

REPAIR BOND STRENGTH OF 3-D PRINTED LONG-TERM PROVISIONAL MATERIAL: THE EFFECT OF SURFACE ROUGHENING METHOD AND COMPOSITE RESIN MATERIAL

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ABSTRACT

Objective: This study was designed to assess the shear bond strength of 3D printed Polymethylmethacrylate (PMMA) material to two different types of repair composite resin materials following two different surface roughening techniques.

Materials and methods: Thirty-six discs were 3D printed from PMMA liquid resin, aged and then allocated to 3 groups (n=12 in each) according to the surface roughening method: Group P: As printed and untreated, Group G: discs were grinded using diamond stone and Group A: discs were air abraded by 53 um alumina particles. Universal adhesive was applied, and light cured. Then, half of the discs was bonded to Flowable composite resin, and the other half was bonded to injectable composite resin according to the standards of shear bond strength test (SBS). All discs were stored in distilled water for 7 days and then SBS test was carried out using a universal testing machine. The mode of failure was observed using a digital stereomicroscope. All calculated data was sent for statistical analysis.

Results: Grinded PMMA discs repaired with Injectable composite showed the highest statistically significant shear bond strength value (21.32 ±1.71) MPa in comparison to untreated (9.52 ±1.86) MPa and air-abraded ones (14.05±4.47) MPa and also higher than Grinded PMMA repaired with flowable composite (11.87 ±4.16) MPa.

Conclusions: It is attainable to establish an acceptable bond with PMMA. Grinded PMMA had a greatly enhanced repair bond strength to injectable composite resin.

KEYWORDS: PMMA, Injectable Composite, Flowable Composite, Abrasion, Grinding.

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INTRODUCTION

Provisional restorations are extremely important from a biological, mechanical, and esthetic standpoint. They serve a variety of purposes, including safeguarding the dentin-pulp complex, maintaining positional stability, and providing functional occlusion. Before the final prosthetic restoration is carried out, they provide the patient the opportunity to confirm the outcome's acceptability from a cosmetic perspective⁽¹⁾. Although temporary restorations are typically needed for few days, in some cases, long-term temporary restorations are mandatory. These cases include full occlusal reconstruction and dental implant treatments^(2,3).

The most widely used materials for the fabrication of interim restorations are various types of self- or light-curing acrylic resins, including polymethyl methacrylate (PMMA) resin, bis-acryl composite resin, and visible light-cured urethane methacrylates⁽⁴⁾. Modern polymeric materials, such as CAD/CAM blocks made of PMMA, can be machined into temporary restorations. In comparison to traditionally polymerized restorations, those milled from PMMA-based CAD/CAM blocks exhibit better mechanical characteristics, a wider range of translucency, and a higher resistance to discoloration⁽⁵⁾. However, they are constrained by the challenges of recycling the waste materials and milling undercuts or inaccessible areas. Researchers recently discovered that additive manufacturing techniques, such as 3D printing, provide dental restorations with more accuracy than subtractive techniques^(6,7). The mechanical qualities of 3-D printed crowns are sufficiently adequate for use as temporary restorations⁽⁸⁾. A repair of broken connectors or adjustments to add contact points, maximize marginal adaptability, or alter contours are frequently needed for provisional restorations⁽¹⁾. The clinical ease of repairability of these materials should be taken into consideration, as well as the durability of the repaired interface.

Literature offers multiple repair protocols for PMMA restorative material. Protocols include the surface treatment of the repaired surface, the type of bonding agent, and the repair material itself. Surface roughening is mentioned repeatedly in the literature. Evidence-based methods of roughening include grinding with abrasive stones, sandblasting, methyl methacrylate application and silica-coating^(9,10). Some authors investigated the use of adhesive or lack of adhesive during the repair process⁽¹¹⁾. Other studies were concerned with the repair material itself. Among the most commonly used repair materials is the flowable composite due to its affordability and ease of use^(11,12).

Recently, injectable composite resin materials have been introduced to the market. Injectable composite resin materials are low-viscosity resin-based composites. Unlike conventional flowable composite resin materials, injectable composite resin materials provide sufficient mechanical properties and surface wear resistance due to their high filler loading. They also display an increased wettability and improved adaptability to the substrate. This allows less instrumentation to the substrate, which is considered an advantage in clinical settings⁽¹³⁾.

There is a gap of knowledge in the literature - to date - to provide a consensus about the optimal repair protocol and material for PMMA restoration. Therefore, this study is designed to assess the shear bond strength of Polymethylmethacrylate (PMMA) long-term temporary restorations to two types of composite resin material following two surface roughening techniques. The null hypothesis stated that neither the surface roughening method nor the composite resin material type would affect the repair bond strength of PMMA provisional material.

MATERIALS AND METHODS

Sample Size determination

A power analysis was created to have sufficient power to apply a statistical test of the null

hypothesis, which stated that there is no difference in shear bond strength between the tested groups. The envisioned total sample size (n) has been identified to be (36) samples, with 18 samples per group and 6 samples per subgroup, based on the adoption of an alpha (α) level of 0.05 (5%), a beta (β) level of (0.2) (i.e., power=80%), and effect size (f) of (0.653). The sample size calculation was based on a previous study by *El Bahri et al*⁽¹⁾ utilizing G*Power 3.1.9.7⁽¹⁴⁾.

Ethical approval:

The Faculty of Dentistry at Ain Shams University's Research Ethics Committee authorized this investigation's methodology. FDASU-RecPC072338 was the approval code.

Samples 3D printing:

A 3D printer (EPAX 3D, North Carolina, USA) was used to manufacture 36 discs (3 mm thickness x12 diameter) with fluid PMMA resin (Next Dent C&B, Zetterberg, Netherlands). A standard tessellation language (STL) file (3Shape Cambridge, Copenhagen K Denmark) was used for constructing the disc-shaped specimens with supporting components. The printer was instructed to begin printing in a vertical position with successive layers that were approximately 100 μ m thick. A partially cured 3D-printed disc was created. The printed discs were cleaned with isopropyl alcohol before being cured for an additional 30 minutes in an ultraviolet lightbox (Bredent, Bre. Lux power unit 2, Germany).

Thermocycling was performed for all samples using a Robota thermocycler (Alexandria, Egypt) for 5000 cycles at temperatures ranging from 5 to 55 degrees Celsius with dwell times of 30 seconds in each bath and 20-second breaks between baths at room temperature. Each disc was then mounted in an acrylic base to help in its attachment to the universal testing machine during testing.

Samples grouping

The discs were subsequently categorized into three groups based on the surface roughening technique:

- Group P (As printed): no roughening was applied, n= 12.
- Group G: grinding using abrasive diamond stone, n= 12.
- Group A: air abrasion using alumina particles, n= 12.

According to the kind of composite resin material utilized for the repair, each group was afterward divided into two subgroups:

Subgroup F: Flowable composite, n=18.

Subgroup I: Injectable composite, n=18.

Surface roughening methods:

Twelve discs (Group G) were grinded using a green-coded (coarse) diamond abrasive stone (TR13, ISO 198/018 standard 125-150 micrometer particle size, Mani Dia-Burs, Tochigi, Japan), mounted on a high-speed contra-angled hand-piece (NSK, Japan), under copious air/water spray. The grinding was performed by the same operator in one direction for only one stroke. The bur was changed every 3 discs.

Another twelve discs (Group A) were air abraded using 53 μ m aluminum oxide particles for 10 seconds, at 3 bar pressure and 10 mm distance by the same operator. Air abrasion was performed using an intraoral device (AquaCare, Velopex International⁰, London,UK). Prior to bonding methods, contaminants on the surface of the discs had been eliminated using an ultrasonic cleaner (Eumax @, Hong Kong, Model number: UD80SH-2.6L) for 10 minutes. The surface morphology following surface roughening had been observed using a Scanning Electron Microscope (Inspect S, FEI company, USA) at a 1000X magnification for one disc from each group.

Bonding procedures:

A universal adhesive (All Bond Universal, BISCO Schaumburg, USA) was used as a bonding agent in this study following the manufacturer's instructions. Two separate applications of adhesive were actively applied with a micro brush. Each coat was allowed to air dry for 10-15 seconds. Excess solvent within each coat was evaporated by air-drying with an air syringe for at least 10 seconds, till there is no visible movement of the adhesive. Transparent Polyvinyl tubes were sliced into small tubes, each had a 2 mm diameter and 2 mm height. Each tube was attached to the center of each sample, and then each sample was light-cured for 10 seconds with a light-curing device (DeepCure-L LED curing light, 3M™ Elipar™). The light output was $\approx 1470 \text{ mW/cm}^2$ (10%/+20%) as described by the manufacturer.

Tetric N Flow, a flowable composite (Ivoclar Vivadent, Germany) was injected into the tubes attached to half of the samples (PF, GF, and AF) via its dispensing tip until the tube was filled, while G-aenial Universal Injectable composite resin (GC, Japan) was injected into the other half (PI, GI, and AI). A 20-second light curing of the composite resin was done. A circle was drawn around each tube to indicate the area of bonding during the microscopic inspection following the shear bond strength test. To create a composite cylinder, the tubes were cut and removed using a sharp scalpel blade number 11. All the samples were kept for a week at 37 °C in an incubator with distilled water until tested for Shear Bond Strength (SBS).

Shear bond strength test:

To assess the shear bond strength, a circular interface shear test has been utilized. Each sample was placed separately on a computer-controlled materials testing device (Model 3345; Instron

Industrial Products, Norwood, USA) with a load-cell of 5 kN, and data were obtained using computer software (Bluehill Lite; Instron Instruments).

Each sample was attached to a custom-made holder and tightened to the lowest fixed compartment of the testing equipment. The shear test was performed by compressive mode of load applied at PMMA-Resin interface using a mono-beveled metallic rod in the shape of a chisel that was attached to the upper moveable compartment of the testing apparatus and moved at a crosshead speed of 0.5 mm/min. The load needed to break the bond was measured in Newtons.

Shear bond strength calculation:

The load at failure was divided by bonding area to express the bond strength in MPa:

$$\tau = P / \pi r^2$$

where ; τ =shear bond strength (MPa, P =load at failure(N)

$\pi = 3.14$ and r =radius of resin disc (mm)

Mode of failure analysis:

Samples were examined using a USB digital microscope with a 35X magnification (U500x Digital Microscope, Guangdong, China) after debonding. The camera (3 Mega Pixels resolution) was positioned vertically 2.5 cm away from the samples. About a 90° angle existed between the lens' axis and the sources of illumination. Images were captured at a resolution of 1280 x 1024 and then transmitted to an IBM-compatible computer running the Image-tool program (Image J 1.43U, National Institutes of Health, USA) in order to identify the failure mode pattern in accordance with the following categories: cohesive failure within the composite resin or PMMA, adhesive failure at the Resin/PMMA interface, and mixed adhesive/cohesive failure.

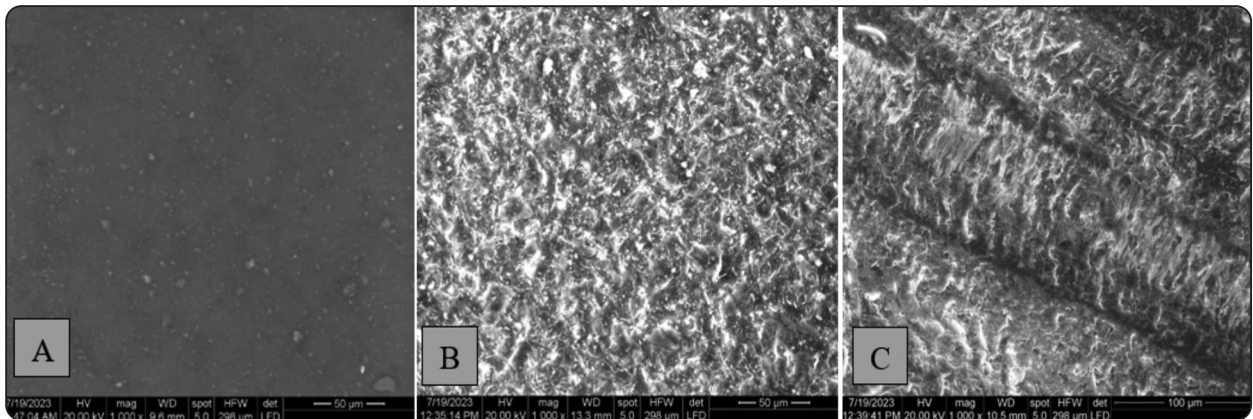


Fig (1): SEM images of As printed (A), air-abraded (B), and ground (C) PMMA at 1000X magnification.

RESULTS

a) Scanning Electron Microscope (SEM) assessment

As presented in Fig (1A), The surface of the untreated sample was relatively smooth. The air-abraded sample SEM image (Fig 1B) showed many irregularities while the samples grinded with diamond stone exhibited many evident oblique grooves, micropores and irregularities (Fig 1C).

b) Statistical analysis of shear bond strength

Numerical data was presented as mean and standard deviation (SD) values. To check for normality, the Shapiro-Wilk test was performed. There was evidence of parametric data. Using Levene's test, the homogeneity of variances was examined. The homogeneity assumption had been maintained and the data had a normal distribution. A two-way ANOVA was used to examine intergroup comparison. When significance was found, Tukey Post Hoc test was performed. For all tests, the significance level was set at $p < 0.05$. Gathered raw data were analyzed using IBM SPSS® program (IBM Corp. New York, USA), operated on Windows 8 (Microsoft Corporation, Washington, USA).

For the effect of the surface roughness methodology regardless of the composite resin type: it was revealed that: The highest SBS value

was for “Grinded group” (G) (16.6 ± 5.8) MPa which was statistically significant from “As printed” group (P) (9.9 ± 2.17) MPa, ($p = 0.002$). There was also statistically significant difference between the Air-abraded group (A) (14.26 ± 4.1) and “As printed” group ($p = 0.048$). However, there was no statistically significant difference between Air-abraded and Grinded groups ($p = 0.39$). Also, there was no statistically significant difference between Injectable (I) (14.96 ± 5.1) MPa and Flowable composite (F) (12.24 ± 3.84) MPa regardless of the surface roughening method used ($p = 0.1$).

The two-way interaction of variance showed that: when Injectable composite was used, the highest shear bond strength value was for the GI group (21.32 ± 1.71) MPa, which showed a statistically significant difference compared to AI group (14.05 ± 4.47) MPa, and PI group (9.52 ± 1.86) MPa. PI showed the lowest SBS values within all groups. There was no statistically significant difference between AI and PI groups as shown in table (1).

Also, when flowable composite was used, there was no statistically significant difference between all surface roughening methods. The SBS for AF, GF, and PF groups were (14.48 ± 4.03) MPa, (11.87 ± 4.16) MPa and (10.37 ± 2.55) MPa respectively. Within the same surface roughening method, there was no statistically significant difference between

injectable and flowable composites when used as repair material except for the GI group which demonstrated a higher statistically significant value than GF as shown in table (1).

c) Mode of failure:

All modes of failure were observed (Fig 2) and the percentages were demonstrated in table (2). Mostly the mode of failure in GI and GF samples was mixed cohesive/adhesive failure, while cohesive failure in PMMA was a major one in AF samples. The dominant mode of failure in untreated “as printed samples” was adhesive failure.

TABLE (1) Comparisons of means of SBS.

Comparison	P value
PI vs GI	< 0.001*
PI VS AI	0.28
GI VS AI	0.008*
PF vs GF	0.97
PF vs AF	0.3
GF vs AF	0.75
AI vs AF	1
GI vs GF	< 0.001 *
PI vs PF	1

Statistical analysis using Tukey Post Hoc Test. *P < 0.05

TABLE (2): Mode of failure Percentages in all groups.

	As printed (P)		Grinding (G)		Air abrasion (A)	
	Injectable (PI)	Flowable (PF)	Injectable (GI)	Flowable (GF)	Injectable (AI)	Flowable (AF)
Adhesive	50%	100%	12.5%	25%	37.5%	12.5%
Cohesive in PMMA	0	0	25%	0	25%	50%
Mixed cohesive/adhesive	50%	0	62.5%	75%	37.5%	37.5%

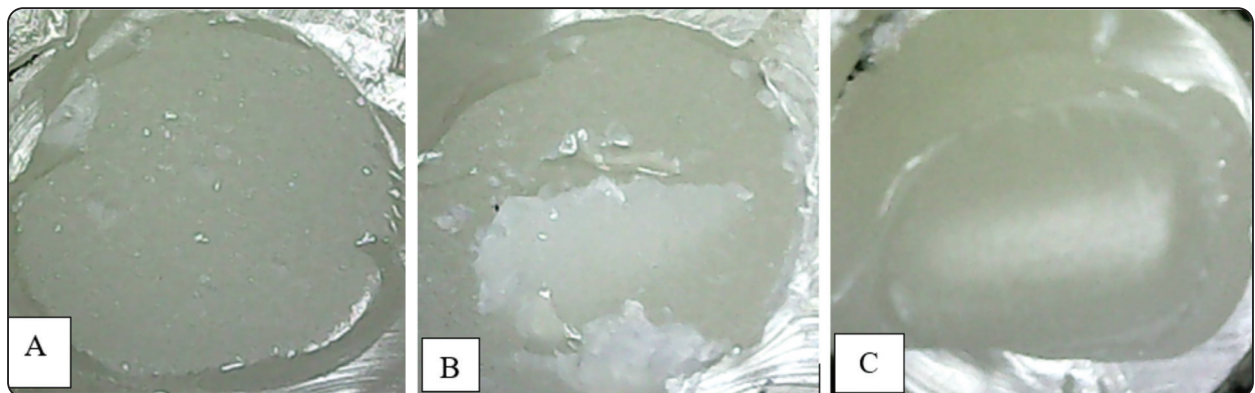


Fig. (2) Digital microscope photos of different failure modes: A: Adhesive, B: Mixed Cohesive/Adhesive, and C: Cohesive in PMMA.

DISCUSSION

Manufacturers established CAD/CAM additive methods to print PMMA restorations. PMMA restorations proved some qualities like adequate mechanical properties and pleasing appearance⁽⁸⁾. In clinical practice, temporary restorations are frequently repaired or relined since they can be used for a long time and because intraoral fractures can happen in-between visits. Because it is quicker and more cost-effective, repairing the restoration can be the most beneficial option for minor flaws⁽¹¹⁾.

It can be assumed that since industrially polymerized materials exhibit a high degree of conversion, the quantity of remaining monomers or free radicals is very small or even insufficient to permit co-polymerization with the repair material⁽¹⁵⁾. Therefore, this study was conducted to assess the influence of two surface roughening methods and two types of composite resin on the repair bond strength of aged PMMA. Samples were aged by thermocycling according to *Weigand et al.*,⁽⁵⁾ since repairing restorations typically became essential after months or years of clinical service and requires long-term stability. Affected by aging circumstances, aging greatly reduces the shear bond strength of repaired methacrylate-based composites⁽¹⁶⁾.

Using universal adhesive in this study for chemical bonding was recommended by *AlShali et al.*⁽¹¹⁾. The diffusion of the adhesive monomer into micro retentions and high surface wettability must be taken into account. The industrial polymerization of CAD/CAM resin results in a higher degree of conversion and a lower amount of unsaturated C-C bonds. It is known that new Universal adhesive systems, such as the one used in this study, contain 10-methacryloyloxy-decyl dihydrogen phosphate (MDP) monomer that has a high affinity to inorganic filler particles, which may form a covalent bond to the unreacted methacrylate groups on the matrix or to the inorganic compound^(5,15).

The various benefits of light-cured resin composites have led to their recommendation for the intraoral repair of temporary restorations in this study. These benefits include availability in a wide range of shades and viscosities, ease of handling, sufficient working time, minimal odor, minimal polymerization shrinkage, and improved marginal accuracy⁽¹⁷⁾. Both Flowable and injectable composite resin materials were utilized because of their good wettability and adaptability⁽¹³⁾. *Papacchini et al.*⁽¹⁸⁾ claimed that using a flowable resin led to greater composite-to-composite adhesion for repair purposes.

In this investigation, the shear bond strength test was used to evaluate the bond strength of repaired PMMA as it was considered a reliable and uncomplicated in vitro test for measuring bond strength according to *Soliman et al.*⁽¹⁹⁾. According to the requirements of ISO 10,477, the minimum permissible SBS value at the interface between resin-based materials and the substrate is 5 MPa⁽²⁰⁾. *Beher et al.*⁽²¹⁾ on the other hand recommended that the clinically appropriate SBS value can be 10 MPa. All shear bond strength values in our study were above 10 MPa except for “as printed” groups.

Our study's findings revealed that there was a statistically significant effect of the surface roughening method on SBS regardless of the composite type where, the highest SBS value was for “Grinded group” (G) (16.6 ± 5.8) MPa, followed by Air-abraded group (A) (14.26 ± 4.1) then “As printed” group (P) (9.9 ± 2.17) MPa. However, there was no statistically significant difference between air-abraded and grinded groups. This can be attributed to the micro irregularities developed on the surface of the samples that contributed to micromechanical interlocking with adhesive and composite resin. This could be supported by our SEM images that demonstrated many micro irregularities on the abraded sample (Fig. 1B) and many evident wide grooves, many micropores, and irregularities on the

surface of Grinded samples (Fig. 1C). The untreated “as printed samples were relatively smooth (Fig. 1A). This was also emphasized by *Menna-Serrano et al.*⁽²²⁾ who stated that diamond bur application has been reported to render retentive properties at the micro and macro levels. Also, these findings were in agreement with *Weigand et al.*⁽⁵⁾ and *Bahadir et al.*⁽²³⁾.

There was no statistically significant difference between Injectable (I) (14.96 ± 5.1) MPa and Flowable composite (F) (12.24 ± 3.84) MPa regardless of the surface roughening method used. These findings were supported by *AlBahri et al.*⁽¹⁾. On the other hand, *Gonulol et al.*⁽¹²⁾ concluded that aged composite could be better repaired with the same substrate composite resin rather than injectable composite resin. They attributed their finding to the chemistry of the composite resin of both the substrate and the one used for repair.

Grinded PMMA discs repaired with Injectable composite showed the highest statistically significant shear bond strength value (21.32 ± 1.71) MPa in comparison to untreated PI (9.52 ± 1.86) MPa and air-abraded ones AI (14.05 ± 4.47) MPa and higher than Grinded PMMA repaired with flowable composite GF (11.87 ± 4.16) MPa. This can be attributed to micromechanical retention coupled with chemical bonding. The highly filled injectable composite resin had better wettability and adaptability in comparison to Flowable composite resin and a unique thixotropic viscosity that might allow its high penetration to the evident grooves and micropores causing efficient mechanical interlocking and chemical bond through MDP-containing adhesive with the PMMA^(5,15,13). This explanation could be supported by the dominant mixed adhesive/cohesive failures (62.5%) in this study followed by Cohesive failure in PMMA (25%).

The different modes of failures presented could entail more about the bonding behavior and mechanism. For surface treated samples the major mode of failure was mixed cohesive/adhesive

failure for GF samples and cohesive in PMMA for AF samples. While the dominant mode of failure for “as printed untreated” PI and PF samples was adhesive. This could be attributed to the combined micromechanical and chemical bonding. The relatively smooth surface of P samples as in our SEM image (Fig.1A) might explain its low bond strength values and adhesive failure mode.

According to the findings of this study, the null hypothesis was partially rejected. The lack of long-term water storage or artificial aging by thermal cycling for bonded samples was a methodological constraint of this investigation. Additional research in a clinical context will help in selecting repair material and enhancing the repair procedure for 3D-printed temporary restorations.

CONCLUSIONS

Within the limitations of our study the following conclusions could be drawn:

1. Grinding with diamond abrasive or air abrasion with alumina improved the bond strength of PMMA to the repair composite resin.
2. Grinded PMMA had a greatly enhanced repair bond strength to injectable composite resin.
3. Injectable and flowable composite resin materials had nearly equal repair bond strength to air-abraded PMMA.

Clinical recommendations:

For adequate repair bond strength, it was recommended to roughen the surface of PMMA provisional restoration either by grinding using diamond abrasive stone or air abrasion using alumina particles followed by application of universal adhesive. Injectable composite resin was recommended after grinding the surface with diamond abrasive stone to obtain an improved bond strength. Both Injectable and Flowable composite resin materials could be used following air abrasion.

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