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Shear Bond Strength of Four Different Repair Materials Applied to Bis-acryl Resin Provisional Materials Measured 10 Minutes, One Hour, and Two Days After Bonding

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Clinical Relevance

Strong bonding can be achieved between bis-acryl base and bis-acryl repair material. Time after bonding is an important factor for bond strength of repaired bis-acryl provisional restorations.

SUMMARY

This study investigated the shear bond strength of repaired provisional restoration materials 1) to compare the bond strengths between bis-acryl resin and four different materials and 2) to investigate the effect of the amount of time elapsed after bonding on the bond strength. The self-cured bis-acryl resin (Luxatemp) was used as the base material, and four different types of resins (Luxatemp, Protemp, Z350 flowable, and Z350) were used as the repair materials. Specimens were divided into three groups depending on the point of time of shear bond strength measurement: 10 minutes, one hour, and 48 hours. Shear bond strengths were measured with a universal testing machine, and the fracture surface was examined with a video measuring system. Two-way analysis of variance revealed that the repair materials ($p<0.001$) and the amount of time elapsed after bonding ($p<0.001$) significantly affected the repair strength. All of the repaired materials showed increasing bond strength with longer storage time. The highest bond strength and cohesive

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Table 1: *Materials Used in the Experiment*

Product	Major Components	Polymerization Mode	Manufacturer	Lot
Luxatemp-Plus	Bis-acryl	Self-cured	DMG	681163
Protemp 4	Bis-acryl	Self-cured	3M ESPE	B 483558
				C 482519
Filtek Z350 XT Flowable	Bis-GMA	Light-cured	3M ESPE	N 420221
Filtek Z350 XT	Bis-GMA	Light-cured	3M ESPE	N 453720

Abbreviation: bis-GMA, bisphenol A diglycidyl ether dimethacrylate.

failure were observed for bonding between Luxatemp base and Luxatemp at 48 hours after bonding.

INTRODUCTION

During the past decade, bis-acryl resin composites have become popular as provisional restoration materials as a result of their ease of use, minimal polymerization shrinkage, and low exothermic reaction.[1-3]

A vacuum-formed acrylic splint or polyvinyl siloxane impression is frequently used as a direct matrix for provisional formation with bis-acryl resins.[4] In the process of fabricating provisional restorations, relining or repair is inevitable because of trapped air,[5] shortened margin,[6,7] and fracture.[8] There have been clinical attempts to use bis-acryl resin for self-repair, but that strategy proved ineffective.[9]

Light-cured flowable resins have been suggested[10,11] for the repair of bis-acryl resin provisional restorations. They are easily manipulated and bonding with bis-acryl resin is effective and durable.[12,13] Light-cured packable composite resin has the same components as light-cured flowable resin. However, to date there has been no published study on the bond strength between a bis-acryl composite and light-cured packable composite resin.

Shear bond strength tests are commonly used to evaluate the bonding between experimental substances and bis-acryl resins, and most laboratory bond strength tests are performed 24 hours after the bonding process and water storage of the specimens. However, in most clinical situations, the repaired interface of a provisional restoration is subjected to various kinds of stress immediately after bonding. These stresses result from adjustments and polishing procedures of the repaired provisional restorations as well as physical loading in the oral environment because it is necessary for provisional restorations to replace removed tooth structure as soon as possible after preparation as an interim treatment of the fixed prosthodontic treatment. Therefore, the translation of previous laboratory results to the clinical setting is difficult, and time is considered an important factor in the design of experiments on shear bond strength of repaired provisional restorative materials.

The purposes of this *in vitro* study were 1) to compare the bond strengths between bis-acryl resin and four different materials, including light-cured packable composite resin, and 2) to investigate the effect of the storage time on the bond strength at the repaired interface of provisional restorations.

METHODS AND MATERIALS

Materials

The self-curing bis-acryl resin (Luxatemp AM Plus, DMG, Hamburg, Germany; shade A2, Lot 681163) was used as the base material. Four different types of resins (Luxatemp, Protemp, Z350 flowable, and Z350) were used as repair materials, and Table 1 provides an overview of the materials tested. All commercial materials were used according to the manufacturers' recommendations.

Preparation of Specimens

The shear bond strength test required the use of a sample holder to fix the specimen in the universal testing machine perpendicular to the orientation of the shear force. Transparent acrylic-glass rods (30 mm in diameter; Polymicar, Tae-guang, Korea) were fabricated for this purpose. The mixed self-curing bis-acryl resin (Luxatemp AM Plus) was dispensed into a 12-mm-wide hole in the acrylic-glass rod and was allowed to set for 20 minutes. Procedures for fabricating each specimen, including preparing base materials and bonding with repair resin, were finished within 30 minutes.

Prior to repair, the surfaces of the specimens were ground with silica-carbide (SiC) paper (grit 600), rinsed with water for 10 seconds, and dried for 30 seconds with an air syringe. Hard transparent gelatin capsules (4.0 mm in diameter) were used as

Table 2: *Study Design in the Experiment*

	Luxatemp + Luxatemp	Luxatemp + Protemp	Luxatemp + Z350 (Flowable)	Luxatemp + Z350
10 min	Group 1 (LT-LT/10 min)	Group 2 (LT-PT/10 min)	Group 3 (LT-F/10 min)	Group 4 (LT-P/10 min)
1 h	Group 5 (LT-LT/1 h)	Group 6 (LT-PT/1 h)	Group 7 (LT-F/1 h)	Group 8 (LT-P/1 h)
48 h	Group 9 (LT-LT/48 h)	Group 10 (LT-PT/48 h)	Group 11 (LT-F/48 h)	Group 12 (LT-P/48 h)

matrices for the production of columns of relining materials bonded to the bis-acrylic provisional resin surface. The capsule was partially filled with one of four repair materials to limit the thickness of the bonded material to 2 mm. Light-cured flowable resins (Filtek TM Z350 XT Flowable, 3M ESPE, St. Paul, MN, USA) and light-cured packable composite resins (Filtek TM Z350 XT, 3M ESPE) were light-cured with a commercial light-curing unit (Elipar freelight 2, 3M ESPE) for 40 seconds. After polymerization, specimens were removed from the mold and stored in a dry oven (WiseVen, Daihan Scientific Co, Seoul, Korea) at 37°C. Table 2 describes the groups tested. Bonding strength of repaired specimens was measured 10 minutes, one hour, and 48 hours after bonding. Each group included 10 specimens.

Determination of Shear Bond Strength

After the storage time, the shear bond strength was measured with a universal testing machine (AG-10KNX, Shimadzu Co, Japan). A knife-edge shearing rod at a crosshead speed of 2 mm/min was used to place a load on the specimens until fracture occurred. The shear bond strength in MPa was calculated from the peak load of failure. The aspect of each bonding failure was determined using a video measuring system (Optical video measuring system, Seven Ocean, Korea) at 10× magnification and was recorded.

Statistical Analysis

The mean and standard deviation of the shear bond strengths were calculated for each group. Data were evaluated for homogeneity of variance based on the Levene test (p=0.05). The influence of independent variables, including materials and storage times, was analyzed by a two-way analysis of variance (ANOVA) (p=0.05). Comparison of bond strength between repaired specimens was conducted by one-way ANOVA and Tukey multiple comparison tests (p=0.05). All statistical analyses were carried out with SPSS for Windows (release 12.01; SPSS Inc, Chicago, IL, USA).

RESULTS

The results of shear bond strength testing for each experimental group are summarized in Table 3 and Figure 1. Tables 4 and 5 show comparisons among repaired specimens evaluated by one-way ANOVA ($p<0.05$). Two-way ANOVA revealed that there were significant differences in shear bond strength because of the repair resin variables and the storage times ($p<0.001$; Table 6).

The results for detachment at 10 minutes after bonding indicated that group 4 (LT-P/10 minutes) had the highest shear bond strength, followed by group 3 (LT-F/10 minutes), group 1 (LT-LT/10 minutes), and group 2 (LT-PT/10 minutes). However, there were no statistically significant differences between groups 4 and 3 and between groups 1 and 2 (Table 4). Thus, the shear bond strengths of group 3 (LT-F/10 minutes) and group 4 (LT-P/10 minutes) were statistically higher than those of group 1 (LT-LT/10 minutes) and group 2 (LT-PT/10 minutes) ($p<0.05$).

Group 8 (LT-P/one hour) showed the highest shear strength at one hour after bonding, which was significantly higher than that of group 5 (LT-LT/one hour) and group 6 (LT-PT/one hour) ($p<0.05$; Table 4).

The shear bond strengths of group 9 (LT-LT/48 hours) were the highest measured in this study, which significantly differed with the values of the

Table 3: *Mean Shear Bond Strength Value (Standard Deviation [SD]) in MPa for Repaired Specimens*

	Luxatemp + Luxatemp	Luxatemp + Protemp	Luxatemp + Z350 (Flowable)	Luxatemp + Z350 (Packable)
10 min	3.6 (0.7)	3.3 (1.3)	7.4 (2.5)	8.0 (1.6)
1 h	7.1 (1.7)	6.8 (1.9)	9.0 (1.6)	11.1 (1.7)
48 h	18.7 (4.0)	11.7 (2.4)	14.0 (2.7)	12.7 (1.5)

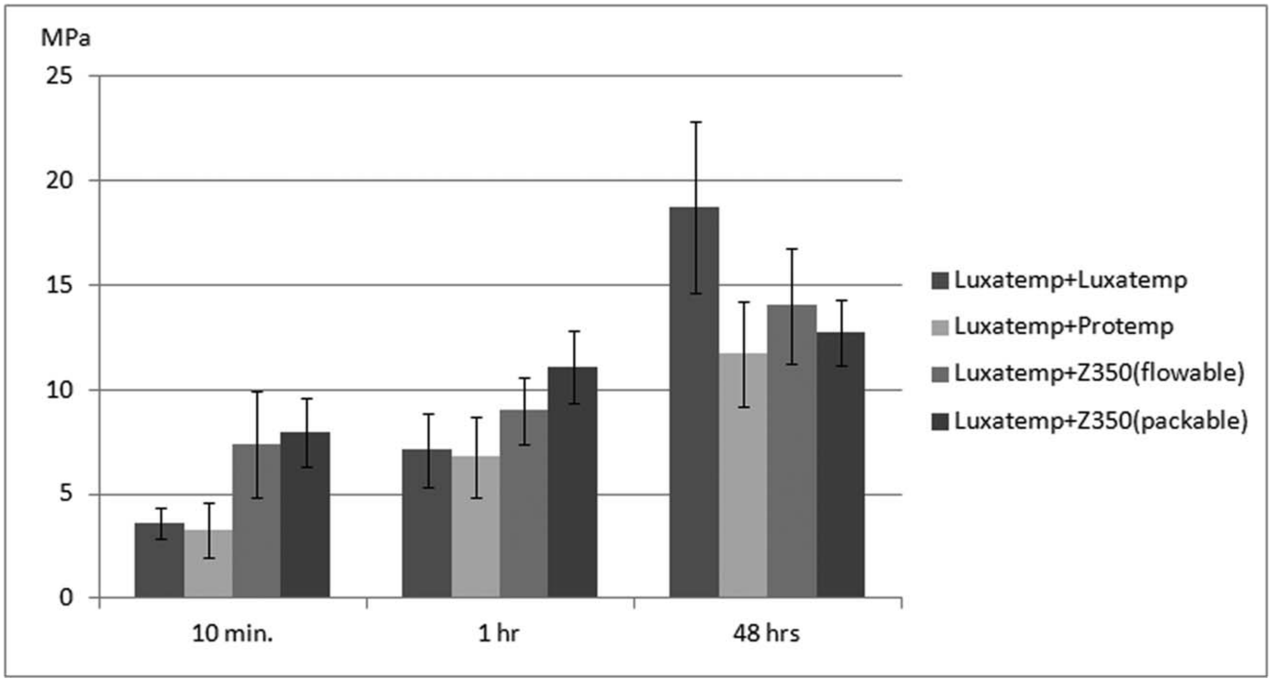

Figure 1. *The bond strengths at 10 minutes, one hour, and 24 hours of each of the repaired materials.*

other groups that were measured after 48 hours ($p<0.05$; Table 4).

All of the repaired materials showed increasing bond strengths with longer storage time. LT-LT and LT-PT revealed significantly increasing bond strengths at all period-of-time factors ($p<0.05$), and the largest differences were found with the LT-LT groups with respect to the storage time (Table 5).

Specimens showed three types of failure: adhesive (interface), cohesive (only at the provisional resin base material), and mixed (interface and base material). The distribution of fracture aspects on the debonded specimen surface is shown in Table 7. All of the groups measured after 10 minutes or one hour showed adhesive failure. While the groups measured after 48 hours showed adhesive, mixed,

Table 4: *Comparison Among Repaired Specimens with Measuring Times*[a]

	LT + PT	**LT + F**	**LT + P**
10 min			
LT + LT	n.s.	0.001	0.001
LT + PT	—	0.001	0.001
LT + F	—	—	n.s.
1 h			
LT + LT	n.s.	n.s.	0.006
LT + PT	—	n.s.	0.003
LT + F	—	—	n.s.
48 h			
LT + LT	0.002	0.048	0.025
LT + PT	—	n.s.	n.s.
LT + F	—	—	n.s.

Abbreviations: F, Z350 (flowable); LT, Luxatemp; P, Z350; PT, Protemp..
[a] *One-way analysis of variance (ANOVA) test (p values given in Table); n.s., not significant (p>0.05).*

Table 5: *Comparison Among Repaired Specimens with the Sorts of Materials*[a]

	1 h	**48 h**
LT + LT		
10 min	0.011	0.000
1 h	—	0.000
LT + PT		
10 min	0.004	0.000
1 h	—	0.000
LT + F		
10 min	n.s.	0.001
1 h	—	0.013
LT + P		
10 min	0.022	0.001
1 h	—	n.s.

Abbreviations: F, Z350 (flowable); LT, Luxatemp; P, Z350; PT, Protemp..
[a] *One-way analysis of variance (ANOVA) test (p values given in Table); n.s., not significant (p>0.05).*

Table 6: *Results of the Two-way Analysis of Variance (ANOVA) for All Test Groups*

Independent Variable	Significance
Material	$p < 0.001$
Storage time	$p < 0.001$

and cohesive failures, only group 9 (LT-LT/48 hours) exhibited cohesive failure (Figure 2).

DISCUSSION

The present study examined whether bond strengths between bis-acryl resin and light-cured packable composite resin are adequate to repair provisional restorations formed with bis-acryl and the effect of time elapsed after bonding on the bond strength at the repaired interface of provisional restorations. Light-cured packable composite resins seem to be appropriate for use as the repair material for bis-acryl provisional restorations. The bond strengths of all of the groups showed a tendency to increase with longer storage time, and, in particular, the bonding strength between Luxatemp base and Luxatemp dramatically increased.

Similar to results from previous studies,[9-11] light-cured flowable resin (Z350 flowable) showed higher bond strengths with bis-acryl resin (Luxatemp) compared to bis-acryl resin groups (Luxatemp, Protemp) upon measurement at 10 minutes after bonding. As the repaired provisional restorations usually receive a load immediately after bonding in the clinic, bond strength at 10 minutes after bonding may be the most valid index. It is for this reason that many articles[9,11,14] have recommended the use of light-cured flowable resin to repair provisional restorations made with bis-acryl. Additional advantages of using light-cured flowable resin include ease of application and manipulation, adequate working time, minimal odor, low polymerization shrinkage, and increased marginal accuracy.

For the shear strength tests at 10 minutes and one hour, the bond strengths of light-cured packable composite resins (Z350) were higher than for light-cured flowable resins (Z350 flowable). It can be presumed that the reason for this finding is that light-cured packable composite resin (Z350) has similar structure to light-cured flowable resin (Z350 flowable), and the material strength of these resins is higher than that of light-cured flowable resin (Z350 flowable). Light-cured packable composite resin has the additional virtues of low polymerization shrinkage and the ability to be molded prior to polymerization, compared with light-cured flowable resin. For these reasons, light-cured packable resin can be appropriately used to repair large defects of provisional restorations formed with bis-acryl.

The shear bond strength between bis-acryl resin and light-cured flowable resin was relatively low compared to the findings from previous studies.[9,15] A couple of reasons can be postulated for this discrepancy. First, to diminish differences of surfaces between groups, SiC paper (grit 600) was used in this study; however, the roughness of the SiC paper was relatively fine compared to that of the sandpaper used in previous studies.[15,16] Second, the crosshead speed of the knife-edge shearing rod was 2 mm/min in this study, which was faster than in previous studies.[15-17]

Most of the failure aspects were adhesive failures, except for the bonding between Luxatemp base and Luxatemp after 48 hours. All of the bonding failure aspects between Luxatemp base and Luxatemp after 48 hours were cohesive, and this group was found to have the highest bonding strength. Moreover, groups that showed mixed failure aspects had a relatively high bonding strength. These results suggest that

Table 7: *Distribution of Fracture Aspects on the Debonded Specimen Surface (N=Number)*

	Type of Failure	Luxatemp + Luxatemp	Luxatemp + Protemp	Luxatemp + Z350 (Flowable)	Luxatemp + Z350 (Packable)
10 min	Adhesive	10	10	10	10
	Mixed	—	—	—	—
	Cohesive	—	—	—	—
1 h	Adhesive	10	10	10	10
	Mixed	—	—	—	—
	Cohesive	—	—	—	—
48 h	Adhesive	—	6	7	6
	Mixed	—	4	3	4
	Cohesive	10	0	—	0

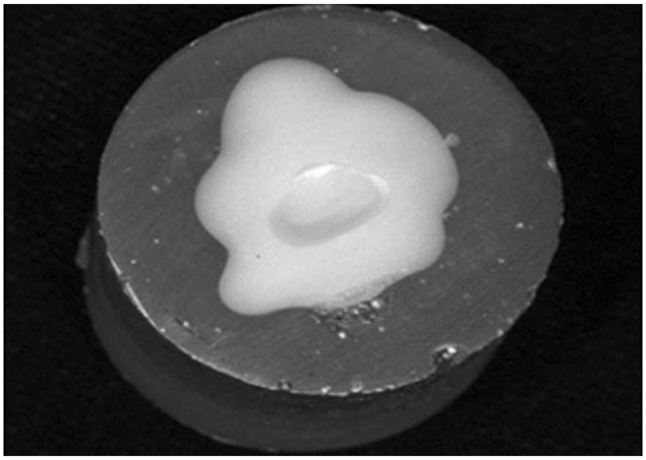

Figure 2. *All specimens from group 9 (LT-LT/48 hours) showed cohesive failure aspects.*

bonding strength at the interface is more important than the mechanical properties of the repair materials.

In general, the chemical similarity of the materials seems to be of great importance in polymer repair.[15] Using a provisional base and repair resins with similar chemical composition appears to provide greater bond strengths compared to using materials of dissimilar composition.[15] In contrast to this apparently common-sense rationale, the bond strength between bis-acryl and bis-acryl was considered to be weak. In this study, the same result was observed upon measurement at 10 minutes after bonding; however, the bond strength between bis-acryl resins rose dramatically with longer storage times. In particular, the bond between Luxatemp base and Luxatemp at 48 hours had the strongest bond strength measured in this study. This result suggests that bis-acryl and bis-acryl composites also have strong bonding but that a relatively long time is required to complete the bonding process. Expanded research about accelerating bonding procedures between bis-acryls may be useful in terms of the clinical use of bis-acryl.

The high bonding strength between Luxatemp and light-cured resin was to be expected because the chemical compositions of bis-acryl and light-cured resin bisphenol A diglycidyl ether dimethacrylate are similar.[18] The reason for the much higher bonding strength between Luxatemp and light-cured resin compared with Luxatemp base and Luxatemp at 10 minutes after bonding may correlate with the differences in polymerization speed: the polymerization of light-cured resin is accelerated with the light-curing machine and reaches complete polymerization in a shorter period of time than does Luxatemp.

To apply the results of this study to the clinical setting would require allowing bis-acryl resin polymerization to process for a relatively long period of time, and further studies to determine the exact time for complete bonding between bis-acryl resins will need to be performed. In addition, the development of methods to accelerate the polymerization of bis-acrylic resins would be a valuable extension of this work.

CONCLUSIONS

1. Light-cured packable composite resin is appropriate for repairing the large defects in provisional restorations formed with bis-acryl resin.
2. With longer storage time, the bond strengths of repaired bis-acryl provisional restorations tend to increase.
3. Strong bonding between bis-acryl base and bis-acryl can be accomplished, but a relatively long period of time is required for complete bonding.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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