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Repair Bond Strength Of 3D Printed PMMA With Dichloromethane Surface Treatment -An Invitro Study

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Abstract:
Background*: Digital denture fabrication using 3D printing offers improved accuracy and faster production. However, fractures still occur due to stress or impact. Although it has effect on bond strength, repair is often preferred over replacement as it is economical and saves chair side and laboratory time. Chair side repair methods like surface roughening, monomer application and dichloromethane application enhanced the bond strength of conventional PMMA with autopolymerizing PMMA repair material. But there were no studies done so far to assess the repair bond strength of dichloromethane surface treated 3D printed PMMA material with autopolymerizing PMMA repair material. Hence this study is done to assess and compare the repair bond strength of 3D printed material, surface treated with dichloromethane and without dichloromethane.*
Materials and Methods*: Thirty specimens of dimension 10x10x3mm were fabricated from a 3D printed PMMA resin and all the specimens were sandblasted with aluminum oxide powder. Specimens were divided into 3 groups of 10 specimens each based on surface treatment. Group A is control group not subjected to any surface treatment, Group B is surface treated with Methylmethacrylate and Group C is surface treated with Dichloromethane. An autopolymerizing repair material of dimensions 4mmx6mm is added onto the 3D printed PMMA resin specimens. The specimens were then subjected to testing of shear bond strength using Universal testing machine.*
Results*: Specimens surface treated with dichloromethane has significantly higher repair bond strength compared to the other groups.*
Conclusion: *Dichloromethane can be used to enhance the repair bond strength of 3D printed PMMA resin.*
Key Words: *Dichloromethane, Methylmethacrylate, Acrylic resin, Denture base, Repair, PMMA.*

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I. Introduction

Digital denture fabrication seeks to address the shortcomings of conventional denture fabrication [10]. Two techniques were described in digital fabrication of dentures, additive manufacturing (AM, 3D-printed) and subtractive manufacturing (SM, milled), with minimal manufacturing distortion aiming to produce a prosthesis with precise retention, stability, and support [10][18]. In Additive manufacturing technology, an object is constructed layer by layer using photopolymerized fluid resins with various printing technologies and printing parameters, but in Subtractive manufacturing method, the denture is milled from pre-polymerized PMMA discs using milling burs to the intended configuration [12][15][19].

AM has less mechanical performance as compared to SM, but within clinically acceptable limits; hence both methods can be successfully used for denture fabrication [16]. But AM is preferred over SM because there are no waste materials, does not require milling burs which deteriorate, and high-accuracy designed materials may be produced several times and it is also less expensive [16].

Given the vast dispersion, the development of digital technologies, and the application of both methods for denture fabrication, the denture may break as a result of flexing action and various intraoral tensions or impact forces due to unintentional falls [21][22]. When broken portions can readily reestablish the denture's previous relationship, repair is one of the most acceptable alternatives to fabricating a new denture as it is economical and saves chair side and laboratory time. Numerous elements, including the type of repair material, surface design, and surface treatment, influence the strength of the repair [12][13]. Both mechanical and chemical surface treatments were recommended, and both reported stronger bonds than the untreated groups [8][11].

Studies have shown that surface treatments with methyl methacrylate enhance repair strength through monomer diffusion and interpenetrating polymer network formation [5][7]. Dichloromethane has been widely recommended due to its ability to swell PMMA surfaces and create microporosities, thereby improving mechanical interlocking and transverse strength in the conventional dentures [1][4], but has not been used in repair of 3D printed dentures. The air-abrasive particles, which roughen the surface with alumina particles of varying sizes, are one type of mechanical surface treatment. Furthermore, alumina blasting, either by itself or in conjunction with chemical surface treatment demonstrated superior performance in comparison to the MMA application in conventional dentures [11].

Limited research was done to evaluate the repairability of 3D-printed resins, furthermore, there were no studies done so far to assess the repair bond strength of 3D printed PMMA material surface treated with dichloromethane. Hence my present study was taken up to examine the repair bond strength of 3D-printed PMMA resins surface treated with and without dichloromethane.

Repair strength can be evaluated by shear bond strength (SBS), a load was applied at the interface with vertical forces in an attempt to debond the repaired resin [8]. Therefore, SBS was chosen to assess the repair bond strength while concentrating on the impact of surface treatments in the 3D printed denture base.

II. Methodology

Thirty specimens of dimension 10x10x3mm were fabricated from a stereolithographic (SLA) 3D printed PMMA resin (SUNLU™) (Fig. 1). The specimens were mounted in a resin holder (18mm(d)x10mm(h)) (Fig. 2).

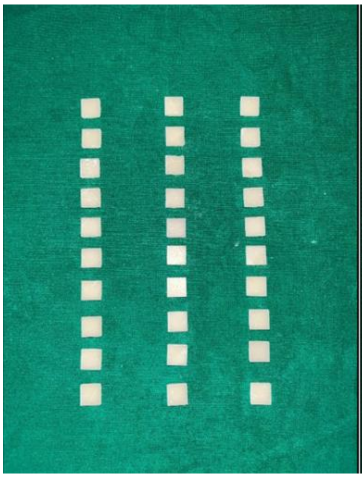
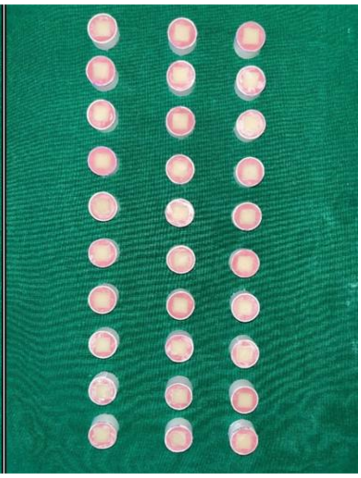

Fig. 1: 3D printed specimens.
Fig. 2: Specimens embedded in resin holder.

All the specimens were sand blasted with aluminum oxide powder (50μm) (Fig. 3).

Fig. 3: Sandblasting of specimens.

Specimens were divided into 3 groups of 10 specimens each based on surface treatment. Group A is control group not subjected to any surface treatment; Group B is surface treated with Methyl methacrylate (IVOCLAR SR Triplex**®**). Methyl-methacrylate is taken and applied uniformly on the surface of the specimen with the help of a microbrush in one direction for 180s (Fig. 4) and Group C is surface treated with Dichloromethane (BFCLAB**®**), Dichloromethane is taken and applied uniformly on the surface of the specimen with the help of a microbrush in one direction for 45s (Fig. 5).

Fig. 4: Application of Methylmethacrylate.
Fig. 5: Application of Dichloromethane.

A customized mold of dimensions 4mmx6mm was fabricated (Fig. 6) and autopolymerizing repair material (IVOCLAR SR Triplex**®)** is added into the custom mold onto the 3D printed PMMA resin specimens. The custom mold was carefully removed after polymerization.

Fig. 6: Custom mold.
Fig. 7: Repaired specimens.

All of the specimens were immersed in distilled water(37ºC) for 2 days and then specimens were then subjected to testing of shear bond strength using universal testing machine (INSTRON Servo-Hydraulic Testing Machine**®) (**Fig. 8), the load was applied at a 1mm/min crosshead speed at the junction between 3D printed PMMA and repair material (Fig. 9).

Fig. 8: INSTRON Servo-Hydraulic Testing. Machine®
Fig. 9: Application of load.

Data was collected and One-Way ANOVA and Tukey Post-hoc Comparison was done.

III. Results

The collected data were entered and analyzed using SPSS version 26. Descriptive statistics, including mean, standard deviation (SD), and standard error (SE), were calculated for each group. A one-way analysis of variance (ANOVA) was performed to compare the mean shear bond strength among the three groups: Control, Methyl methacrylate, and Dichloromethane groups. The ANOVA test determined whether statistically significant differences existed between the group means. When a significant difference was observed, post-hoc analysis using Tukey's Honestly Significant Difference (HSD) test was conducted to identify specific group pairs showing significant variation. A *p*-value of less than 0.05 was considered statistically significant. The results were presented in tabular and graphical forms to provide a clear comparison of the mean shear bond strengths and the pairwise differences among the groups

Table 1.: One-Way ANOVA for Shear Bond Strength (MPa)

Source	F	df1	df2	p-value
Shear Bond Strength Test	6.65	2	27	0.004*

P<0.005 is considered statistically significant

Interpretation:

The one-way ANOVA revealed a statistically significant difference in mean shear bond strength among the three groups ($p = 0.004$). This indicates that at least one of the surface treatment methods resulted in a significantly different bond strength compared to the others.

Table 2.: Group Descriptive Statistics (Shear Bond Strength in MPa)

Group	N	Mean (MPa)	SD	SE
Control Group	10	2.76	0.787	0.249
Methyl Methacrylate Group	10	2.92	0.905	0.286
Dichloromethane Group	10	4.11	1.010	0.319

SD: Standard deviation, SE: Standard error

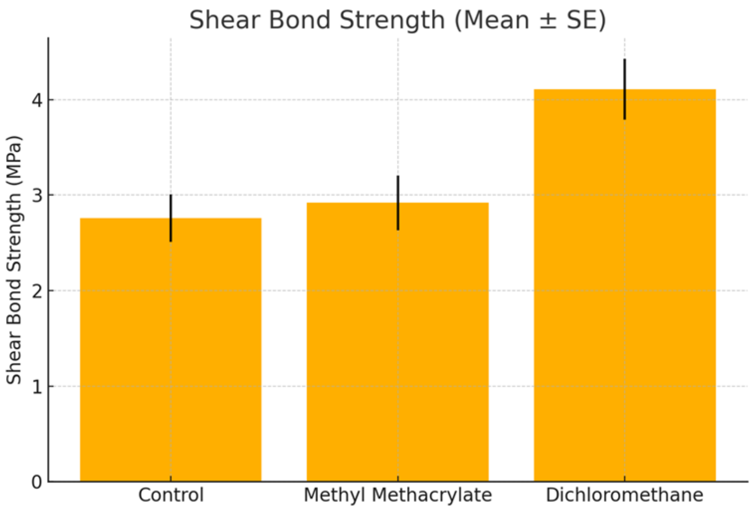

Fig. 10: Shear bond strength

Interpretation:

The Dichloromethane-treated group showed the highest mean shear bond strength (4.11 MPa), followed by the Methyl methacrylate group (2.92 MPa), while the control group exhibited the lowest (2.76 MPa) (Fig. 10). The relatively higher standard deviation in the Dichloromethane group suggests more variability in bond strength values within that group.

Table 3.: Tukey Post-hoc Comparison (Shear Bond Strength in MPa)

Comparison	Mean Difference (MPa)	p-value
Control vs Methyl Methacrylate	-0.159	0.919
Control vs Dichloromethane	-1.35	0.007
Methyl Methacrylate vs Dichloromethane	-1.19	0.018

P<0.005 is considered statistically significant

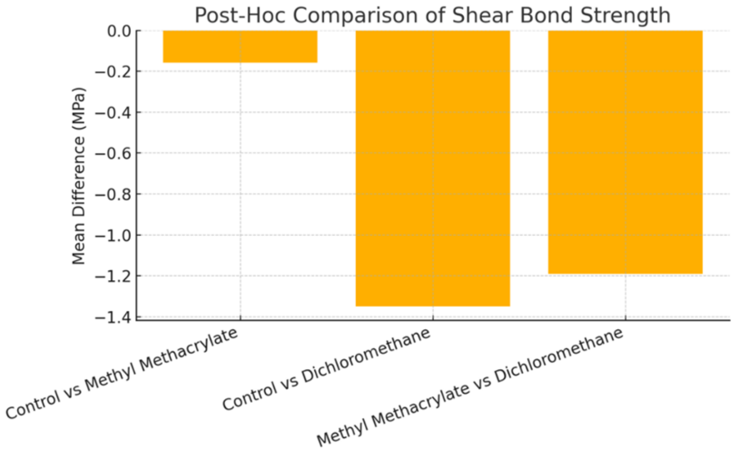

Fig. 11: Post-hoc comparison of shear bond strength

Interpretation:

Post-hoc analysis showed a statistically significant difference in shear bond strength between the Dichloromethane group and both the Control ($p = 0.007$) and Methyl methacrylate groups ($p = 0.018$) (Fig. 11). However, there was no significant difference between the Control and Methyl methacrylate groups ($p = 0.919$). This confirms that treatment with Dichloromethane significantly improves bond strength compared to the other two groups.

The findings from the shear bond strength evaluation indicate that surface treatment significantly affects the bonding efficacy between the materials tested. Among the three groups, the Dichloromethane-treated specimens demonstrated the highest mean bond strength, suggesting superior adhesive performance compared to both the Control and Methyl methacrylate-treated groups.

IV. Discussion

In this invitro study, the effects of the surface treatment on the repair bond strength of 3D Printed PMMA were tested. Surface treatment with Dichloromethane showed superior bond strength compared with Methyl methacrylate group as well as untreated specimens.

Numerous attempts have been made to strengthen the repaired denture base, including gap modifications and the reinforcing of the resins [13][14]. Although repair strength was improved by using either one of these procedures alone or in combination, a poor bond at the repair/interface was noted in terms of adhesive failure [8][11]. The bond between the denture base and the repair material interface is a crucial component to provide the best repair strength. Good bonding depends on a number of factors, including the type of original resin, repair resin, and surface treatment (Chemical or Mechanical) [8][11]. Surface roughening (with Al_2O_3) increases the surface area at the repair contact and causes pits and macro and micro abnormalities[8][11]. Furthermore, with the use of Methyl methacrylate (MMA), the dissolution of the repair interface is succeeded by the diffusion and polymerization of monomers across the repaired interface, resulting in the formation of an interpenetrating polymer network. These modifications together enhance the effective bonding surface for micromechanical adhesion and retention between the repaired surface and the repaired resin [7].

Dichloromethane is a nonpolymerizable, organic solvent that causes surface swelling and allows the polymerizable substance to diffuse. The degree of solvent penetration and the strength of the resulting interwoven polymer network determines the bond strength. On a conventional acrylic resin, dichloromethane pretreatment can form surface pores and channels that are about 1 µm in diameter which improves the bond strength [1][4]. However, there were no studies done so far to evaluate the repair bond strength of 3D printed PMMA surface treated with dichloromethane.

To evaluate the repair strength of the repaired resins, the flexural strength test has been commonly used in previous studies done by ***Gad et al. and Abushowmi et al.*** [13][14]. Shear bond strength (SBS) was recommended for the evaluation of bond strength at the resin–repair interface even though it primarily replicates the stress that dentures are exposed to in the oral cavity [2][3].

In the current study, application of MMA increased the bond strength when compared to control group which agrees to the study done by ***Rupp et al.*** [1]. Surface treatment with dichloromethane significantly improved the bond strength when compared to both control and MMA groups for the 3D printed PMMA resin. This agrees with previous studies done by ***Jain G et al***. that showed increased SBS with surface roughening (SB), followed by MMA and Dichloromethane surface treatments for the conventional dentures [9].

The study's limitations were that it was conducted in vitro, meaning that the testing did not exactly replicate the clinical environment. For example, there were no variations in salivary pH effects, repeated stressors, masticatory forces, or oral conditions. Additionally, the application thickness of the methyl-methacrylate and dichloromethane may vary with the manual application technique and surface roughness analysis, thermocycling and comparison with the heat cure PMMA material were not done.

Furthermore, specimens did not simulate the denture configurations. Therefore, additional investigations under closely simulated clinical conditions are required for evaluating the repair bond strength of 3D-printed resins using different surface treatments for determining the most appropriate surface treatment.

V. Conclusion

Based on the findings of this study, it can be concluded that:

Among the three groups, the dichloromethane-treated specimens demonstrated the highest mean bond strength (4.11MPa), suggesting superior adhesive performance compared to both the control(2.76MPa) and methyl methacrylate(2.92MPa) surface treatment groups.

Dichloromethane surface treatment of 3D printed PMMA resins increases the SBS between repair resins and 3D printed PMMA resin.

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