

Effect of Repair Time Intervals and Surface Treatments on Shear Bond Strength of Bisacryl Resin Material Repairing with Flowable Composite Resin

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Abstract

The objective of this study was to investigate different repair time intervals by means of thermocycling and different surface treatments on shear bond strength of bisacryl resin material repairing with flowable resin composite. A total of 270 bisacryl resin specimens were randomly divided into 3 groups ($n = 90$), categorized by the numbers of thermocycling: (1) no cycle (stored in artificial saliva at 37 °C for 1 h), (2) 194 cycles (equivalent to 1 week in mouth) and (3) 5,000 cycles (equivalent to 6 months in mouth). After aging, the specimens in each group were subdivided into 3 subgroups ($n = 30$), categorized by the methods of surface treatment (no treatment, cylindrical carbide bur, cylindrical carbide bur and Adper™ Single Bond 2). The flowable composite resin (5 mm diameter, 4 mm height) was bonded to all surfaces of bisacryl resin specimens. Specimens were subjected to shear bond strength test by a universal testing machine with a crosshead speed of 0.5 mm/min. Data were analyzed by Two-way ANOVA and Turkey's test ($\alpha = 0.05$). Mode of failure was determined under a stereomicroscope. The highest mean shear bond strength was acquired from the carbide cylindrical bur and Adper™ Single Bond 2 group, whereas the no treatment group exhibited the lowest mean bond strength in all time intervals of repair. Besides, the result revealed that shear bond strength values were decreased when the number of thermocycling cycles increased, making the 5,000 cycle specimens had the lowest shear bond strength. Adhesive failure was the most predominant mode of failure. In conclusion, repair time intervals and surface treatments affect to the shear bond strength of bisacryl resin material repairing with flowable composite resin.

Keywords: Bisacryl resin/ Bisacrylic resin/ Bisacrylic composite/ Provisional restoration/ Repair

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Introduction

Provisional restoration is a critical component of Prosthodontics treatment. It is designed to enhance esthetic appearance and to provide pulpal protection, stabilization, and function before being replaced with definitive restoration. At present, bisacryl resin material has gained popularity as provisional material due to low exothermic reaction, low polymerization shrinkage,¹ good marginal adaptation,²⁻⁴ low wear resistance,² good color stability,² minimal pulpal irritation,⁵ excellent esthetic appearance, minimum unpleasant odor and glossary appearance mimicking natural tooth.⁶ Bisacryl resin is a hydrophobic material consisting of multifunctional substrate. During polymerization, it forms rigid structure of monomer chained cross-linkage, similar

to that of Bis-GMA.⁶ This cross-linkage leads to increase in strength, toughness and durability.³ Bisacryl resin materials can be categorized according to their modes of curing: auto-cured, dual cured and light cured polymerization.⁶ Most of bisacryl resin materials are now available as auto-mixed cartridge in order to make it easily handling and lesser chair time, but it is more costly.⁷ One of the key problems with this material when used in long time provisional restoration is its high rate of fractures during applying functional load. However, long term use of provisional restoration can evaluate tooth sensitivity, and potential pulp damage and also provide aid for definitive treatment planning and maintenance such as endodontic treatment, dental implant placement,

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gingival tissue healing in periodontal patients and orthodontic treatment.⁸⁻¹⁰ Apart from its benefits, complications in fabrication of bisacryl resin material as provisional restoration can occur, such as formation of void, undermargin, chipping or fracture of the material.¹¹ When these situations occurred, repair the defects or refabrication is required. Repair at the defective areas would be less time consuming and cost, compared to fabrication of new provisional restoration. Although, some studies showed that the strength of repaired bisacryl resin will be decreased half.^{12,13} Light-cured flowable resin composite has been suggested as material for bisacryl resin provisional restoration repairs.^{14,15} The use of light-cured flowable resin composite offers several advantages including the availability of several shades, ease of manipulation, ability to polymerize on demand, low cost, high accuracy and durable bonding with the bisacryl resin restoration.¹¹ Particularly, the light-cured flowable composite resin comes with a small tip which makes it possible to be repaired at inaccessible small areas and position. Previous studies evaluated the bond strength of experimental substance and bisacryl resin. The tests were performed after the bonding process and storage of specimens in water for 24-48 h.^{16,17} However, in most clinical situations, long term intra-oral use of this material for more than 48 h has never been found in any of the studies. Therefore, it is interesting to study time intervals of repair and surface treatment whether or not it affects the shear bond strength between bisacryl resin and flowable composite resin.

The objective of this study was to investigate different repair time intervals of bisacryl resin by means of thermocycling and different surface treatments on repair shear bond strength of bisacryl resin material with flowable composite resin.

Materials and methods

A stainless-steel base was used to create 270 cylindrical blocks for bisacryl resin material (Protemp™ 4; 3M ESPE, St. Paul, MN, USA). Polyvinyl chloride tube (PVC) was placed on stainless steel base and auto-cured clear epoxy resin was poured. Bisacryl resin material was injected into the space at the center of cylindrical block, covered with mylar strip and waited 5 mins for final setting according to the manufacturer's instruction. All bisacryl resin blocks were polished with 800-grit silicon carbide paper and then cleaned with distilled water and dried with compressed air. The specimens were stored in artificial saliva and randomly divided into 3 groups (n=90), categorized by the numbers of thermocycling: (1) no cycle (blocks stored in artificial saliva at 37 °C for 1 h), (2) 194 cycles (equivalent to 1 week in mouth) and (3) 5,000 cycles (equivalent to 6 months in mouth). In this experiment, the thermocycling was done in 5-55°C, dwell time of 30 s and transfer time of 2 s.¹⁸ After aging, the blocks of each group were subdivided into 3 subgroups (n=30), categorized by the methods of surface treatment as follows:

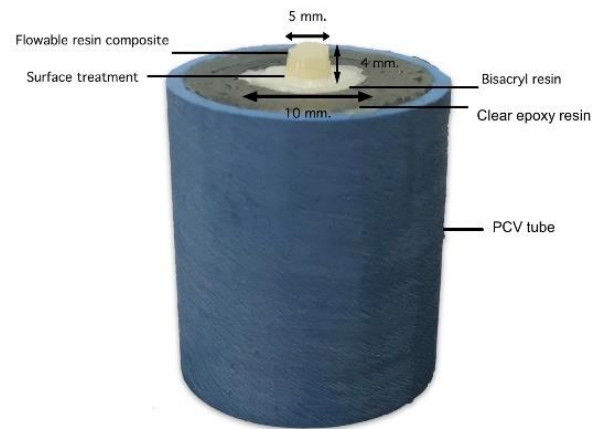
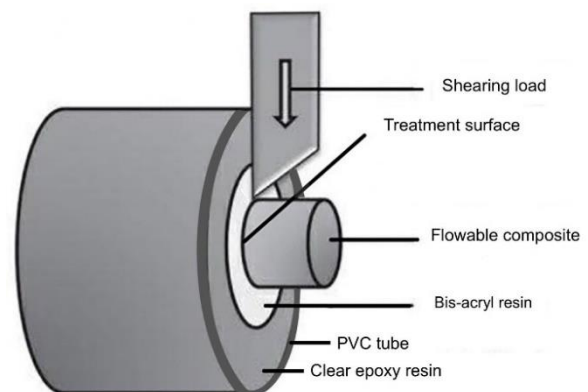
- Subgroup 1: no surface treatment (control group)
- Subgroup 2: bisacryl resin surface was grinded with 8-fluted, 0.9 mm diameter cylindrical carbide bur (Dentsply, Detrey Konstanz, Germany) with slow speed micromotor (40,000 rpm) in one direction toward operator for 5s (1 time/specimen) and air steam for 10s.
- Subgroup 3: bisacryl resin surface was grinded as described in subgroup 2. Then, the Adper™ Single Bond 2 (3M ESPE, St. Paul, MN, USA) bonding agent was applied to the surface with applicator for 15 s and was gently air thinned for 10 s and light cured by LED light-curing system for 20 s (Eliper S10, 3M ESPE, St. Paul, MN, USA) with 1,200 mW/cm² intensity (Table 1).

Table 1 Materials used in study

Materials	Products/ Manufacturers	Main composition	Batch numbers
Provisional material	Protemp™4 (A2 Shade)	Resin : Dimethacrylate polymer	628352 (base)
	3M ESPE, St. Paul, MN, USA	Bis-GMA	644848 (catalyst)
		Filler : Zirconium particles Silica and silane	
Repair material	Filtek™ Z350 XT Flowable Composite Resin (A3.5 Shade)	Resin : Bis-GMA, UDMA, PEGDMA	N941408
	3M ESPE, St. Paul, MN, USA	TEGDMA, Bis-EMA(6)Molecule	
		Filler : 20 nm silica filler, 4-11 zirconia filler, and aggregated zirconia/silica filler	
Surface treatment	Adper™ Single Bond 2	Bis-GMA, HEMA, dimethacrylate resin, polyalkenoic acid, Photo initiator, Ethanol, Water	N922607
	3M ESPE, St. Paul, MN, USA		

Abbreviation: Bis-GMA = Bisphenol A glycol dimethacrylate; UDMA= Urethane dimethacrylate; PEGDMA = Poly (ethylene glycol) dimethacrylate; Bis-EMA = ethoxylated bisphenol A glycol dimethacrylate; TEGDMA = triethyleneglycol-dimethacrylate; HEMA = 2-hydroxyethyl methacrylate

For each specimen, the flowable resin composite material (Filtek™ Z350 XT, 3M ESPE, St. Paul, MN, USA) was injected into cylindrical mold (5 mm diameter, 4 mm height) and light cured over the glass slide for 20 s (Figure 1). Specimens were subjected to shear bond strength test by a universal testing machine (EZ-S, SHIMADZU, Tokyo, Japan) with a crosshead 0.5 mm/min by placing a knife-edged blade adjacent and parallel to the adhesive interface between flowable composite resin and bisacryl resin material (Figure 2). The mode of failure was determined using a stereomicroscope (ML 9300; MEIJI, Saitama, Japan) classified into one of three types: Type I: Adhesive failure, Type II: Cohesive failure and Type III: Mixed failure. Data were then analyzed with Two-way ANOVA ($\alpha = 0.05$) to test for bond strength, substrate effects and its interaction by Statistical Package for the Social Sciences (SPSS 16.0; SPSS Inc., Chicago, IL, USA) and Turkey's test was used to determine differences between groups. The distribution of adhesive, cohesive and mixed fractures was analyzed using Chi-square test ($\alpha = 0.05$).

**Figure 1** Bisacryl resin was repaired with flowable resin composite**Figure 2** Shear bond strength testing with the universal testing machine at a crosshead speed of 0.5 mm/min.

Results

Mean shear bond strength and standard deviation for each repair group was reported (Figure 3) Two-way ANOVA revealed the highest mean shear bond strength was acquired from the carbide cylindrical bur and Adper™ Single Bond 2 group, whereas the no treatment group exhibited the lowest mean shear bond strength in all repair time intervals. Besides, the result revealed that shear bond strength values were decreased when the number of thermocycling cycles increased, making the 5,000 cycle specimens had the lowest shear bond strength for every surface treatment group while specimens in no cycle group showed the highest shear bond strength. In addition, significant differences in the mean shear bond strength were observed among no treatment, cylindrical carbide bur and cylindrical carbide bur combined with Adper™ Single Bond 2 in no cycle group and 194 cycles group. From the graph in Figure 3, although the no-treatment and 194-

cycles-of-thermocycling group had higher shear bond strength than the group without thermocycling but it increase insignificantly. In 5,000 cycles group revealed that there was no significant difference between group using carbide cylindrical bur and cylinder bur combined with Adper™ Single Bond 2. However, there was significant difference between no treatment group and surface treatment groups. The fracture surface examination with stereomicroscope revealed that most failures were adhesive failure (Table 2). The study found the group with no treatment had more significantly adhesive failure than cylindrical carbide bur treatment group and cylindrical carbide bur combined with Adper™ Single Bond 2 for all time intervals of repair. However, the numbers of adhesive failure for different surface treatment revealed insignificant difference among the specimens with the same time interval of repair.

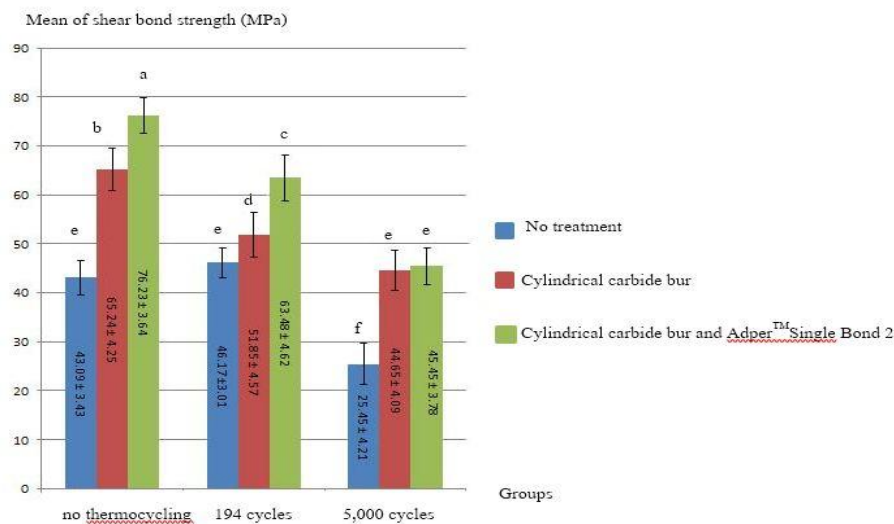


Figure 3 Bar graph for mean of shear bond strength (MPa). Error bars represent the standard deviations. *The same letter meant there were no significant differences between groups.

Table 2 Distribution of failure mode

	(no thermocycling)			(194 Thermocycling)			(5,000 Thermocycling)		
	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
No treatment	28 ^a	1	1	21 ^c	6	3	20 ^c	5	5
Grinding	27 ^a	3	0	23 ^c	7	0	22 ^c	5	3
Grinding+Bonding	21 ^c	6	3	24 ^c	4	2	24 ^c	4	2

Discussions

In this study, shear bond strength test was used to evaluate the bond strength between bisacryl resin and flowable composite resin. This method was found to better simulate the true bond strength of materials in clinical situations, especially for evaluating bond strength or surface treatment.^{12,16} Crack lines, wears or fractures of bisacryl resin provisional restoration may be detected after being exposed to oral environment, especially during functional period. The conditions in oral environment may lead to some changes on the surfaces of bisacryl resin materials. Thus, the time intervals of repair must be taken into consideration in deciding whether to repair it or not. Several studies have found that the bonding mechanism between aged repaired substrate with resin composite is through micro-mechanical retention.^{14,17,19} Roughness of the surface and the size of the filler particle provide possibilities for the resin to flow into undercuts and then to form micromechanical retention.^{20,21} Opdam et.al. reported that use of flowable composite resin for repair can reduce the void at interface and produces a better margin seal due to low consistency, superior to adapt of more viscous material and more retention through mechanical interlocking.²² In addition, other investigations have reported that surface debris and the viscosity of the resin composite filling can attribute to the reduction of repair bond strength.^{23,24} Different surface treatment methods have been proposed to improve repair strength, such as bur roughening, sandblasting and phosphoric acid treatment.^{21,25,26}

In this study, cylindrical carbide bur was chosen as a tool for making surface roughness of specimens because of its simplification and easiness. It imitates chair-side clinical situation, routinely performs provisional crown adjustment. Although in this study, the surface was not shown by scanning electron microscope, the surface of bisacryl resin that was grinded and passed air stream was noticeably

coarser than the non-grinded one. The results showed that, for all time intervals of repair, carbide cylindrical bur group had significantly higher shear bond strength than no treatment group. Previous studies have shown that micro-mechanical retention is the most significant factor in the resin composite repair.^{14,24} However, other studies have found repair bond strength reduction, which possible causes from surface debris or air inclusion.^{27,28} Other surface treatment, such as phosphoric acid, has only cleaning ability on treated bisacryl resin surface because it does not affect to the bond strength when repair with flowable resin composite.²⁶ A previous study has showed that surface treatment with sandblast technique on bisacryl resin prior to adhering with self-curing acrylic resin provided the highest microtensile and higher than no surface treatment and bonding application.²⁹ Söderholm and Roberts³⁰ concluded that surface roughness might enhance the ability of repair substrate to mechanically interlock into the initial substrate, because increased surface area is available for micro-mechanical bonding. Bonding agents can improve surface wetting and promote chemical bonding.^{11,12,31} The possible occurred mechanism is that the chemical bonds are formed between the resin matrix and exposed filler particles. As previously mentioned in the study of repairing composite restoration, it was suggested that the use of intermediate bonding agent has the major role for the resin composite repair success that was exposed to water or to a humid environment.³² Furthermore, it has been found that the coating surface with unfilled resin bonding agents (Bis-GMA/TEGDMA) before coating with the composite substrate can improve surface wetting and promote chemical bonding.^{12,33,34} The polar nature of phosphate groups on chlorophosphate ester of Bis-GMA bonding agents might contribute to bonding with inorganic filler component of composite.³⁵ In addition, Hydrophilic primers in bonding such as 2-hydroxyethylmethacrylate (2 - HEMA) molecule has the

ability to wet the old substrate by altering the surface tension and allowing deep penetration into pits, grooves and porosities of the component.³⁶ This study showed that the cylindrical carbide bur combined with Adper™ Single Bond 2 group had the highest shear bond strength in all repair time intervals and higher bond strength than the carbide cylindrical bur group and no treatment group, except in the 5,000 cycles test that found no significantly difference between the two methods of surface treatment. Although, the effect of bonding agents might contribute to an increase in micro-mechanical retention, leading to the improved mechanical interlocking and the combination of surface treatments and increased shear bond strength of bonding agent.^{37,38} The use of bonding can enhance the repair bond strength by promoting chemical coupling to the resin matrix on bisacryl resin and bonding to the exposed fillers, or micromechanical retention through monomer penetration into the matrix microcrack,³² but if specimens had been immersed in water for an extended period of time or several cycles of thermocycling, materials were inclined to take up water and water will break the chemical structures, such as in carboxyl and hydroxyl groups.³¹ Therefore, the hydrolytic stability of the bonding system had major importance for the success of resin composite repair restoration.³² Thermocycling or thermal cycling is one of the most widely used procedures to simulate the physiological aging in clinical practice. This method is conventionally used to simulate the *in vivo* aging of restorative materials by subjecting them to repeated cyclic exposure to hot and cold temperature, in a water bath to replicate thermal changes occurring in the oral cavity.¹⁸ It also supports the prediction of the of dental material longevity and also reduces the time consumed in conducting the experiment. In this study, the temperature used for thermocycling are between 5 and 55°C, following the ISO 11405 recommendation.³⁹ Restorations were found to become weaker when

they were exposed to the temperature change (5–55°C), similar to those found in the intraoral.^{40,41} Gale and Darvell postulated that approximately 10,000 thermal cycles correspond to 1 year of clinical function.²⁵ This estimate was based on a hypothesis that such cycles might occur 20 to 50 times a day, and this hypothesis has been accepted by several authors.^{42,43} In this study, specimens were placed in thermocycling bath at 5–55 °C, with dwell time of 30 s and transfer time of 2 s. A study comparing the effects of different physicochemical aging methods on the composite resin to those of composite resin on repair bond strength, aging the composite resin substrates through water storage for 2 months showed results similar to the group obtained with thermocycling and boiling in water.^{44,45} In this study, the group treated with bur or bur combined with bonding adhesive in no thermocycling group showed higher shear bond strength than that in 194 cycles and 5,000 cycles group. In addition, shear bond strength values were decreased when the number of thermocycling increases. Similarly, previous studies showed that lower bond strength values were observed after passing thermocycle as aging process.^{46,47} Absorption of water by diffusion process leads to leaching of unreacted monomers and swelling of the matrix.⁴⁸ It degrades the matrix-filler interface by hydrolytic breakdown between the interface and the surface of filler particle.³¹ Water broke chemical structure and the monomer functional group's radical activity was diminished. It was so structurally destroyed that even no one active group could react. It was shown in the research that the surface treatment with both grinding and grinding-with-bonding under 5,000 thermocycling cycles had no statistically significant difference. However, the results in group with 1 hour (no thermocycling) and no surface treatment were found insignificantly lower bond strength than the group with 194 cycles for the same treatment. Likewise, some studies have found the tendency for adhesion to be increased over

time.¹⁵⁻¹⁷ This tendency may be due to some experiments were tested in dry condition or were studied for a short period of time (24-48 h), in which the reaction was completely polymerized.¹⁵⁻¹⁷ The degree of polymerization of resin composite was found affect to the mechanical properties of the composite. Although, Polymerization kinetics is known that the polymerization of resin composite will reach its peak at about 24 h after the beginning of polymerization process.^{49,50} In no treatment and cylindrical carbide bur treatment, found the tendency of adhesion to be increased from 1 h to 194 thermocycling (1 week) but it did not find different in 5,000 thermocycling (6 months), but it had the opposite in cylindrical carbide bur with bonding treatment. Failure mode of no treatment and grinding groups were similar (Table 2). When aging was longer, there was less adhesive failures, and more cohesive failures occurred because of more time for absorbing water into the molecule. The repair adhesion was therefore less effective, especially at the interface of the two substances. However, for grinding with bonding group, the results showed no significant difference in adhesive failure under longer aging. The study found that group with no treatment had more adhesive failure specimens than cylindrical carbide bur treatment group and cylindrical carbide bur combined with Adper™ Single Bond 2 group for all repair time intervals. It means surface treatment can improve retention between two substrates by enhancing less adhesive failure or, in other words, more cohesive/mix failure. The cohesive failures were in the aged specimens, indicating that the bond strength was inferior to the inherent strength of the aged bisacryl resin.

Conclusions

Different time intervals of bisacryl resin repair affect to shear bond strength between bisacryl resin material and flowable resin composite. The longer use of bisacryl resin provisional crown is in the mouth,

the weaker repair bond strength is. The surface treatment of bisacryl resin with cylindrical carbide bur combined with bonding agent could provide higher repair shear bond strength than cylindrical carbide bur without any bonding agent and no treatment, respectively. When repair process is required, bisacryl resin surface should be treated by grinding or grinding and bonding agent especially in the case it has been placed in the mouth for a long time.

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ผลของช่วงเวลาในการซ่อมและการเตรียมผิวต่อค่าความแข็งแรงยึดเหนี่ยวของการซ่อมวัสดุบิสแอะคริลเรซินด้วยเรซินคอมโพสิตชนิดไหลแผ่

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บทคัดย่อ

การศึกษานี้มีวัตถุประสงค์เพื่อประเมินผลความแตกต่างของช่วงเวลาในการซ่อมแซมบิสแอะคริลเรซินโดยกระบวนการเทอร์โมไซเคิล และการเตรียมผิวต่อค่าความแข็งแรงยึดเหนี่ยวของวัสดุบิสแอะคริลเรซินและวัสดุเรซินคอมโพสิตชนิดไหลแผ่ บิสแอะคริลเรซินจำนวน 270 ชิ้น ถูกกลุ่มแบ่งเป็น 3 กลุ่ม กลุ่มละ 90 ชิ้น ตามจำนวนรอบของเทอร์โมไซเคิล กลุ่ม 1 ไม่มีรอบ ขึ้นงานเก็บในน้ำลายเทียม 37 องศาเซลเซียส 1 ชั่วโมง กลุ่ม 2 194 รอบ (เทียบเท่า 1 สัปดาห์ในปาก) กลุ่ม 3 5,000 รอบ (เทียบเท่า 6 เดือนในปาก) ภายหลังจากการจำลองการใช้งาน แบ่งชิ้นงานในแต่ละกลุ่มเป็น 3 กลุ่มย่อย กลุ่มย่อยละ 30 ชิ้น ตามการเตรียมผิว คือ ไม่มีการเตรียมผิว เตรียมผิวด้วยเข็มกรอคาร์ไบด์ทรงกระบอก การเตรียมพื้นผิวด้วยเข็มกรอคาร์ไบด์ทรงกระบอกและทาแอคเพอร์ซิงเกลบอนด์ยู ยึดวัสดุเรซินคอมโพสิตชนิดไหลแผ่ขนาดเส้นผ่านศูนย์กลาง 5 มิลลิเมตร สูง 4 มิลลิเมตร บนผิวบิสแอะคริลเรซินที่ถูกเตรียมผิวแล้ว นำชิ้นทดสอบมาหาค่าความแข็งแรงยึดเหนี่ยวภายใต้เครื่องทดสอบสากลที่ความเร็วหัวกดเท่ากับ 0.5 มิลลิเมตรต่อวินาที วิเคราะห์สถิติโดยใช้ความแปรปรวนสองทางและเปรียบเทียบเชิงซ้อนแบบทูกีย์ ที่ระดับความเชื่อมั่นร้อยละ 95 ตรวจสอบลักษณะความล้มเหลวที่เกิดขึ้นภายใต้กล้องสเตอริโอไมโครสโคป ค่าเฉลี่ยความแข็งแรงยึดเหนี่ยวสูงสุดพบในกลุ่มที่เตรียมผิวด้วยเข็มกรอคาร์ไบด์ทรงกระบอกร่วมกับแอคเพอร์ซิงเกลบอนด์ยู ขณะที่กลุ่มที่ไม่มีการเตรียมผิวแสดงค่าเฉลี่ยความแข็งแรงยึดเหนี่ยวต่ำสุดในทุกช่วงเวลาในการซ่อม นอกจากนี้การทำเทอร์โมไซเคิลจะลดค่าความแข็งแรงยึดเหนี่ยวในทุกวิธีการเตรียมผิว ชิ้นงานที่ผ่านเทอร์โมไซเคิล 5,000 รอบ มีค่าความแข็งแรงยึดเหนี่ยวต่ำสุด ลักษณะความล้มเหลวที่พบส่วนใหญ่คือการแตกหักแบบยึดติด สรุป ช่วงเวลาในการซ่อมแซมบิสแอะคริลเรซินและการเตรียมพื้นผิวมีผลต่อค่าความแข็งแรงยึดเหนี่ยวของการซ่อมบิสแอะคริลเรซินด้วยเรซินคอมโพสิตชนิดไหลแผ่

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