Original Article

Shear bond strength of provisional repair materials bonded to 3D printed resin

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Abstract  Background/purpose: There is limited literature on the materials of choice and their properties when repairing 3-D printed resin-based restorations. The objective of this in-vitro study is to determine the shear bond strength of various repair materials to 3D printed SLA (stereolithography) resin.

Materials and methods: For Group A (control), fifteen cylinders of 3-D printing SLA resin were printed as one unit of a Ø6.8 x 8 mm (diameter and height) cylindrical block with a Ø3 x 5 mm cylindrical block at the center. For the test groups, forty-five specimen cylinders of 3-D printing SLA resin (Ø6.8 x 8 mm) were fabricated and the surfaces were treated with 3 different test materials: Group B: Poly-Methyl Methacrylate (PMMA); Group C: Bis-acrylic composite resin, and Group D: Bis-GMA composite All specimens were tested using an Instron machine at a crosshead speed of 0.5 mm/min. A Shapiro–Wilk test was used to assess normality within the data, then the data was statistically analyzed by a Mann–Whitney test.

Results: There were no statistically significant differences between testing groups, except Group A. Group B displayed mixed (87%) and adhesive (13%) failure at the fractured surface. Group C showed both mixed (60%) and adhesive failure at the fractured surface (40%). All Group D showed mixed fracture patterns, partly cohesive fractured surface within the base cylinder area and partly adhesive fractured surface at the bonded interface.

Conclusion: No statistically significant differences in the shear bond strength of the different repair materials to 3D printed cylinders were observed. The 3D printed cylinder repaired with Bis-GMA composite demonstrated the most predictability from the fractography analysis.

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Introduction

Provisional crowns for fixed restorations serve multiple important purposes over the course of prosthodontic treatment. Some of their functions include: providing pulpal protection, preserving positional stability, maintenance of occlusal function, and verifying functional and esthetic suitability for the patient.\(^1\) One of the requirements for provisional restorations should be the ease of alterability and repair as this factor would be extremely important to the success or failure of treatment outcomes.\(^4\) The most common technical issue with provisional restorations is the need to repair or reline them as they wear or break over the duration of treatment.\(^5\) Other potential complications with conventional provisional restorations are chemical injury from the presence of monomer residue, thermal injury from an exothermic reaction, and mechanical injury resulting from polymerization shrinkage.\(^9\) Computer-aided manufacturing processes have been introduced that allow for the fabrication of long-term provisional restorations which would eliminate some of these complications when fabricating provisional restorations.\(^12\) Provisional restorations milled from PMMA (poly methyl-methacrylate) blocks are routinely used by dentists and laboratories thanks to their increased durability, excellent fit, and improved esthetics, but face the limitations due to the difficulty of reusing the discarded materials and machining undercuts or inaccessible locations.\(^15\) Recently, investigators found that the accuracy of dental restorations fabricated using additive manufacturing methods is higher than that of subtractive methods.\(^16\) The internal and marginal fit of 3D printed provisional restorations was also found to be comparable to that of crowns fabricated using a milling method, thus, making the 3D printing of provisional restorations a viable alternative.\(^17\) For the mechanical properties of 3D printed provisional restorations, studies showed that they had a comparable modulus of elasticity to conventional PMMA crowns and provided adequate wear resistance, suggesting that 3-D printed crowns possess sufficient mechanical properties for use as provisional restorations.\(^20,21\) However, the repair or reline of the 3D printed provisionals still remains a challenge.

Dental composite resins (Filtek Bulk Fill, 3M ESPE) are one of the materials used in the repair of provisional restorations. These materials are mainly composed of Bis-GMA (2,2-bis[4-(2-hydroxy-3-methacryloyloxypoxy)phenyl]propane), Bis-EMA (2,2-Bis[4-methacryloxypropylphenyl]propane), Procrylat (2,2-bis[4-(3-methacryloxypropoxy)phenyl]propane) and UDMA (urethane dimethacrylate). The major filler is zirconia/silica with particle size range of 0.01–3.5 \(\mu\)m. Bis-acrylic resins (Protemp Plus, 3M ESPE) are commonly used for the fabrication and repair of provisional restorations and are composed of bis-GMA, and a second functionalized dimethacrylate resin. Silanated zirconia-silica and fumed silica fillers are used to impart physical strength, radiopacity, and wear resistance.\(^22\) There have been many studies on the use of flowable composite resins, bis-acryl, PMMA materials or other surface treatments to improve the repair of conventional provisional materials,\(^23–27\) however, there exists a void in the literature when it comes to the materials of choice when repairing 3-D printed resin-based restorations. The objective of this in-vitro study was to assess the shear bond strength of the various resin-based repair materials to 3-D printed resin, and qualitatively assess the fractured surface patterns.

Materials and methods

All the base cylinders used in this study were designed on a CAD software, Autodesk MeshMixer (Autodesk, San Rafael, CA, USA) with specific dimensions.

Control group preparation

The cylinder for Group A (control) \((n = 15)\) was designed as a single combined form of a Ø6.8 \(\times\) 8 mm base cylinder and a Ø3 \(\times\) 5 mm upper cylinder, positioned in the center of the upper surface of the base cylinder (Fig. 1A). The designed

![Fig. 1 Specimens for shear bond strength test. (A) Control (Group A): base cylinder Ø6.8 × 8 mm with Ø3 × 5 mm cylindrical block in the center. (B) Test groups (Groups B, C and D): base cylinder Ø6.8 × 8 mm. The circle represents the testing surface for the repair materials to be bonded by a Ø3 × 5 mm upper cylindrical block.](image-url)
cylinders were fabricated using a stereolithography (SLA)-based 3D printer, VIDA (EnvisionTEC, Dearborn, MI, USA), with the layer thickness of 73 μm, the resin color A2, and 90-degree build orientation. Once the printing was complete, the cylinders were soaked in 99% isopropyl alcohol for 60 s, the cylinders were then sprayed completely dry with compressed air. The cylinders were post-cured under UV light for 15 s.

Test group preparation

The cylinders for test groups \( (n = 45) \) were designed with the same dimension as only the base cylinder \((Ø6.8 \times 8 \text{mm})\) (Fig. 1B). The fabrication of the base cylinders followed the same process as the control group preparation. The testing surfaces for the repair materials were roughened with airborne particle abrasion \((30-μ\text{m }\text{Al}_2\text{O}_3 \text{ particles})\) at 2.5 bar pressure for 5 s. The cylinders were ultrasonically cleaned to remove any trapped residue.

The prepared base cylinders for test groups were then divided into three different groups and bonded with the various repair materials as follows: Group B \((n = 15)\): A \(Ø3 \times 5 \text{mm} \) upper cylindrical block of PMMA bonded to the base cylinder of \(6.8 \times 8\text{mm}\). Group C \((n = 15)\): A \(Ø3 \times 5 \text{mm} \) upper cylindrical block of Bis-Acryl composite bonded to the base cylinder of \(6.8 \times 8\text{mm}\). Group D \((n = 15)\): A \(Ø3 \times 5 \text{mm} \) upper cylindrical block of Bis-GMA composite bonded to the base cylinder of \(Ø6.8 \times 8\text{mm} \) (Fig. 2).

For Group B, the testing surface of the base cylinder were initially wetted with monomer using a brush. The PMMA, Alike (GC America, Alsip, IL, USA), powder and monomer liquid were mixed in a dappen dish using a 1:3 ratio by volume. The mixture was stirred rapidly with a cement spatula for 10–15 s. The acrylic mixture was placed in a monoject syringe and injected through the opening of the dispensing mold \((Ø3 \times 5 \text{mm} \text{ upper cylindrical block})\). The acrylic was allowed to set according to manufacturer’s instructions.

For Group C, Bis-Acryl material, Protemp Plus (3M ESPE, St. Paul, MN, USA), was dispensed into the mold \((Ø3 \times 5 \text{mm} \text{ upper cylindrical block})\) and allowed to set for 2 min 30 s after onset of mixing.

For Group D, two consecutive coats of adhesive, Adper Single Bond Plus (3M ESPE), were applied on the test surface of the base cylinder for 15 s, and then gently dried with compressed air for 5 s. The bonding agent was light-cured for 10 s. A Bis-GMA composite resin, Filtek Bulk Fill (3M ESPE), was dispensed into the mold \((Ø3 \times 5 \text{mm} \text{ upper cylindrical block})\) and cured by exposure to LED light with a minimum intensity of 550 mW/cm² in the 400–500 nm range for 40 s.

Shear bond strength test

All specimens were placed in a customized stainless-steel holder and mounted on a jig in a universal testing machine, Instron 3345 (Instron, Norwood, MA, USA), at a crosshead speed of 0.5 mm/min (Fig. 2). The load was applied directly at the interface between the 3D printed base cylinder and the resin-based repair cylinder at 0.5 mm/min crosshead speed until fracture. The shear bond strength (MPa) was calculated by dividing the fracture load (N) by the area of the bonded interface (mm²). After the test, two representative failed specimens were
ultrasonically cleaned and platinum-coated. Each fractured surface was examined by field emission scanning electron microscopy, FE-SEM S4700 (Hitachi, Tokyo, Japan), for the fractography analysis.

Statistical analysis

The fracture modes were classified as follows: cohesive in the base cylinder, adhesive at the bonded interface, and mixed. A Shapiro–Wilk test was used to assess normality within the data, then the data was statistically analyzed by a Mann–Whitney test.

Results

The results of the shear bond strength testing are presented (Fig. 3). The mean shear bond strength and standard deviation of the control group A was 278.80 ± 10.28N and the mean value was the highest compared to other testing groups. The test groups exhibited mean shear bond strength values of 102.19 ± 18.05N for group B, 116.74 ± 38.95N for group C and 95.30 ± 17.82N for group D. The statistical analysis in Table 1 demonstrates a statistically significant difference when comparing control group A to the other groups (B, C, and D). However, no statistical differences were found when comparing the test groups to each other (B vs C, B vs D, C vs D). Based on the microscopic examination (Fig. 4), all specimens (100%) of group A showed cohesive failures at the fractured surface within the base cylinder area. Most of the specimens in group B (87%) displayed mixed failures, while several specimens (13%) showed adhesive failure modes at the fractured surface. The specimens of group C showed both mixed failure (60%) and adhesive failure (40%) at the fractured surfaces. All specimens (100%) of group D showed mixed fracture patterns, with partly cohesive failure within the base cylinder area and partly adhesive failure at the bonded interface (Table 2).

Discussion

Current 3-D printing technology allows for the very accurate fabrication of complex dental lab work such as orthodontic appliances, surgical guides, dental casts, occlusal guards and RPD frameworks.12–14,18 The advantages of additive manufacturing include flexibility in printing multiple specimen at once, and passivity. It also overcomes the disadvantages of subtractive methods like milling CoCr that would result in wear, noise and heat. In addition, additive manufacturing results in a low percentage of wasted raw

| Comparison | P value |
|------------|---------|
| A vs B     | <0.001* |
| A vs C     | <0.001* |
| A vs D     | <0.001* |
| B vs C     | 0.14    |
| B vs D     | 0.08    |
| C vs D     | 0.07    |

Statistical analysis using the Mann–Whitney Test, *P < 0.05.
Unit: Newton; Group A (Control); Group B (PMMA); Group C (Bis-Acryl composite); Group D (Bis-GMA composite).

Fig. 3 Box plots of the maximum, minimum, mean, and standard deviation of shear bond strength of provisional repair materials bonded to 3D printed resin for the four groups. The unit is Newton. Group A (Control); Group B (PMMA); Group C (Bis-Acryl composite); Group D (Bis-GMA composite). "54" indicates an outlier in Group D.
materials, whereas subtractive manufacturing can waste as much as 96% of the block material.\textsuperscript{17}

One of the challenges of provisional materials is its low mechanical strength compared to the definitive materials, and there have been many studies which have investigated its mechanical strengths and proposed ways to improve its properties.\textsuperscript{6–8,23,27,28} Another challenge is repairability. In many instances, provisional restorations require a repair of fractured connectors or a modification to add contact points, refine marginal adaptation, or modify contours. A few studies have shown methods to improve the repair or modification of provisional materials and have investigated its outcome.\textsuperscript{23–26} However, there is a void in the literature regarding the protocols and materials that could be used to repair 3-D printed provisional restorations. These unknowns may limit the use of the 3-D printed resins even though the materials have the aforementioned advantages over the traditional materials used for the fabrication of provisional restorations.

In this study, the shear bond strength of various repair materials bonded to 3-D printed resin was compared to that of the fully intact 3-D printed resin, itself, in order to explore potential protocols for the repair of printed provisional restorations. The samples were designed to have the same dimensions in all groups and the repair process of the testing groups followed the standard protocols provided by manufacturer’s instructions. The group A served as a control with the 3-D printed monolithic resin which was compared to the various repair materials. The results showed a marked statistical difference in shear bond strength between the control (Group A) and the test groups (Group B, C and D). This indicated that the repair materials were not able to produce the same strength as that of 3D printed monolithic resin. Regardless of the material used, a repaired restoration would not be as robust as a fully intact

\begin{table}
\centering
\caption{Qualitative analyses of fracture patterns under the scanning electron microscope.}
\begin{tabular}{ccc}
\hline
 & Cohesive & Adhesive & Mixed \\
\hline
Group A & 100\% & & \\
Group B & 13\% & 87\% & \\
Group C & 40\% & 60\% & \\
Group D & 100\% & & \\
\hline
\end{tabular}
\end{table}

\textsuperscript{Group A (Control); Group B (PMMA); Group C (Bis-Acrylic composite); Group D (Bis-GMA composite).}

\begin{figure}
\centering
\includegraphics[width=\textwidth]{fracture_patterns}
\caption{Fracture patterns analyzed under the scanning electron microscope. (A) Group A specimen demonstrating cohesive fracture. (B) Group B specimen showing adhesive fracture. (C) Group B specimen exhibiting mixed cohesive and adhesive fracture. (D) Group C specimen demonstrating adhesive fracture. (E) Group C specimen showing mixed cohesive and adhesive fracture. (F) Group D specimen exhibiting mixed cohesive and adhesive fracture.}
\end{figure}
printed restoration. Therefore, the bonding of the repair materials to the 3-D printed resin is not predictable enough to be used as a long-term solution or to replace the remake of significantly fractured provisional restorations.

Comparisons between the test groups showed no statistical difference between the shear bond strength of PMMA, Bis-acryl or Bis-GMA composite to 3D printed resin. The results did not show a preferential choice of materials with which to repair 3D printed provisional resin from a statistical standpoint. However, the fractography analysis showed a majority of the specimens in Groups B and D exhibiting a mixed fracture type with cohesive in the base cylinder area and adhesive in the repaired cylinder area. Group C (Bis-Acryl composite resin) showed a wide distribution of bonding strength values, which can be attributed to the fact that 40% of the samples showed an adhesive fracture pattern. On the other hand, 100% of the Group D (Bis-GMA composite resin) specimens showed a mixed fracture type. This indicates that the repair with Bis-GMA composite resin provides a more consistent bonding effect when compared to other repair materials even though there was no statistical difference in the bonding strength among the sample groups. This finding is also reflected in the results of the bond strength testing that shows less variability in the values for Group D.

According to the results of this study, group D (Bis-GMA composite resin) appears to be the most predictable repair material for 3D printed resin when compared to Bis-Acryl and PMMA. However, it is unknown if this is due to the application of the bonding agent or the properties of the Bis-GMA material itself. When assessing the dimensions of the test specimens, the surface used for bonding the test materials to the 3D printed cylinder was 3 mm in diameter, which equates to about a 7 mm² surface area for bonding. This is a clinically realistic dimension when considering the scenarios where a repair of a provisional restoration could be indicated, such as when adding an occlusal or interproximal contact point, modifying contours and refining marginal adaptation. The results suggest that the use of the repair materials tested are not as predictable as remaking the provisional restorations, however, minor refinements and adjustments can be effectively performed using Bis-GMA composite resin following the manufacturer’s instructions. It can be assumed that a larger surface area for bonding will result in a more robust connection between the printed resin and the repair material.

Due to the inherent limitations of an in vitro study, this study may not show a direct correlation when applied in a clinical setting. However, the study shows the relative bond strength measurements for different repair methods in a standardized setting. Further studies in a clinical setting will aid in deciding a choice for repair material and improving the repair process for 3D printed provisional restorations.

In conclusion, the fracture strength of a monolithic 3D printed resin is significantly higher than the bond strength of the repair materials tested. There was no statistically significant difference in the bond strengths of the tested materials to the 3D printed resin. However, Bis-GMA composite resin demonstrated most consistency from the fractography analysis. Within the limitations of this in-vitro study, it can be concluded that minor repairs or modifications of 3D printed provisional restorations appear to be feasible with the tested materials. A remake of the provisional restoration is recommended if major repair is needed.

Declaration of Competing Interest

The authors deny any conflicts of interest related to the present study.

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