Repair Shear Bond Strength of Aged Provisional 3D-Printed Resin: Role of Repair Materials

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Abstract

This study compared the shear bond strength of aged provisional 3D-printed resin repaired with different materials. Forty specimens (n=10) were fabricated from 3D-printed resin using a digital light processing (DLP) printer. The control (no repair) was printed as a single unit composed of cylindrical base (∅20×15 mm) and cylindrical block (∅5×3 mm) at the center.

In the test groups, specimens were fabricated as a cylinder with the same dimension (∅20×15 mm). All specimens were aged by thermocycling and specimens in the tested groups were divided into 3 subgroups based on repair materials: polymethylmethacrylate (PMMA), bis-acryl composite and flowable composite. Then, specimens were sandblasted and repaired following manufacturer's instructions. After 24 hours in 37°C distilled water, the shear bond strength testing was performed, and fracture surfaces were examined using stereomicroscopy (40×). Data were analyzed using one-way ANOVA and Tukey's test (α=0.05).

The highest shear bond strength was 20.08±0.84 MPa (positive control) followed by 14.35±1.49 MPa (flowable composite), 12.19±0.92 MPa (PMMA) and 10.78±2.34 MPa (bis-acryl composite), respectively. Statistical analysis showed significantly different in shear bond strength among groups except those of PMMA and bis-acryl composite which were comparable (p>0.05).

Cohesive failure was predominantly found. Flowable composite is a material of choice to repair aged provisional 3D-printed resin because of its highest repair shear bond strength compared to PMMA and bis-acryl composite.

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Introduction

Provisional fixed restorations are intended to be used for a limited time during prosthodontic treatment. They serve several important purposes such as esthetics, function, pulpal and periodontal protection, prevention of abutment migration, and being used as a diagnostic tool to evaluate occlusion, vertical dimension, and maxillo-mandibular relation particularly in extensive occlusal reconstruction cases.[1] They are subjected to occlusal load and thermal change in the oral cavity during function and may require to be relined or repaired when they fracture or wear down.

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According to their composition, provisional materials can be classified into 2 groups: monomethacrylates or acrylic resins and the dimethacrylates or bis-acryl composite.[2] The monomethacrylates include polymethylmethacrylate (PMMA) and polyethyl/butyl methacrylate (PEMA). PMMA is widely used as a provisional material because of its acceptable strength, short-term color stability, biocompatibility, ease of reline/repair and adjustment. However, it requires extensive chair side adjustment due to the polymerization shrinkage of PMMA and its mechanical properties reduced overtime because of internal porosity causing water sorption, discoloration and eventually failure of the restoration.[3,4] Moreover, exothermal heat during polymerization and residual monomer are the concern of this material.[5]

Bis-acryl composite is composed of functional monomers such as bisphenol A-glycidyl dimethacrylate (Bis-GMA), urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA) that crosslinks to form polymer network. Bis-acryl composite has superior strength, color stability and polish ability as well as low polymerization shrinkage and exothermal heat relative to PMMA. It is commercially available as an automix system that potentially reduces voids and porosity of provisional restorations.[1]

Currently, computer - aided design/computer - aided manufacturing (CAD/CAM) technology has become a new method for fabrication of provisional prostheses because it can overcome the drawback of the conventional technique such as better flexural strength, color stability, marginal adaptation.[6] CAD/CAM technology can be either subtractive/milling or additive manufacturing/3D-printing technique. Additive manufacturing or 3D-printing generates a three-dimensional object by stacking materials layer by layer. This technique reduces waste materials by printing only as much as needed of the end-product and allows the creation of more complex geometries.[7] In terms of accuracy, studies reported the comparable or even better marginal fit of the 3D-printing provisional crowns compared the milling ones.[8,9] Additionally, elastic modulus of 3D-printed provisional resin was comparable to conventional Jet acrylic suggesting its suitable for intraoral use.[10] Dental applications of 3D-printing include fabrication of working models, surgical guides, occlusal splints, custom tray, fixed and removable prostheses such as artificial teeth and denture base resin.[11-13]

Despite the various benefits, provisional 3D-printed prostheses still require chairside customization because they are prefabricated before the abutment is prepared. Chairside relining is necessary especially at the finishing line for good marginal adaptation. In addition, when provisional fixed prostheses have been used in the oral cavity and undergone thermal and functional stress, fracture or chipping commonly occurs especially the long-term use such as the oral rehabilitation cases. Repairing the restoration is the first choice for minor issues because it is economical and less time-consuming.[14]

Despite several reports on repair bond strength and protocols for provisional 3D-printed resin, most studies have performed non-aging condition.[7,15-17] Thus, selecting materials of choice for repairing aged 3D-printed resin is inconclusive. Therefore, the purpose of this in vitro study was to evaluate the effect of repair materials on shear bond strength (SBS) of aged 3D-printed resin. The null hypothesis tested was that repair shear bond strength of aged 3D-printed resin are comparable regardless of repair materials.

Materials and methods

Specimen fabrication

Forty cylindrical specimens (n=10) were designed (3D Builder version 18.0.1931.3, Microsoft Corporation, Washington, USA) and printed (ASIGA® 3D Resin DentaTOOTH, shade A2) using a digital light processing (DLP) resin-based 3D printer (ASIGA composer, Sydney, Australia) with a 50 µm slice layer and a 90-degree build orientation. Then, specimens were immersed in 99% isopropyl alcohol for 60 seconds, ultrasonically cleaned (GT SONIC-D3, China) for 5 minutes to remove retained residue, dried using compressed air and post-cured under UV light for 200 seconds for the final polymerization in a light chamber (HiLite power 3D, KULZER, Australia). All specimens were examined under a stereomicroscope (10×) and the ones with cracks or defects were excluded.

There are 4 groups in this study: a positive control and 3 test groups (polymethylmethacrylate/PMMA, bis-acryl composite and flowable composite)

Positive control group

Specimens were printed as a cylindrical base (20×15 mm, diameter×height) with a small cylindrical block (5×3 mm) at the center (Figure 1A). These intact monolithic specimens were designed to measure the cohesive strength (no repair) of the 3D-printed material.

Test groups

Specimens in the test groups were printed as a cylinder with the dimension of 20×15 mm (diameter×height, Figure 1B).

Aging and repair protocols

Prior to repair, all specimens were aged by thermocycling (5°C to 55°C, dwell time of 30 seconds, transfer time of 5 seconds) for 1,500 cycles. After aging, specimens were sandblasted (Aeroetcher Abrasive Blaster, USA) for 10 seconds with 50 µm Al_2O_3 particles with an air pressure of 2 bars and 10 mm from specimen's surface. Then, specimens were cleaned with water for 10 seconds and dried with oil-free compressed air for 10 seconds. Sandblasted specimens were randomly divided into 3 additional groups according to repair materials: PMMA, bis-acryl composite and flowable composite.

Specimens were subsequently repaired with bonded surface area of 5×3 mm (diameter×height). To control the bonded surface area, a polyethylene tape (3×0.1 mm) was placed at the center of the specimen surfaces. A detachable teflon mold with an internal diameter of 5×3 mm is placed on the bonded surface. Repair material was injected into the mold and polymerized following manufacturer's instruction as follows:

PMMA (GC UNIFAST Trad, Alsip, IL, USA): the bonding surface was initially wetted with monomer using a microbrush. PMMA was mixed for 10-15 seconds using a 1:3 ratio (powder: liquid) by volume. The mixture was injected into teflon mold with a monoject syringe and allowed to polymerize for 2 minutes.

Bis-acryl composite (Protemp 4, 3M ESPE, St. Paul, MN, USA): the material was dispensed from a cartridge in a dispensing gun through a mixing tip into the Teflon mold and allowed to polymerize for 2.5 minutes.

Flowable composite (Filtek Supreme XT Flowable Restorative, 3 M ESPE, ST. Paul, MN, USA): the material was injected into the Teflon mold and light-cured for 20 seconds using LED light (Mini LED™ Standard F, ACTEON, Norwich, UK) with a minimum intensity of 1,250 mW/cm^2.

After polymerization, polyethylene tapes and teflon molds were removed. Repair specimens were stored at 37°C in distilled water for 24 hours prior to SBS testing.

Shear bond strength (SBS) testing

All specimens were mounted in a universal testing machine (Instron, ElectroPuls™ E1000, England) and subjected to shear load with a knife-edge shear blade at a crosshead speed of 0.5 mm/min until fracture. SBS (in MPa) was calculated by dividing the maximum load (N) by the bonded area (mm^2).

Failure mode analysis

Fracture surfaces were examined using a stereomicroscope (Olympus SZX16; Tokyo, Japan) at 40× magnification. Modes of failure are classified as adhesive (between the 3D-printed resin and repair material), cohesive (within the 3D-printed resin or within the repair materials), or mixed (combination of adhesive and cohesive failures).

Scanning electron microscopy (SEM) analysis

Representative failure modes were sputter coated with gold in a vacuum cold sputter (SPI Sputter Coater, SPI Supplies, PA, USA). SEM images were obtained at 20× magnification using a scanning electron microscope (SEM) (LEO1455VP, Angstrom Scientific, England).

Statistical analysis

Data were analyzed using one-way ANOVA and multiple comparisons were performed using Tukey's HSD test. The level of significance is set at $p<0.05$.

Repair materials	Shear bond strength (MPa)	Percent relative to the control
Positive control (no repair)	20.08 ± 0.84^{A}	100
PMMA	12.19 ± 0.92^{B}	60.71
Bis-acryl composite	10.78 ± 2.34^{B}	54.69
Flowable composite	14.35 ±1.49^{C}	71.48

Table 1. Mean shear bond strength (SBS, MPa), standard deviations of aged 3D-printed resin repaired with different materials, and SBS compared to the positive control (%).
Different superscript letters indicate significant difference among groups ($p<0.05$). Positive control was the cohesive strength of 3D-printed resin fabricated as the same dimension as those used in the repair groups. Abbreviation: Bis-GMA: bisphenol A-glycidyl methacrylate; UDMA: Urethane dimethacrylate; Bis-EMA: bisphenol A ethoxylated dimethacrylate TEGDMA: triethylene glycol dimethacrylate.

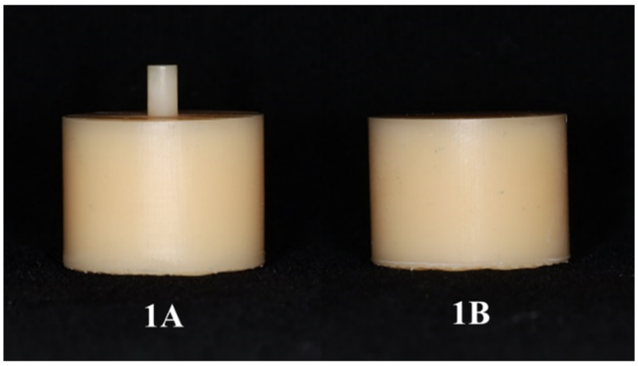

Figure 1. Specimens of the positive control (A) and test groups (B).

Results

Table 1 showed mean SBS (MPa) and standard deviation of the positive control and test groups. The highest SBS was 20.08±0.84 MPa (positive control) followed by 14.35±1.49 MPa (flowable composite), 12.19±0.92 MPa (PMMA) and 10.78±2.34 MPa (bis-acryl composite). One-way ANOVA showed significant difference in SBS among groups of study except SBS of PMMA and bis-acryl composite which did not significantly different (p>0.05).

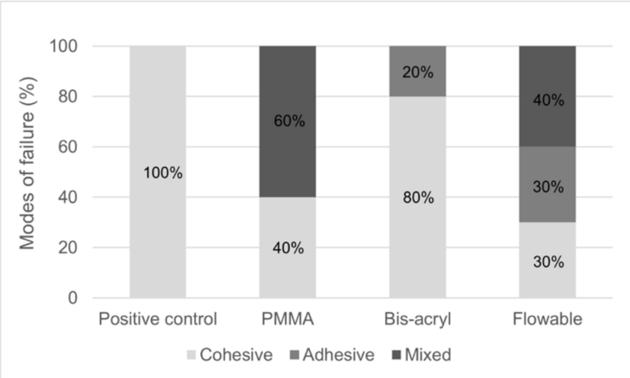

Figure 2. Modes of failure (%) examined using a stereomicroscope (40×).

As shown in Figure 2, cohesive failure (80%) and mixed failure (60%) were mostly found in aged 3D-printed resin repaired with bis-acryl composite and PMMA, respectively. Modes of failure of that repaired with flowable composite were approximately equal. Failure in the positive control (no repair) was classified as cohesive failure.

Representative scanning electron microscope micrographs of fractured surfaces classified as cohesive, adhesive and mixed failures are shown in Figure 3.

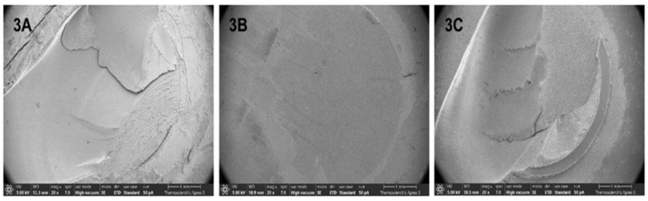

Figure 3. Representative SEM images of failure modes at 20× magnification. 3A: cohesive failure (PMMA), 3B: adhesive failure (bis-acryl composite) and 3C: mixed failure (flowable composite).

Discussion

Currently, 3D-printed resin has become an alternative option for fabrication of provisional restorations. Although it poses multiple advantages over conventional auto-polymerized materials such as higher degree of conversion, low water absorption/solubility, and high mechanical properties, provisional materials have inferior mechanical properties compared to definitive materials.[18] The need for reline or repair for marginal adaptation, fracture repair, or contouring of restoration, unavoidably occurs particularly in the case when long term use is necessary and materials is aged and subjected to masticatory forces. For this reason, this study focused only on aged 3D-printed resin when repaired with different materials. One-way ANOVA showed significant differences in repair SBS among groups of study. Therefore, null hypothesis is rejected.

The positive control was selected as intact monolithic 3D-printed specimens (Figure 1A) without the bond interface compared to the other groups. It is intended to measure the cohesive strength of the aged specimen with the same dimension and subjected to load as occurred in the test groups. In addition, this also allows us to compare the SBS between the repaired groups and the intact monolithic group. SBS of the bis-acryl composite, PMMA and flowable composite groups was only 54.69%, 60.71% and 71.48%, respectively, compared to the positive control (Table 1) which considered as 100% (p<0.05). These results indicated that the repair specimens could not reach similar strength as that of aged intact monolithic 3D-printed resin regardless of repair materials used. Therefore, repair provisional restoration may not be the best option for a very large fracture. Making a new restoration would receive a more favorable outcome.

In this study, the highest SBS was observed when flowable composite was used as a repair material, followed by PMMA and bis-acryl composite which were comparable (p>0.05). This suggested that the flowable composite is a material of choice for repairing aged 3D-printed resin. However, Albahri et al. reported contradictory results.[15] They compared repair SBS of provisional 3D-printed resin showed no favorable material of choice (p>0.05) among PMMA, bis-acryl composite and bulk fill composite. The contrasting results are possibly due to artificial aging which was not performed in their study. Aging by thermocycling accelerates hydrolysis of polymer and repetitive contraction and expansion induce stress causing crack and deterioration of the surface.[19] Furthermore, the number of unreacted monomers affects the repairing potential of the substrate through chemical bonding.[20] Unreacted monomers diminish after thermocycling resulting in low chemical bonding potential of the aged substrate.[21] For this reason, repair materials would have no significant effect on SBS of non-aged 3D-printed resin as observed in Albahri et al.'s study.

Artificial aging by thermocycling was used to simulate intraoral condition prior to repair. It was reported that 10,000 cycles of thermocycling were approximately 12 months of physiological aging in the oral cavity.[19,22] However, specimens were aged only 1500 cycles (approximately 7 weeks in service). This number was chosen because in the extensive occlusal reconstruction, the provisional restoration is suggested to be used at least 6-8 weeks.[23] Results of this study showed significant difference among groups indicating that aging (even only for 1500 cycles) had negative effect on repair bond strength compared to Albahri's study which did not demonstrate the difference in non-aging condition. Therefore, the longer aging time would make materials more difficult to repair or the repair bond strength might not be as durable as that of the freshly fabricated one.

Regarding the fracture surface analysis, mixed and cohesive failures were the most common. Only 5 specimens in all test groups were classified as adhesive failure. Other studies also found similar results.[15,24,25] This finding is associated with sandblasting technique that creates a strong adhesion at the repairing interface causing predominantly mixed and cohesive failures. Interestingly, all cohesive failure occurred only within the aged 3D-printed resin. Lim el al.[7] reported similar results as all cohesive failure occurred within 3D-printed resin. Artificial aging deteriorates 3D-printed resin making it vulnerable to fracture.

To investigate the sole effect of repair materials on SBS, sandblasting with aluminum oxide was selected as the only surface treatment, other mechanical surface treatment methods such as grinding, application of adhesive were excluded because studies reported increased repair bond strength with sandblasting compared to other surface treatments.[24,26,27] Sandblasting produces micro-retentive features through increased roughness and bonding area, and it also removes impurities creating high surface wettability which is favorable for repairing.[28-30]

It is unknown why flowable composite produced higher SBS relative to PMMA and bis-acryl composite since all specimens were aged and sandblasted in the same manner prior to repair. Two possible explanations may be its viscosity and curing modes that promote flowable composite to flow into surface pits and depressions. Sandblasting with aluminum oxide creates surface roughness (Ra) approximately of 1.36-2.23 μm.[31] Papacchini et al. found that the repair SBS increased when the viscosity decreased[32] because repair materials need to flow and closely contact for micromechanical interlocking. Flowable composite exhibits "shear-thinning" behavior in which its viscosity decreases with increased shear rate[33] allowing material to be injected easily and flow into the sandblasted surface. The longer working time of light cured flowable composite rather than the chemical cured PMMA and bis-acryl composite affects the ability of materials to penetrate the sandblasted surface because chemical cured PMMA, and bis-acryl composite gradually thicken overtime. The combined effects of "shear-thinning" behavior and light cured materials would make flowable composite favorable to micromechanical retention which results in higher SBS.

The limitation of this study is not only related to the *in vitro* study which could not simulate actual oral condition such as intraoral moisture, pH, repetitive stress and masticatory force, roles of different surface treatment or combined with bonding agent and the durability of repair bond strength is unknown. These drawbacks could be evaluated in future studies.

Conclusions

Flowable composite is a material of choice to repair aged 3D-printed resin following sandblasting because it provided the highest SBS compared to PMMA and bis-acryl composite.

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Declaration of Interest

The authors report no conflict of interest.

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