) at the fracture of the specimen was recorded. Fracture toughness ($K_{IC}$) (MPa) was calculated according to the following equation provided by ASTM standard E399-83 [23-26].

$$K_{IC}= \frac{3PL}{2BW^{1.5}}\left\{1.93\left(\frac{a}{W}\right)^{0.5} - 3.07\left(\frac{a}{W}\right)^{1.5} + 14.53\left(\frac{a}{W}\right)^{2.5} - 25.11\left(\frac{a}{W}\right)^{3.5} + 25.8\left(\frac{a}{W}\right)^{4.5}\right\}$$

where P is load at fracture (N), L is the length, W is the width, B is the thickness, and a is the notch length (all in mm).

## 3. Statistical analysis

Statistical analysis was performed using SAS version 9.4 (SAS Inc., Cary, NC, USA). The mean and standard deviation of micro-tensile bond strength and fracture toughness was calculated respectively. For the normality test of variables, Kolmogorov-Smirnov test was used. The data were analyzed respectively using one-way ANOVA, followed by Bonferroni correction test as a post hoc analysis. For all statistical analyses, significance level of 0.05 was used.

# III. Results

## 1. Micro-tensile bond strength test

Table 2 and Figure 3 show the mean and standard deviation of micro-tensile bond strength (μTBS) of tested groups.

**Table 2.** Mean and standard deviation of micro-tensile bond strength (MPa) of tested groups.

| | Group NT | Group ML | Group CB |
|---|---|---|---|
| **micro-tensile bond strength** | 26.79±5.60[b] | 36.08±6.87[a] | 23.36±6.25[b] |

Same superscript letters indicate no statistically significant difference between groups ($p > 0.05$).

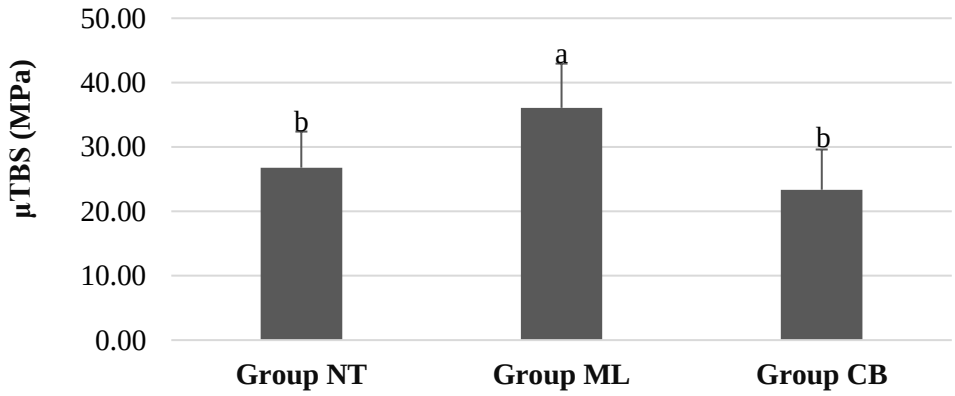


**Figure 3**. Bar graphs for mean and standard deviation of micro-tensile bond strength (MPa) of tested groups. Same superscript letters indicate no statistically significant difference between groups ($p > 0.05$).

For the mean μTBS, Group ML had the highest value, which was significantly higher than that of the Group NT and that of Group CB ($p < 0.05$). On the other hand, the mean μTBS of Group CB was similar to that of Group NT and there was no statistically significant difference between them ($p > 0.05$). Table 3. shows the post hoc test results.

**Table 3.** Post hoc test results of the micro-tensile bond strength test.

| | **Group NT vs Group ML** | **Group NT vs Group CB** | **Group ML vs Group CB** |
|---|---|---|---|
| ***p*-value** | <.0001 | 0.2664 | <.0001 |

Table 4 and Figure 4 show failure mode distribution of tested groups.

**Table 4.** Failure mode distribution of tested groups.

| | **Group NT** n (%) | **Group ML** n (%) | **Group CB** n (%) |
|---|---|---|---|
| **Adhesive** | 16 (80) | 1 (5) | 5 (25) |
| **Cohesive, 3D printing resin** | 4 (20) | 15 (75) | 7 (35) |
| **Cohesive, Conventional provisional resin** | 0 (0) | 4 (20) | 0 (0) |
| **Cohesive, Bonding agent** | - | - | 1 (5) |
| **Mixed** | 0 (0) | 0 (0) | 7 (35) |

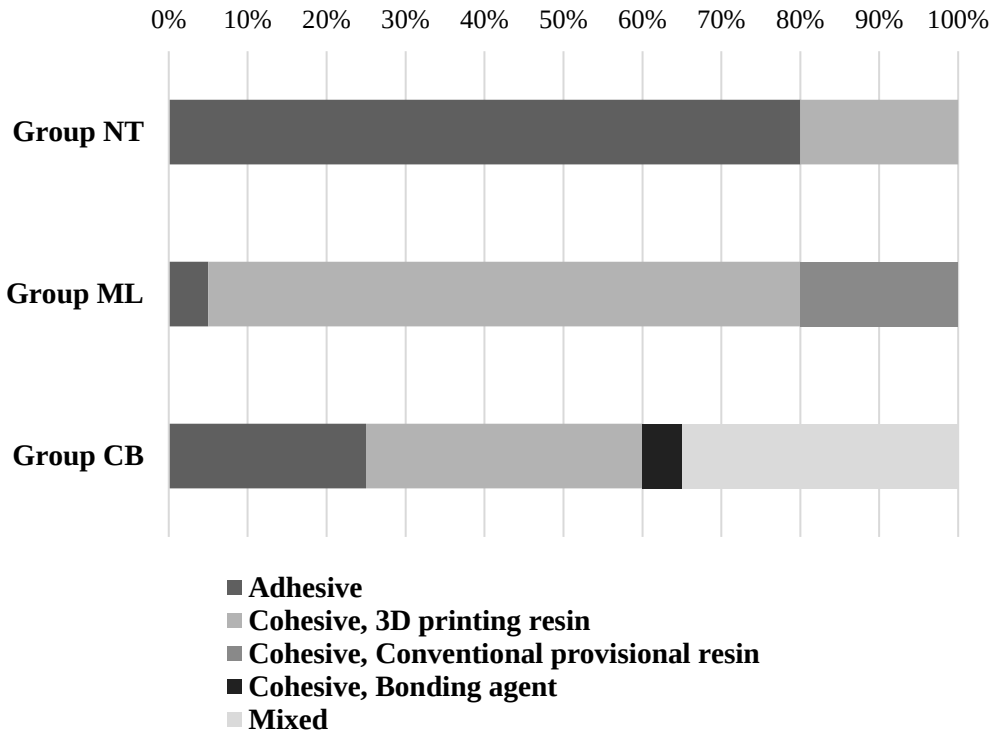


**Figure 4.** Failure mode distribution of tested groups.

In Group NT, most of the specimens showed adhesive failure (16/ 20, 80%), while several specimens displayed cohesive failure within 3D printing resin (4/ 20, 20%). On the other hand, in Group ML cohesive failure within 3D printing resin (15/ 20, 75%) was most frequent, followed by cohesive failure within conventional provisional resin (4/ 20, 20%). In Group CB, the percentage of cohesive failure within 3D printing resin and mixed failure was the same (7/ 20, 35%), followed by adhesive failure (5/ 20, 25%).

## 2. Fracture toughness test

Table 5 and Figure 5 show the mean and standard deviation of fracture toughness of tested groups.

**Table 5.** Mean and standard deviation of fracture toughness (MPa $m^{0.5}$) of tested groups.

| | Group 1 | Group 2 | Group 3 | Group 4 |
|---|---|---|---|---|
| **Fracture toughness** | $26.65 \pm 3.05^{b}$ | $27.36 \pm 4.20^{b}$ | $32.58 \pm 4.74^{a}$ | $32.99 \pm 6.73^{a}$ |

Same superscript letters indicate no statistically significant difference between groups ($p > 0.05$).

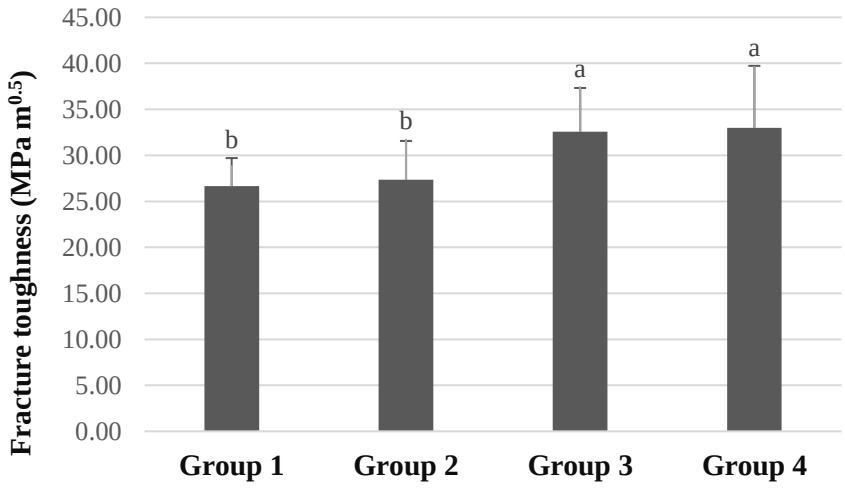


**Figure 5.** Bar graphs for mean and standard deviation of fracture toughness (MPa $m^{0.5}$) of tested groups. Same superscript letters indicate no statistically significant difference between groups ($p > 0.05$).

The mean fracture toughness was high in the order of Group 4, Group 3, Group 2, and Group 1. For the mean fracture toughness, Group 4 which consisted of 1.5-mm-thick 3D printing resin showed significantly higher values than Group 1 which consisted of 1.5-mm-thick conventional provisional resin ($p < 0.05$).

When 3D printing resin and conventional provisional resin were combined, the mean fracture toughness was affected by how much thickness both materials occupied within the same thickness. Group 3 which consisted of 1.0-mm-thick 3D printing resin and 0.5-mm-thick PMMA resin, showed similar value to Group 4, and there was no statistically significant difference between them ($p > 0.05$). On the other hand, Group 2 which consisted of 0.5-mm-thick 3D printing resin and 1.0-mm-thick PMMA resin, show not only significantly lower value than Group 4 but also than Group 3 ($p < 0.05$). In addition, there was no statistically significant difference between Group 2 and Group 1 ($p > 0.05$). Table 6. shows the results of the post hoc test.

**Table 6.** Post hoc test results of the fracture toughness test.

| | **Group 1 vs Group 2** | **Group 1 vs Group 3** | **Group 1 vs Group 4** | **Group 2 vs Group 3** | **Group 2 vs Group 4** | **Group 3 vs Group 4** |
|---|---|---|---|---|---|---|
| ***p*-value** | >.9999 | 0.0092 | 0.0045 | 0.029 | 0.0149 | >.9999 |

# IV. Discussion

In this study, the mean micro-tensile bond strength of the Group ML was significantly higher than that of the Group NT and Group CB. Therefore, the first null hypothesis that different surface treatments of 3D printing resin do not affect the micro-tensile bond strength between 3D printing resin and conventional provisional resin was rejected.

The 3D printing resin used in this study is monomer based acrylic ester without any filler. Conventional provisional resin used in this study is polymethyl methacrylate (PMMA) which is based on monomethacrylates or acrylic resins. The physical surface treatment used in this study was only 800-grit SiC paper grinding.

In the Group NT with no further treatment, the bonding mechanism of PMMA resin to 3D printing resin can be considered in two ways. The first way is to achieve micromechanical retention by impregnation of PMMA resin into irregularities of the grounded 3D printing resin surface. The second way is to achieve chemical coupling between PMMA resin and the 3D printing resin. When it comes to repair, chemical bonding between layers of a resin composite relies on co-polymerization between new resin monomer and residual unreacted methacrylate groups [27]. If unconverted C=C double bond remains available on the surface of the 3D printing resin, there is possibility of chemical coupling with relining conventional temporary resin. However, as failure mode analysis in Group NT revealed that most of the fractures occurred at the interface between two materials, it seems that the bond between 3D printing resin and PMMA resin is mainly acquired not by chemical coupling but by micromechanical retention.

In the Group ML where the surface of the 3D printing resin was treated with monomer liquid for 60 seconds, the monomer liquid would have increased the bond strength on the surface of the 3D printing resin specimen by the following mechanism; Mainly, monomer liquid would have been penetrated into the matrix of 3D printing resin and contributed to

micromechanical retention. Additionally, it may have wetted the surface of 3D printing resin better due to its low viscosity. Also, it may have dissolved the surface structure of 3D printing resin. Vallittu et al. [28] reported that when a heat polymerized acrylic resin was wetted with methacrylate monomer, the monomer liquid dissolved the surface structure of PMMA resin, which resulted in high bond strength with autopolymerizing acrylic resin. This may explain the results of this study. As cohesive failure within 3D printing resin was most frequently observed in Group ML with sparse adhesive failure, it can be thought that softening the surface of 3D printing resin by the monomer might occurred and this contributed to better bond strength at the interface than Group NT. However, Lim et al.[21] showed that pretreatment of methyl methacrylate monomer on 3D printing resin did not significantly increase shear bond strength between 3D printing resin and conventional provisional resin, despite the observation of dissolved and swelled surface of 3D printing resin. So further studies about the effect of monomer liquid application on the bond strength between 3D printing resin with relining materials are needed.

In the Group CB, bond of Clearfil SE Bond was used in this study because Clearfil SE Bond is commonly used two step self-etching system in clinical practice and its bond part can provide hydrophobic layer. Many studies have reported that the bond strength is increased when bonding agents are used for resin repair [29-32]. The main purpose of the application of bonding agents during resin repair is to improve chemical coupling with the resin matrix or filler particles with additional improvement of micromechanical retention. However, in this study, bond of Clearfil SE Bond did not have a significant effect on the bond strength. Thus, bond of Clearfil SE Bond does not seem to chemically react with the 3D printing resin substrate nor to achieve better micromechanical retention enough to improve bond strength.

As a result of the 3-point bending test, the mean fracture toughness of the Group 3 which consisted of thick 3D printing resin combined with thin conventional provisional resin was significantly higher than that of the Group 2 which consisted of thin 3D printing resin combined with thick conventional provisional resin. Therefore, the second null

hypothesis that different thickness of both 3D printing resin and conventional provisional resin does not affect the fracture toughness of combined two resins, was rejected.

3D printing resin is a light-curing resin, while PMMA resin is autopolymerizing resin. The degree of conversion of light-curing resin is higher than that of autopolymerizing resin. Tahayeri et al. [14] reported that degree of conversion of 3D printed resin by SLA method was higher than that of conventional provisional resin. High conversion rate of resin enhances the physical and mechanical properties. Cho et al. [16] revealed that the temporary restorative resin, made by using the SLA and DLP 3D printers, showed higher fracture strength than the temporary restorative resin (PMMA) made by the traditional method. Also, Ahn et al. [15] reported that 3D printing resin made by SLA and DLP 3D printer had significantly lower wear loss than conventionally self-cured resin.

Furthermore, 3D printing resin in this study was subjected to post-curing. Reymus et al. [33] reported post-curing of 3D printed resin increased the degree of conversion. Also, Kim et al. [34] reported that post-curing resulted in a significant increase in the degree of conversion of 3D printing resin and it led to better mechanical properties.

In this study, 1.5-mm-thick specimens were made considering the ideal amount of functional cusp reduction required for tooth preparation for cast metal crown. In clinical situation, sometimes we experienced fracture of the provisional restoration made with 3D printing resin relined by acrylic resin. According to the result of this study, this could happen when the thickness of the 3D printing resin is less than 1 mm in combined provisional restoration. To avoid the fracture, the final thickness of 3D printing resin in relined restoration should be more than 1 mm even after occlusal adjustment.

The limitation of the study is that only conventional provisional material based on acrylic resin was used in this study. There are two types of conventional provisional resin; those based on monomethacrylates or acrylic resin and those based on dimethacrylates or bis-acryl/composite resin. Methacrylate resins are mono-functional, low-molecular weight and linear molecules, while the bis-acryl resins contain multifunctional cross-linked

monomers and inorganic fillers.

For the fracture toughness, Astudillo-Rubio et al. [9] reported in their systematic review and meta-analysis that dimethacrylate-based provisional restorations presented better mechanical behavior than monomethacrylate-based ones in terms of flexural strength and hardness but there was no significant difference in fracture toughness.

For the bond strength, Lim et al. [21] reported that bis-acrylic resin showed higher shear bond strength to DLP 3D printed resin compared to PMMA resin. Also, Jeong et al. [20] reported that dimethacrylate resin showed higher shear bond strength to DLP and SLA 3D printed resin than monomethacrylate resin. However, Albahri et al. [22] reported that there was no significant difference in the shear bond strength of PMMA resin and bis-acrylic resin to SLA 3D printed resin. Therefore, further studies including both types of conventional provisional resin with various materials are needed.

Also, in this study, only one 3D printing resin material made by LCD 3D printer was used. Manufacturers do not release all the information of 3D printing materials and it remains unclear if the chemical composition of 3D printing provisional material differs from conventional provisional materials [35]. Further studies are needed with various types of conventional provisional resin and 3D printing resin as well.

# V. Conclusion

Within the limitation of the present study, the conclusion was as follows.

Monomer liquid application to the surface of 3D printing resin for 60 seconds significantly improved the micro-tensile bond strength between 3D printing resin and conventional provisional resin.

3D printing resin showed significantly higher fracture toughness compared to conventional provisional resin. Also, when 3D printing resin and conventional provisional resin are combined, those comprised of thick 3D printing resin and thin conventional resin showed higher fracture toughness than those comprised of thin 3D printing resin and thick conventional resin.

Thus, when provisional crowns fabricated by 3D printing resin are relined by conventional provisional resin, pretreating the inside surface of the crowns with monomer liquid and ensuring the thickness of 3D printing resin would be recommended clinically.

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**Abstract (In Korean)**

# 3D 프린팅 레진의 표면처치에 따른 임시 보철물 제작용 레진과의 미세인장결합강도 및 두 레진의 결합두께에 따른 파괴인성

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## Ⅰ. 목적

본 논문의 목적은 1) 서로 다른 표면처치를 한 3D 프린팅 레진과 임시보철물 제작용 레진 사이의 미세인장 결합강도를 비교하고, 2) 두 레진이 서로 다른 두께로 결합했을 때 파괴인성을 비교하기 위함이다.

## Ⅱ. 방법 및 재료

3D 프린팅 레진으로 RAYDENT C&B를, 임시보철물 제작용 레진으로 Tokuso Curefast를 사용하였다. 서로 다른 표면 처치 (NT 군: 표면 처치

없음, ML 군: 액상 모노머 60초 도포, CB 군: Clearfil SE Bond의 bond 도포 후 광중합)를 한 3D 프린팅 레진 블록에 임시보철물 제작용 레진을 결합시킨 후 잘라서 군 당 20개의 시편 (1 mm x 1 mm x 10 mm)을 제작하였고 이를 대상으로 미세인장 결합강도 시험을 시행하였다.

일정한 두께 안에서 서로 다른 레진 구성 (1 군: 1.5 mm 두께의 임시보철물 제작용 레진, 2 군: 0.5 mm 두께의 3D 프린팅 레진과 1.0 mm 두께의 임시보철물 제작용 레진이 결합됨, 3 군: 1.0 mm 두께의 3D 프린팅 레진과 0.5 mm 두께의 임시보철물 제작용 레진이 결합됨, 4 군: 1.5 mm 두께의 3D 프린팅 레진)을 가진 시편 (25 mm x 2 mm x 1.5 mm)을 군 당 15개씩 제작하였고, 이를 대상으로 3점 굽힘 시험을 시행하여 파괴인성을 구하였다.

## Ⅲ. 결과

평균 미세인장 결합강도는 ML 군이 NT 군과 CB 군에 비해 유의하게 높았다 ($p < 0.05$). 평균 파괴인성은 3 군과 4 군이 1 군과 2 군보다 유의하게 높았다. ($p < 0.05$).

## Ⅳ. 결론

임상적으로 3D 프린팅 레진으로 만든 임시치관을 임시보철물 제작용 레진으로 이장하는 경우, 임시치관 내면을 액상 모노머로 전처치하고, 3D 프린팅 레진의 두께를 확보하는 것이 이점을 줄 것이다.

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핵심되는 단어: 미세인장 결합강도; 임시보철물 제작용 레진; 파괴인성; 3D 프린팅 레진