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# The effect of adhesive material and thermocycling on bond strength of intraoral repair materials on adhesive fracture in porcelain fused to metal restoration

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***Abstract*---**Porcelain fused to metal restoration often fails in the form of adhesive fracture in the porcelain veneered layer. Intraoral repair can be used to repair this type of fracture. In intraoral repair, adhesive material is used for bonding chemically to the oxide layer on the metal surface. 10-MDP adhesive material is commonly used in dental practice. Another adhesive used is 6-MPHA because it is a new adhesive material that has a C-P bond in the middle of the compound chemistry which more stable and can withstand harsh chemical treatments. Bond strength of the adhesive material is affected by usage (aging). Aging can be simulated by using thermocycling in laboratorium research. Objectives: to determine the effect of 6-MPHA, 10-MDP on the adhesive strength of intraoral repair materials on adhesive fractures of PFM restorations in the thermocycling group of 500, 1000, and 3000 cycles, and to determine the effect of


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thermocycling 1000, 3000 cycles on adhesive strength of intraoral repair materials in adhesive fractures of PFM restorations using 6-MPHA and 10-MDP adhesives. Material and method: Sampel was made in the form of cylindrical metal with a diameter of 7 mm and a thickness of 3 mm with total sampels 30 pieces. The sample group was divided into 2 groups based on the adhesive material, 6-MPHA and 10-MDP. Then each sample group was further divided into two groups based on the number of thermocycling 500, 1000, and 3000 cycles. The measurement of the value of the shear bond strength was carried out using a universal testing machine. Results: There was an effect of adhesive material on the adhesive strength of intraoral repair materials on adhesive fractures of PFM restorations in the thermocycling group of 500 cycles, 1000 cycles, and 3000 cycles with p value= 0,003 (500 cycle), 0,044 (1000 cycle), 0,002 (3000 cycle) ($p<0.05$), there was no effect of thermocycling on the adhesion strength of intraoral repair materials on fractures adhesive of PFM restorations using 6-MPHA with p value= 0,151 ($p>0.05$), and there was an effect of thermocycling on the adhesive strength of intraoral repair materials on adhesive fractures of PFM restorations using 10-MDP with p value= 0,012 ($p< 0,05$). Conclusion: that 10-MDP is a type of adhesive material that can be used for clinical use because it has a minimal bond strength that is suggested for clinical usage.

***Keywords*---**adhesive material, thermocycling, PFM fracture, shear bond strength.

## Introduction

Porcelain fused to metal (PFM) restoration is the type of restoration that is most widely used in dentistry because it is more durable than any other types of restoration.[1] The most common found failure is fracture of the porcelain veneer layer in PFM restorations which can cause aesthetic complaints in patients with incidence of by 34%. Based on Friedman's classification, porcelain fractures can be divided into 3 such as static fractures, cohesive fractures, and adhesive fractures. Static fracture occurs in only one small piece of porcelain, with the remaining porcelain intact. Cohesive fracture occurs in the porcelain body due to the tensile forces of the mastication. Adhesive fracture is a condition of failure at the interface between porcelain and metal so that the metal is exposed.[2]

Repair of PFM restorations can be performed extraoral, intraoral, or combination. Among these techniques, it can be seen that intraoral repair has many advantages such as being able to be done at the same visit, good esthetics, and inexpensive when compared to the other two techniques.[3] There are several problems found in adhesive fracture compare to cohesive fracture, such as there is very little silica ($SiO_2$) component on metal surface so that it can not bond well with the silane coupling agent directly compared to porcelain surfaces containing silica components.[4] Metal surfaces also cannot be modified using acid etching.[5] These problems lowered the bond strength of the adhesives in intraoral repair on the metal surfaces.

Adhesive fractures in PFM restoration can be repaired by using adhesive material containing metal primer or silane coupling agent.[6] Adhesive materials produce physicochemical interaction between resin (adhesive) and metal (substrate). The physical contribution of the adhesive material depends on the surface treatment and the topography of the substrate. The chemical contribution of the adhesive material depends on the oxide layer that is present in the substrate surface because it is chemically bonded to the substrate oxide layer. Adhesive materials that can be used for intraoral repair must have a metal primer containing active monomers such as 10-MDP, and 6-MPHA.[7] According to the literature, intraoral repair materials are indicated for clinical usage when the adhesive bond strength is >10 MPa.[8]

The success of the repair depends on the longevity of the repair material in the patient's mouth. Cinar et al. (2019), many studies report that the durability of dental materials decreases when used in the oral cavity for a certain period of time. This condition should be considered in in-vitro studies to simulate clinical conditions with artificial aging.[9] Thermocycling is the most effective protocol in artificial aging.[10] Although thermocycling can not perfectly simulate conditions in the oral cavity, such as clinical conditions, humidity, and stress caused by antagonistic teeth, but to a certain extent thermocycling can simulate the condition of the oral cavity through aging procedures on teeth and restorations so that the research results will more closely resemble to the results obtained from clinical conditions.[11] The purpose of this paper is to determine the effect of 6-MPHA, 10-MDP on the adhesive strength of intraoral repair materials on adhesive fractures of PFM restorations in the thermocycling group of 500, 1000, and 3000 cycles, and to determine the effect of thermocycling 1000, 3000 cycles on adhesive strength of intraoral repair materials in adhesive fractures of PFM restorations using 6-MPHA and 10-MDP adhesives.

**Material and Method**

Thirty cylindrical samples were fabricated using Ni-Cr alloy with a diameter of 7 mm and a thickness of 3 mm. To standardize the diameter and size of the samples, a metal jig (Figure 1) was made with a mold diameter and thickness of 7 mm and 3 mm. Thirty wax patterns were made by melting inlay casting wax (Eisenbaher Dentalawaren, Germany) into a metal jig. The wax pattern was sprue up in the middle of the wax pattern diameter, then attached to the center of the crucible former with a minimum distance of 6-8 mm between the pattern and the casting ring. The wax pattern was then vacuum invested using 500 g of phosphate bonded investment material and 90 ml of mixing liquid (S.P.E, China) according to the manufacturer's instructions. After the investment setting, the crucible former was removed and the casting ring was inserted into the heating furnace (Renfert, Germany) with the sprue on the bottom and heated to a temperature of 750$^0$C and maintained for 45 minutes. The Ni-Cr metal was then melted and casted using an electric casting machine. After casting, the mold was removed and the remaining investment material could be cleaned by sandblasting and ultrasonic cleaning. Sprue was removed from the metal sample, the porous metal sample was excluded.

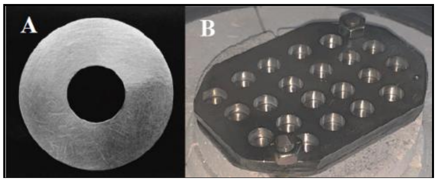

Figure 1. Mold for fabricating sample (A. individual; B. Multiple)

The metal sample was then embedded in an autopolymerized acrylic resin with the help of a metal jig with an internal diameter and height 2 cm. Make sure the metal surface was in line with the acrylic surface (Figure 2). Mounted samples were then finished using sand paper sizes 80, 120, 180, 240, 320 grit with a figure eight grinding pattern and cleaned with ultrasonic cleaning for 15 minutes to remove trapped residue.[4,12] The sample was then sandblasted using aluminum oxide (particle size 50 μm) at a pressure of 3 bar / 0.3 MPa for 15 seconds. Samples were washed with water for 20 seconds and air-dried for 20 seconds. The sample was then applied with intraoral repair material.


Figure 2. Sample embedded in an autopolymerized acrylic resin with the help of a metal jig

**6-MPHA (15 sample)**

The sandblasted metal surface was applied with M.L Primer (Shofu, Japan) and left for 10 seconds, then Cera Resin Bond 1 (Shofu, Japan) was left for 10 seconds and followed by Cera Resin Bond 2 (Shofu, Japan) left for 10 seconds and light cure for 20 seconds. Apply a thin layer of opaque (IPS Empress Direct Opaque, Ivoclar, Liechtenstein) on the metal surface and then light cure for 20 seconds.

**10-MDP (15 sample)**

Apply 1 layer of metal primer (Monobond, Ivoclar, Liechtenstein) on the sandblasted metal surface left for 60 seconds and then dried with air spray. Apply 1 layer of adhesive resin (Heliobond, Ivoclar, Liechtenstein) to metal surface and remove excess liquid with air spray and light cure for 20 seconds. After the adhesive was applied, the composite resin was applied using a metal matrix mold (Figure 3) with an internal diameter of 4 mm and a thickness of 2 mm which was placed in the middle of the metal surface. Composite resin was then light cure for 20 seconds.[13]

Figure 3. Metal matrix mold for applying composite resin

Samples that had been bonded with repair material (Figure 4) are immersed in distilled water for 24 hours at a temperature of $37^{0}C$. The samples were then divided into 3 groups, with each group being 500 cycles, 1000 cycles, and 3000 cycles in water with a temperature of $5^{0}C$ and $55^{0}C$, dwelling time: 20 seconds.[14,15]

Figure 4. Sample bonded with repair material

The shear bond strength was measured using a universal testing machine (UTM) (Tokyo Testing Machine MFG Co., Switzerland) with a 10-kN load cell and 0.5 mm/min crosshead speed. The value of the shear bond strength is obtained by = Force (N) / area ($mm^2$).

## Result

Table 1 showed mean and standard deviation of the bond strength of the adhesive material. Based on the T test, it could be seen that there was a significant effect between the adhesive materials in each thermocycling cycle ($p < 0.05$)

Tabel 1
Average bond strength value of the adhesive material in each thermocycling group

| Thermocycling (Cycle) | Group | Mean (MPa) | SD | P value |
|---|---|---|---|---|
| 500 | 6-MPHA | 7.73 | 2.29 | 0.003* |
| | 10-MDP | 12.82 | 1.48 | |
| 1000 | 6-MPHA | 6.74 | 1.95 | 0.044* |
| | 10-MDP | 9.96 | 2.29 | |
| 3000 | 6-MPHA | 5.22 | 1.32 | 0.002* |
| | 10-MDP | 8.99 | 1.32 | |

* significant difference

Based on the one-way ANOVA (Table 2), it can be seen that there is no significant effect of thermocycling on the 6-MPHA adhesive, while the 10-MDP adhesive had a significant effect, then followed by a post hoc test to see results between the thermocycling groups. From the results of the post-hoc test to 10-MDP group, it was found that there was a significant difference between the groups of 500 and 1000 cycles (0.024*), 500 and 3000 cycles (0.005*), but there was no significant difference between the groups of 1000 and 3000 cycles (0.403).

Table 2
One-way ANOVA test on the effect of thermocycling on the adhesive material

| Group | Thermocycling (cycle) | Mean (MPa) | SD | P value |
|---|---|---|---|---|
| 6-MPHA | 500 | 7.73 | 2.29 | 0.151 |
| | 1000 | 6.74 | 1.95 | |
| | 3000 | 5.22 | 1.32 | |
| 10-MDP | 500 | 12.82 | 1.48 | 0.012* |
| | 1000 | 9.96 | 2.29 | |
| | 3000 | 8.99 | 1.32 | |

* significant difference

**Discussion**

In all thermocycling groups between the 2 adhesives, it was seen that the lowest value of bond strength was found in the 6-MPHA adhesive group. This may be due to the difference in the bonds between the middle of the chemical compound. The C-P bond in the 6-MPHA adhesive which is more resistant to water may actually cause the strength of the adhesive itself to be weaker than that of the 10-MDP adhesive. Another possibility that may occur is due to the different coefficients of thermal expansion and hygroscopic expansion between the 2 types of adhesives.[16] The lowest bond strength in each adhesive material group was in 3000 cycles and the highest was 500 cycles (Table 1). This is probably because thermocycling can cause artificial aging through 2 mechanisms such as hot water can accelerate the occurrence of hydrolysis and decomposition in the component interphase area, and repeated contractions and thermal expansion can cause stress on the metal and composite resin interfaces which can synergistically increase the degradation. hydrolytic on the adhesive.[17] Bakhsh et al. (2021), in his research also stated that thermocycling can cause a larger gap between the tooth and the restorative material.[16]

The absence of thermocycling effect on the 6-MPHA adhesive (Table 2) may be due to the presence of C-P bonds in the 6-MPHA adhesive which causes this type of adhesive to be more water resistant so that it is less affected by thermocycling compared to other types of adhesives. The C-P bond contains less oxygen than the O-P bond, so it is more stable and resistant to chemical treatments, such as boiling in strong acid or strong wetness.[18] In the 10-MDP adhesive, it can be seen that there is a significant decrease in bond strength after thermocycling. This condition may be caused by the hydrophilic nature of the phosphate ester group in the 10-MDP adhesive which binds to the metal surface so that it is easily hydrolyzed by water which causes a decrease in adhesive strength. In this research, it could be seen that in 1000 and 3000 cycle thermocycling group, there was no significant difference in bond strength between each group. This result is in line with the research of Xirouchaki et al (1997) which stated that the 2500 cycle thermocycling had no significant effect on the adhesive strength of the adhesive and Aguilar's (2002) study which also stated that there was no significant difference in the adhesive strength of the adhesive to enamel above 3000 cycles. This condition may be caused by post-polymerization strengthening of the adhesive in the early phase and retardation of degradation of the adhesive material.[20]

## Conclusion

The 10-MDP adhesive material had a higher adhesive strength for all periods of clinical use than the 6-MPHA adhesive. The adhesive strength of 10-MDP adhesive is adequate for clinical use because it has a minimum value of shear bond strength recommended for clinical use. The clinical implication of this study is that 10-MDP adhesive can be used to repair adhesive fractures of porcelain fused to metal restorations.

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