





Effect of Various Surface Treatments on the Repair Bond Strength of Nanofilled Composite to Composite

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Effect of Various Surface Treatments on the Repair Bond Strength of Nanofilled Composite to Composite

A Master's Thesis Submitted to the Department of Dentistry And the Graduate School of Yonsei University In partial fulfillment of the Requirements for the degree of Master of Dental Science

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June 2024



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마지막으로, 10 년이라는 긴 시간 동안 아낌없이 지원해주시고 응원해 주시는 아버지, 어머니, 그리고 동생에게도 감사와 사랑의 마음을 전합니다.

> 2024년6월 성이수



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Abstract

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This study aimed to evaluate how various treatments impact the repair bond strength of an aged nanofilled composite resin. One hundred thirty-five composite resin blocks were made with Filtek Z350XT (A3 body shade, 3M ESPE). All tested samples except the positive control group (n=15) underwent an aging procedure by 10,000 cycles of thermocycling. The aged samples were randomly assigned to groups based on mechanical



roughening methods: carbide bur, diamond bur, and sandblasting. Each group was then divided into halves, with one half receiving silane treatment and the other half remaining untreated. Single Bond Universal Adhesive (3M ESPE) was used for bonding agent. After 20 s of light curing, the same composite resin (Z350XT) was applied in 2 mm to the treated surface using a plastic mold with a bonding area of 4.45mm². Shear bond strength test and inspection of debonded surfaces were conducted.

The degree of conversion was also analyzed at three different points in time: immediately, after 24 hours, and after 10,000 cycles. Sixty composite resin blocks were additionally fabricated using a same nanofilled composite resin (A3 body shade, Filtek Z350XT) and microfilled composite resin (Shade A3, Metafil CX) (n=30 each).

Surface roughness analysis was also conducted to evaluate the roughness of surfaces treated with different mechanical methods : negative control, carbide bur, diamond bur, and sandblasting.

For statistical analysis, one-way ANOVA followed by Tukey's Post hoc test, one-way ANOVA with repeated measures followed by Bonferroni post-hoc tests, independent t-test, and Mann-Whitney U test was performed. In order to determine the relationship between surface roughness and shear bond strength, Pearson's correlation test was used. All significance levels were set at p = 0.05.

Groups that underwent sandblasting followed by silane treatment exhibited the highest mean shear bond strength. Only the groups that underwent sandblasting (SA and SA-S) had



bond strengths comparable to the positive control group. All other groups showed significantly lower bond strengths compared to the positive control group. All mechanically treated groups showed significantly higher bond strength to the negative control group, whereas only silane applicated group (S) did not. Failure analysis was conducted, revealing that the groups with the highest mean bond strength (SA and SA-S) exhibited a higher percentage of cohesive failures.

Results from FTIR showed a high conversion rate in Z350XT and limited number of available C=C bonds. Therefore, the repair bond strength primarily relied on mechanical retention.

A result of AFM showed that sandblasting yields significantly higher roughness values than negative control and carbide bur group. Moreover, a moderate positive correlation between shear bond strength and surface roughness was observed (p = .003; r = 0.522).

In conclusion, it is recommended that all aged composite resin surfaces be roughened through sandblasting and have silane applied before undergoing repair procedures.

Keywords: AFM; bond strength; composite resin repair; degree of conversion; FTIR; nanofilled composite resin; SEM; surface roughness; universal adhesive



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I. Introduction

Composite resin is widely accepted in clinical settings because of its many advantages, such as its ability to bond effectively to tooth structures, easy handling, superior mechanical characteristics, outstanding esthetics, and the growing popularity of minimal invasive approaches (Joulaei et al., 2012; Mamanee et al., 2015).



The oral cavity undergoes dynamic fluctuations in pH and temperature due to factors like diet, saliva, and aging, resulting in the degradation of composite resin (Sarkar, 2000; Suzuki et al., 2005). These changes put composite resin into various situations such as microleakage, discoloration, chipping, or fracture, potentially leading to replacement (Rinastiti et al., 2011). However, complete replacement could weaken the structure of the tooth or cause damage to the pulp, particularly when the existing composite resin remains clinically intact. Thus, repairing restorations emerges as a viable alternative to complete replacement (Padipatvuthikul & Mair, 2007; Papacchini et al., 2007). Repairing composite resin over complete replacement emerges as a more economical solution, contributing to prolonged longevity for both the tooth and the restoration (Fernández et al., 2015).

The successful repair of an aged composite resin primarily depends on establishing a strong bond with the pre-existing restoration (Hiraishi et al., 2005). Because of the reduced availability of C=C bonds for interaction with the new composite, achieving a strong adhesion to aged composite resin is challenging (Miletic & Santini, 2008). Thus, the success of repair depends on various factors, including surface characteristics (Papacchini et al., 2007), wettability (Rosales-Leal et al., 2001), roughness (Bouschlicher et al., 1997), and the methods used for surface conditioning (Ozcan et al., 2007; Papacchini et al., 2007). To increase the bond strength, several methods for surface conditioning have been explored in the current literature (Fawzy et al., 2008; Padipatvuthikul & Mair, 2007; Teixeira et al., 2005).



Nanofilled composite resins are known for their durability, low shrinkage, easy polishability, and excellent aesthetics (Jung et al., 2007; Turssi et al., 2006). However, according to previous studies, repairing nanofilled composites often fails to achieve the initial bond strength (Loomans, Cardoso, Opdam, et al., 2011; Staxrud & Dahl, 2011). This is due to the high conversion rate of polymerization in nanofilled composites, which consequently restricts the availability of unreacted C=C bonds (Staxrud & Dahl, 2011). Even after the initial polymerization, unreacted double bonds are still present, but as the material ages, its quantity decreases. This reduction may subsequently impair the adhesive properties of the resin, potentially compromising its bonding abilities (Fawzy et al., 2008). Consequently, in nanofilled composites, repair bond strength primarily relies on a mechanical retention (Staxrud & Dahl, 2011).

Surface roughness plays a crucial role in enabling micromechanical interlocking between composite surfaces (Kupiec & Barkmeier, 1996). This can be accomplished mechanically through various methods, including abrasive blasting with aluminum oxide particles (Cavalcanti et al., 2007; Yesilyurt et al., 2009), etching with hydrofluoric acid (Loomans, Cardoso, Roeters, et al., 2011), and with diamond burs (Cavalcanti et al., 2007; da Costa et al., 2012; Rathke et al., 2009).

In addition to micro-mechanical interlocking, repair bond strength can also be effectively achieved through chemical bonding (Brosh et al., 1997). Using a silane coupling agents improves a chemical bonding process between the fillers present in the aged and the new composite (Lung & Matinlinna, 2012; Matinlinna et al., 2004). A systematic review



and meta-analysis also confirmed that additional steps of silane application increases the repair bond strength of composite resins (Mendes et al., 2020).

Dental adhesives enhance surface wettability, which significantly increases the repair bond strength. (Burtscher, 1993). An introduction of universal adhesives has questioned the need for a separate silanization step. These adhesives commonly incorporate an acidic functional monomer known as 10-MDP along with silane. This presents a significant advantage in composite repair applications, particularly when dealing with varied cavity surfaces such as dentin, enamel, and composite materials (Chuenweravanich et al., 2022).

To date, an ideal method for repairing aged nanofilled composite resin restorations has not been determined (Mendes et al., 2020; Valente et al., 2016). Moreover, there are limited studies on the degree of conversion when repairing composite resin. Additionally, research on the relationship between bond strength and surface roughness remains scarce. Therefore, the purpose of this study is (a) to investigate the effect of various surface treatments on the repair bond strength of an aged nanofilled composite, (b) to measure the degree of conversion of the nanofilled composite resin before and after thermocycling, and (c) to evaluate the roughness of surfaces treated with different mechanical roughening methods.



II. Materials and methods

Materials used in this study, including their classification, manufacturers, compositions, and instructions for use, are shown in Table 1.

1. Shear bond strength

1.1. Specimen preparation

A total of one hundred thirty-five composite resin blocks were made using the a nanofilled composite resin (A3 body shade). The samples were prepared using a circular silicone mold (diameter : 10 mm, height : 2 mm). Each layer was placed in 2-mm increments and light-cured for 20 seconds using an LED curing light unit (Bluephase, Ivoclar Vivadent, Liechtenstein) with power intensity of 1200 mW/cm². Each composite resin block underwent an additional 20-second curing process from the top surface after removing the mold. To make a homogeneous surface, 400-grit carbide abrasive sheets were used to polish the top surfaces. This was achieved using a polishing machine (Ecomet 30, Buehler, Lake Bluff, IL, US) under tap water irrigation to eliminate contaminants. Then, all samples except the positive control group underwent an aging procedure by 10,000 cycles of thermocycling at 5-55°C for a dwell time of 30 s (R&B, Daejun, Korea) which equate to one year of aging in clinical settings (Morresi et al., 2014). After aging, using a polypropylene cylindrical mold (diameter : 10 mm, height : 2mm, Ultradent, South Jordan,



USA), all blocks were molded in an orthodontic acrylic resin (Ortho-Jet, Lang Dental Manufacturing Co. Inc.). Before further testing, the samples were kept in distilled water at 37°C for 24 hours and examined defects at the bonded interface with a stereomicroscope (SZ 61, Olympus Ltd., Tokyo, Japan) at ×30 magnification. Any flawed samples were discarded and replaced.

1.2. Surface treatments

Based on the surface treatments, aged samples were divided into seven experimental groups (n=15 each). Meanwhile, 30 samples were designated as negative and positive control group (n=15 each). Samples of positive control groups did not undergo any aging process or surface treatment. Table 2 shows the distribution of the sample groups.

Group CA and CA-S were treated using a fissure carbide bur (FG 701, Hager & Meisinger, Neuss, Germany), while group D and D-S were treated with a coarse grit diamond bur (FG850G 014, Hager & Meisinger, Neuss, Germany). Under water spray cooling, these samples were roughened with high-speed handpiece by moving the burs in a single direction. After every 10 working strokes, the burs were replaced. Group SA and SA-S were treated using a grit-blasting method with 50-µm aluminum oxide via an intraoral sandblaster (Microetcher, Henry Schein, New York, USA). After these three mechanical surface treatments, all samples were cleaned with 35% phosphoric acid (V-etch, Vericom, Gangwon-do, Korea). Then, Monobond N (Ivoclar Vivadent, Liechtenstein) and



Single Bond[®] Universal (3M ESPE, St. Paul, MN, USA) was applied to the designated groups. The application of phosphoric acid gel, sandblasting, the silane coupling agent, and the adhesive system were all carried out according to the steps described in Table 2.

1.3. Composite resin repair

Same type of composite resin (Z350XT) was then applied in 2 mm to the treated surface using a plastic bonding mold (Ultradent, South Jordan, USA) with a bonding area of 4.45mm². It was then light cured for 20 seconds. An excessive flash of bonding agent was removed with No.11 surgical blade (Paragon, Swann-Morton Limited, Sheffield, England).

1.4. Shear bond strength test (SBS)

A universal testing machine (Instron series IX, ITW, MA, USA) was used for the test. The samples were tested at a crosshead speed of 1.0 mm/min until breaks occurred (Figure 1), and the shear bond strengths were measured in newtons (N). The shear bond strength (MPa) was calculated with the following formula. The area of bonded surface was 4.45mm².

Shear bond strength (MPa) = $\frac{\text{Maximum load (N)}}{\text{Bonded surface (mm^2)}}$





Figure 1. Scheme of the shear bond strength test.

1.5. Evaluation of the failure modes

Following the shear bond strength test, the debonded surface was examined using a stereomicroscope (SZ 61, Olympus Ltd., Tokyo, Japan) at a magnification of ×30 to determine the type of failure. Failures were categorized into three types : adhesive failure, cohesive failure, and mixed failure. A field emission scanning electron microscope (JEOL-7800F, JEOL Ltd., Tokyo, Japan) was used to examine representative images of the debonded surfaces.



Materials	Main components	Instructions for use*	Lot number
Filtek [™] Z350XT (3M ESPE, St. Paul, MN, USA)	Silane Treated Ceramic; Silane Treated Silica; Silane Treated Zirconia; UDMA; BisEMA; BISGMA; PEGDMA; TEGDMA	Apply a layer of 2mm,	9533551
Metafil CX	UDMA (34 wt% organic TMPT filler (40 wt%) micro silica (26 wt%) photoinitiator (with aromatic tertiary amine) pigments	layer.	GS1
Single Bond® Universal (3M ESPE, St. Paul, MN, USA)	Bis-GMA; HEMA; DDMA; ethanol; Silane treated silica; water; 2-propenoic acid; 2- Methyl-; reaction products with 1,10-decanediol and phosphorous oxide; copolymer of acrylic and itaconic acid; DMAEMA; CQ; EDAB; 2,6-di-tert- butyl-P-cresol	Apply adhesive to the treated surface with rubbing action for 20 s and then direct a gentle stream of air to the surface for 5 s, light cure for 10 s.	30327B
V-etch (Vericom, Gangwon-do, Korea)	35% Phosphoric acid, synthetic amorphous silica as thickening agent, water	Apply to the treated surface for 30 s, and subsequently wash with air-water spray for 30 s, air dry for 10 s.	VT2N3100
Monobond N (Ivoclar Vivadent, Liechtenstein)	3-methacryl oxypropyltrimethoxysilane, ethanol, distilled water, acetic acid	Apply silane with a brush to the pre-treated surfaces. Allow the material to react for 60 seconds. Then, disperse any remaining excess with a strong stream of air.	Z03YD5
Aluminum oxide 50 µm (DynaFlex, Lake St. Louis, USA)	Alumina oxide 50 μm	At 2.5 bar pressure, 10mm distance for 15 s.	L12YD

Table 1. Composition and guidelines for the materials used in this study.



Group (n=15 each)	Mechanical surface treatment	Chemical surface treatment (Silane application)
NC (negative control)	None	Х
S	None	0
СА	Carbida hur	Х
CA-S	Carolae bui	0
D	Diamond hur	Х
D-S		0
SA	Sandblast	Х
SA-S	Sandolast	0
PC (positive control)	None	Х

Table 2. Distribution of the sample groups. Seven experimental groups according to the surface treatments (S:Silane, D:Diamond bur, SA:Sandblast, CA:Carbide bur). The negative control (NC) group did not undergo any mechanical or chemical treatment. Immediate resin placement was performed on the positive control (PC) group.



2. Degree of conversion (DC)

2.1. Specimen preparation

For the analysis of DC, thirty composite resin blocks were additionally fabricated using a nanofilled (A3 body shade) composite resin. Microfilled composite resin (shade A3, Metafil CX) was also fabricated as a control (n=30). Samples were fabricated using a circular silicon mold (diameter : 10 mm, height : 2mm) and only the top surfaces were light cured. 20 samples (n=10 each) were then light cured for 20 s, and underwent an aging procedure by 10,000 cycles of thermocycling. An additional 20 samples (n=10 each) were kept in light-proof containers under dry conditions at room temperature for 24 hours (Felix & Price, 2003). Remaining 20 samples (n=10 each) were analyzed immediately after light cured. Top surfaces of all samples were marked after light curing.

2.2. Fourier transformation infrared spectroscopy (FTIR) measurement

Degree of conversion was assessed on the polymerized samples using Fourier transform infrared spectroscopy (FTIR; NICOLET IS10, Thermo Fisher Scientific, Waltham, MA, USA). A measurement was taken at three different time intervals throughout the study : immediately after light curing, after 24-hour storage in dark room, and after 10,000 thermocycling. Top surfaces of all samples (n=10 each) were measured. The absorption spectra of the cured and uncured composite samples were collected in the 400–



4,000 cm⁻¹ wavelength range using 16 scans at a resolution of 4 cm⁻¹. By comparing the absorbance peak area linked to aliphatic C=C bonds (wavelength of 1,638 cm⁻¹) with the aromatic C=C bond (wavelength of 1,608 cm⁻¹), the percentage of unreacted C=C bonds (%) was calculated. The DC was then calculated using the following equation.

 $DC(\%) = 1 - \frac{Aliphatic C = C / Aromatic C = C (after curing)}{Aliphatic C = C / Aromatic C = C (before curing)} \times 100$



3. Surface roughness

3.1. Specimen preparation

A total of forty composite resin blocks were additionally made using a nanofilled composite resin (A3 body shade) for the analysis of surface roughness. The samples were fabricated in a same manner as described above (see 1.1. Specimen preparation) and underwent 10,000 cycles of thermocycling. All samples were randomly distributed into four groups according to the mechanical treatments (n=10) : negative control, carbide bur, diamond bur, and sandblasting. The specific procedures for each group are detailed above (see 1.2. Surface treatments). To ensure standardized conditions, care was taken to obtain a flat polished surface. Subsequently, all samples were rinsed with distilled water, subjected to ultrasonic cleaning for 15 minutes, and then kept in a dry condition at 37°C for 24 hours before further analysis.

3.2. Atomic force microscopy (AFM) analysis

To evaluate the surface morphology and roughness, a non-contact mode atomic force microscope (NC-AFM NX-10, Park Systems, Suwon, South Korea) with a particular cantilever, the OMCL-AC160TS (Park Systems, Suwon, South Korea) was used. The scan rate varied between 0.3 and 0.4 Hz, and the image resolution was fixed at 256×256 pixels. Double-sided carbon tape was used to firmly attach the composite resin samples to a metal



slide. Samples were positioned beneath the cantilever at randomly selected locations. From these acquired pictures, the average surface roughness (Ra) was then calculated.

3.3. Scanning electron microscopy (SEM) analysis

Four different composite resin samples that underwent mechanical treatment were additionally prepared for Scanning Electron Microscopy (SEM) analysis. The surfaces of the samples were mechanically roughened in the same manner as described above (see 3.1. Specimen preparation). These samples were subjected to an ion sputter carbon coating (LEICA EM ACE600), and the surface was assessed at magnifications of x1000, x3000, and x5000 using field emission SEM (MERLIN, ZEISS).





Figure 2. Schematic study design of SBS, FTIR, and AFM



4. Statistical analysis

All statistical analyses were conducted using SPSS 27 software (IBM Corp., New York, NY, USA). The normality of the shear bond strength (SBS), degree of conversion (DC), and surface roughness (Ra) was assessed using the Shapiro-Wilk test for equal variances. It was found that, except for the Ra, the data of SBS and DC were normally distributed. Therefore, comparative analysis of SBS was performed using one-way ANOVA followed by Tukey's post hoc test. Also, independent t-tests were performed to evaluate the effects of silane treatment within each mechanically treated group. To evaluate DC at different time intervals, one-way ANOVA with repeated measures and Bonferroni post-hoc tests was performed. To evaluate the statistical difference in surface roughness, a Mann-Whitney U test was performed. Pearson's correlation test was performed to evaluate the correlation between shear bond strength and surface roughness. All significance level was set at p = .05.



III. Results

1. Shear bond strength

The results are presented in Table 3 and Figure 3. Group SA-S exhibited the highest average bond strength, whereas group S showed the lowest average bond strength. The results of the one-way ANOVA indicated a significant difference between group SA-S and all other mechanically treated groups (p < 0.05). Groups SA-S and SA were the only ones comparable to the positive control (PC) group. Furthermore, significant differences were observed between all mechanically treated groups and the negative control (NC) group. Based on the independent t-test results, an additional silane application resulted in an increase in the bond strength within the same mechanically treated group. However, the difference was statistically significant only within the sandblasted group (p < 0.05).



	Group (n=15 each)			
	Mechanical treatment	Chemical treatment	Shear bond strength (MPa)	p value
NC	Nana	None	11.05±3.18ª	0.1(1
S	Inone	Silane	12.40±2.36 ^{ab}	0.101
CA	Contribution from	None	16.39±4.35 ^{bcd}	0.404
CA-S	- Carbide bur	Silane	18.10±5.45 ^{cd}	0.404
D	- Diamond hur	None	16.44±2.23 ^{bcd}	0.216
D-S	Diamond bur	Silane	17.80±2.59 ^{cd}	0.210
SA	C 41-14	None	19.68±3.81 ^{de}	0.022*
SA-S	Sanuolast	Silane	22.89±3.51°	0.032
PC	None	None	23.24±4.98 ^e	-

Table 3. Means and standard deviations of the shear bond strength (MPa). Different lowercase letters indicate a statistical difference (p < 0.05). * indicates a significant difference within each mechanically treated group by the independent t-test (p < 0.05).





Figure 3. Means and standard deviations of the shear bond strength (MPa). Different lowercase letters indicate a statistical difference (p < 0.05). Columns above the same horizontal line indicate no significant differences within each mechanically treated group, while * indicates a significant difference (p < 0.05).

2. Failure mode analysis

After the shear bond strength test, a stereoscopic microscope was used to examine the debonded surfaces. The distribution of failure modes is illustrated in Figure 4. The group SA-S exhibited the highest rate of cohesive failure (60.0%), followed by 53.3% in group SA. Cohesive failure rates in the other groups ranged from 20.0% to 40.0%. Representative images of the debonded surfaces were observed with SEM at \times 30 magnification (Figure 5).





Figure 4. Distribution of failure mode after shear bond strength test.





Figure 5. SEM images after shear bond strength test. Representative images of failure surfaces of composite resin using various repair protocols. (a) Adhesive failure in group D. (b) Adhesive failure in group NC. (c) Cohesive failure in Group CA. (d) Mixed failure in group SA.



3. Degree of conversion

The degree of conversion values at various time intervals are shown in Table 4. A significant difference was found between the immediate measurement and after 10,000 thermocycles for Filtek Z350XT. For Metafil CX (control), however, the values significantly increased at every measurement (p < 0.05). At every time interval, the conversion rate of Filtek Z350XT was higher than that of Metafil CX.

		Group (n=20 each)	
	Immediate	24h	10,000 thermocycling
Filtek Z350XT	69.62±0.29 ^{Aa}	$69.80{\pm}0.30^{ABa}$	$70.16{\pm}0.78^{\text{Ba}}$
Metafil CX	50.06±1.33 ^{Ab}	53.20±0.71 ^{Bb}	62.89 ± 1.18^{Cb}

Table 4. Degree of conversion. Different uppercase letters in a row indicate a statistical difference within the same material at different measurement times (p < 0.05). Different lowercase letters in a column indicate a statistical difference in the degree of conversion between two different materials at the same measurement time (p < 0.05).



4. Surface roughness

Table 5 presents the mean surface roughness values along with statistical comparisons regarding mechanical surface treatments. A significant difference in surface roughness was found between the sandblast group and both the negative control and carbide bur groups. Although not statistically significant, a slight increase was found in a following order : negative control group, carbide bur group, and diamond bur group. Representative 3D topographies of different mechanically treated groups are illustrated in Figure 6.

Type of surface treatments	Mean roughness value (±SD)
Negative control	0.28±0.11ª
Carbide bur	$0.40{\pm}0.09^{a}$
Diamond bur	$1.41{\pm}0.40^{ab}$
Sandblast	1.44±0.28 ^b

Table 5. Surface roughness. Different lowercase letters indicate statistical differences in surface roughness values (p < 0.05).





Figure 6. Representative images of AFM. a. AFM image of the negative control group. A nodular surface texture can be found on the surface. b. AFM image of the carbide bur group. Ridge and valley are found between nodules. c. AFM image of the diamond bur group. A surface texture similar to Figure. 6b is observed, but with deeper scratches and fewer nodules. d. AFM image of the sandblast group. The scratches are the deepest among all groups.

5. Scanning electron microscopy (SEM) analysis

SEM images displaying different surface textures produced by mechanical roughening are presented from Figures 7 to 10. In groups D and CA (Figure 8, 9), scratches, grooves, and a smear layer are observed. Meanwhile, a grit-blasting particles was noted in the sandblasted group (Figure 10).





Figure 7. Representative SEM images of the negative control group observed at x1000 and x3000 magnification. a. x1000 magnification image of the negative control group. Scratches and grooves filled with a smear matrix were detected. b. x3000 magnification of the negative control group. Loss of fillers is observed (red arrow).



Figure 8. Representative SEM images of the diamond bur group observed at x1000 and x3000 magnification. a. x1000 magnification image of the diamond bur group. Minor topographical alterations in the form of scratches induced by the diamond bur treatment. b. x3000 magnification image of the diamond bur group. Loss of fillers is observed (red arrow), while some smear matrix can be found (blue arrow).





Figure 9. Representative SEM images of the carbide bur group observed at x1000 and x3000 magnification. a. x1000 magnification image of the carbide bur group. A layer of matrix is found on most of the surfaces. b. x3000 magnification image of the carbide bur group. Loss of fillers is observed (red arrow), while a layer of smear matrix can be found (blue arrow).



Figure 10. Representative SEM images of the sandblast group observed at x1000 and x3000 magnification. a. x1000 magnification image of the sandblast group. A highly irregular topography was produced, with numerous grit-blasting particles. b. x3000 magnification image of the sandblast group. Aluminum oxide particles are found on the treated surface.



5. Correlation between SBS and surface roughness

Pearson's correlation analysis was used to determine whether there was a correlation between surface roughness and shear bond strength. A positive correlation was found between two variables (r = 0.522, moderate correlation, p < 0.01).



Figure 11. Correlation between SBS and surface roughness.



IV. Discussion

Repairing an aged composite resin can be challenging due to surface degradation processes such as hydroscopic/stress expansion, hydrolytic degradation, resulting in reduced unsaturated double bonds (Curtis et al., 2008; Miletic & Santini, 2008). These effects contribute to the lower shear bond strengths observed in aged composite groups compared to the positive control group.

When light-cured composite resins polymerize, the conversion of monomers is never fully complete (Peutzfeldt, 1997). Viscosity rises quickly when composite resin polymerizes, restricting free radicals inside the network of polymers (Mohamad et al., 2007). This phenomenon, known as the gel effect, occurs due to the reduced diffusion rate of components, thereby restricting complete polymerization (Obici et al., 2004). The degree of conversion in composite resins, especially Z350XT, is reported from 35% to 77% (Aleixo et al., 2014; Catalán et al., 2022). These results are in line with this study, which showed about 69% of conversion rate.

It is believed that composites having a higher degree of conversion than those with a lower conversion rate produce more rigid networks (Mohamad et al., 2007). As a result, in composites that are initially highly polymerized, it becomes more difficult for the remaining unpolymerized free radicals to engage in post-polymerization (Amorim et al., 2020). This was also confirmed by the results of this study. The initially high conversion



rate of Z350XT exhibited less increase after 24 hours of storage and thermocycling compared to Metafil CX, which had a lower initial conversion rate.

A final conversion rate is determined by its chemical structure, initial viscosity, and diffusion restrictions (Dickens et al., 2003; Lovell et al., 1999). Bis-GMA is the least flowable monomer, while UDMA is the most flowable due to its imino (-NH-) groups. With its lower viscosity and chemical structure, UDMA facilitates the post-polymerization reaction, resulting in an increased conversion rate. Meanwhile, when Bis-GMA is mixed with TEGDMA, a high conversion rate was observed compared to UDMA-based composites (Sideridou et al., 2002). The collaborative effects of Bis-GMA and TEGDMA explain the results of this study, which showed a higher conversion rate in Z350XT than in Metafil CX, which is based on UDMA.

A high conversion rate in Z350XT consequently restricts the availability of unreacted C=C bonds after thermocycling (Table 4). As a result, the repair of aged composite resin mostly depends on micro-mechanical retention (Staxrud & Dahl, 2011). Surface roughness is a well-known property that enhances micromechanical interlocking among the components of a material, thus improving its adhesive properties (Söderholm & Roberts, 1991).

Atomic force microscopy (AFM) has become popular for measuring the surface roughness of various materials (Covani et al., 2007; Kakaboura et al., 2007; Ko et al., 2007). AFM provides three-dimensional (3D) imaging at nanometric resolution without requiring



vacuum conditions or extensive specimen preparation. With this method, evaluating surface roughness has become reliable (Kakaboura et al., 2007). Non-contact AFM was used in this study to assess the surface roughness.

According to Rinastiti, M. et al. (Rinastiti et al., 2010), the surface roughness of composites increased significantly after silica coating, which aligns with the results of this study. By removing contaminated layers, this process makes it possible for surfaces to be roughened and mechanically combine with the adhesive. This, in turn, increases bonding potential by creating a larger surface area for adhesive interaction (Valandro et al., 2006). In Figure 6d and 10, sandblasting creates a surface with varying heights and sizes of peaks, resulting in a three-dimensional pattern that may enhance micromechanical retention. This implies that the adhesive area is greater compared to that achieved by using burs. Also, the result of surface roughness in this study showed that sandblasting yields significantly higher roughness values than negative control and carbide bur group. Furthermore, there was a moderate positive association found between surface roughness and bond strength (p = .003; r = 0.452).

To improve micro-mechanical retention, and consequently, repair bond strength, various surface treatments were performed. Results presented in Table 3 and Figure 3 demonstrate that various surface treatments significantly influenced repair bond strength. In the absence of surface treatment (group NC), the shear bond strength and surface roughness exhibited the lowest values. SEM analysis further validated these results, showing only minor changes in surface topography. These findings emphasize the



importance of mechanical treatment of aged composite surfaces before initiating any repair procedure. Previous studies also emphasized the importance of diamond bur abrasion or sandblasting for achieving micromechanical retention (Bonstein et al., 2005; Chuenweravanich et al., 2022; da Costa et al., 2012).

Non-aged composite resins were used as a positive control in this study. Our ultimate goal was to achieve similar bond strength to that of positive control group. Among all groups, only group SA-S exhibited bond strength comparable to the positive control group, significantly exceeding the repair bond strength of any other group. This suggests that sandblasting played a key role in the significant increase in bond strength, aligning with findings from other research studies (Brosh et al., 1997; Papacchini et al., 2007). The kinetic energy of air abrasion is directly related to the square of both the particles' mass and their velocity, with 50-µm particles preferred for composite resin repair (Loomans, Cardoso, Roeters, et al., 2011; Ritter et al., 2020). Consistent with its purpose of repair, we also selected 50-µm particles. The presence of aluminum oxide particles embedded after blasting can increase repair bond strength (Németh et al., 2023; Nishigawa et al., 2016). SEM image (Figure 10) clearly revealed remnants of aluminum on samples prepared with sandblasting. Previous studies of SEM observations suggested that a sandblasting with aluminum oxide produced a micro-mechanical surface, enhancing the surface available for bonding (Lucena-Martín et al., 2001; Shahdad & Kennedy, 1998).

Numerous studies on repair bond strength, reporting the distribution of failure modes, can be easily found. In a study by Imbery et al. (2014), cohesive failures were not reported,



possibly due to the lack of mechanical roughening in the tested samples (Imbery et al., 2014). In other studies using a nanofilled composite resin (Filtek Supreme XTE), a higher incidence of cohesive failures was observed in sandblasted groups compared to diamond-roughened or negative control groups, which is consistent with the findings of this study (Altinci et al., 2018; Fornazari et al., 2017). This findings is due to the adhesive force achieved with sandblasting being higher than the cohesive strength of the composite material (Nishigawa et al., 2016). Adhesive failures were frequently observed in the negative control group, leading to the lowest bond strength observed in the study.

The use of a silane coupling agent in composite resin repair improves chemical bonding by forming bonds between the fillers on the aged surface of composite resin and the newly applied composite resin (Hisamatsu et al., 2002; Lung & Matinlinna, 2012). In this study, an additional silane application step increased bond strength within each mechanically treated group, which aligns with a systematic review and meta-analysis conducted by Valente et al. (Valente et al., 2016). Silane contains a silanol group that bonds with alumina or silica on air-abraded surfaces to form siloxane links (Lung & Matinlinna, 2012; Matinlinna et al., 2004). This linkage potentially explains the statistical difference in shear bond strength between groups SA and SA-S, which are the only groups exhibiting a significant difference within the same mechanically roughened groups.

Yoshihara et al. revealed that the silane included in Single Bond Universal adhesive did not demonstrate comparable effectiveness to a separate silane application (Yoshihara et al., 2016). This phenomenon is due to instability of incorporated silane. The acidic nature



of this bonding agent promotes the process of hydrolysis. This characteristic indicates that an additional step involving the application of silane may be necessary when repairing composite resin. An increase in bond strength was observed in this study with an extra silanization step. This raises questions about the efficacy of the silane present in the universal adhesive. However, it would have been advantageous for this study to examine several bonding agents, including one with silane and another without it.

A limitation of this study is that it only used one kind of nanofilled composite resin, which may restrict the generalizability of the findings to other composite resins. Additionally, the reliance on thermocycling alone for artificial aging, while common in laboratory settings, may not fully replicate the clinical conditions. Therefore, further research involving clinical investigations is needed to explore the longevity of composite resin repair and to establish standardized repair protocols.



V. Conclusion

Within the limitations of this study, following conclusions can be derived:

1. Due to the high conversion rate observed in Z350XT, the repair procedure mostly relies on mechanical roughening of the aged surfaces.

2. Sandblasting with aluminum oxide followed by silane application yielded a comparable shear bond strength to that of non-aged positive control group after 10,000 thermocycling.

3. Surface Roughness showed a moderate positive correlation with bond strength, with the highest values found when the surface was sandblasted.



VI. References

- Aleixo, A. R., Guiraldo, R. D., Fugolin, A. P., Berger, S. B., Consani, R. L., Correr, A. B., Gonini-Júnior, A., & Lopes, M. B. (2014). Evaluation of contraction stress, conversion degree, and cross-link density in low-shrinkage composites. *Photomed Laser Surg*, 32(5), 267-273.
- Altinci, P., Mutluay, M., & Tezvergil-Mutluay, A. (2018). Repair bond strength of nanohybrid composite resins with a universal adhesive. *Acta Biomater Odontol Scand*, 4(1), 10-19.
- Amorim, B., Santana Vicentin, B., & Di Mauro, E. (2020). Post-polymerization reactivity of free radicals trapped in resin-based dental restorative materials by ESR spectroscopy. *Polymer Bulletin*, 77.
- Bonstein, T., Garlapo, D., Donarummo, J., Jr., & Bush, P. J. (2005). Evaluation of varied repair protocols applied to aged composite resin. *J Adhes Dent*, 7(1), 41-49.
- Bouschlicher, M. R., Reinhardt, J. W., & Vargas, M. A. (1997). Surface treatment techniques for resin composite repair. *Am J Dent*, *10*(6), 279-283.
- Brosh, T., Pilo, R., Bichacho, N., & Blutstein, R. (1997). Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites. *J Prosthet Dent*, 77(2), 122-126.
- Burtscher, P. (1993). Stability of radicals in cured composite materials. *Dent Mater*, 9(4), 218-221.

Catalán, A., Martínez, A., Muñoz, C., Medina, C., Marzialetti, T., Montaño, M.,



Jaramillo, A. F., & Meléndrez, M. F. (2022). The effect of preheating of nanofiller composite resins on their degree of conversion and microfiltration in dental fillings. *Polymer Bulletin*, *79*(12), 10707-10722.

- Cavalcanti, A. N., De Lima, A. F., Peris, A. R., Mitsui, F. H., & Marchi, G. M. (2007). Effect of surface treatments and bonding agents on the bond strength of repaired composites. *J Esthet Restor Dent*, 19(2), 90-98; discussion 99.
- Chuenweravanich, J., Kuphasuk, W., Saikaew, P., & Sattabanasuk, V. (2022). Bond Durability of a Repaired Resin Composite Using a Universal Adhesive and Different Surface Treatments. J Adhes Dent, 24(1), 67-76.
- Covani, U., Giacomelli, L., Krajewski, A., Ravaglioli, A., Spotorno, L., Loria, P., Das, S.,
 & Nicolini, C. (2007). Biomaterials for orthopedics: a roughness analysis by atomic force microscopy. *J Biomed Mater Res A*, 82(3), 723-730.
- Curtis, A. R., Shortall, A. C., Marquis, P. M., & Palin, W. M. (2008). Water uptake and strength characteristics of a nanofilled resin-based composite. *J Dent*, 36(3), 186-193.
- da Costa, T. R., Serrano, A. M., Atman, A. P., Loguercio, A. D., & Reis, A. (2012).
 Durability of composite repair using different surface treatments. *J Dent*, 40(6), 513-521.
- Dickens, S. H., Stansbury, J., Choi, K., & Floyd, C. (2003). Photopolymerization kinetics of methacrylate dental resins. *Macromolecules*, *36*(16), 6043-6053.

Fawzy, A. S., El-Askary, F. S., & Amer, M. A. (2008). Effect of surface treatments on the



tensile bond strength of repaired water-aged anterior restorative micro-fine hybrid resin composite. *J Dent*, *36*(12), 969-976.

- Felix, C. A., & Price, R. B. (2003). The effect of distance from light source on light intensity from curing lights. J Adhes Dent, 5(4), 283-291.
- Fernández, E., Martín, J., Vildósola, P., Oliveira Junior, O. B., Gordan, V., Mjor, I., Bersezio, C., Estay, J., de Andrade, M. F., & Moncada, G. (2015). Can repair increase the longevity of composite resins? Results of a 10-year clinical trial. J Dent, 43(2), 279-286.
- Fornazari, I. A., Wille, I., Meda, E. M., Brum, R. T., & Souza, E. M. (2017). Effect of Surface Treatment, Silane, and Universal Adhesive on Microshear Bond Strength of Nanofilled Composite Repairs. *Oper Dent*, 42(4), 367-374.
- Hiraishi, N., Nishiyama, N., Ikemura, K., Yau, J. Y. Y., King, N. M., Tagami, J., Pashley,
 D. H., & Tay, F. R. (2005). Water Concentration in Self-etching Primers Affects their Aggressiveness and Bonding Efficacy to Dentin. *Journal of Dental Research*, 84(7), 653-658.
- Hisamatsu, N., Atsuta, M., & Matsumura, H. (2002). Effect of silane primers and unfilled resin bonding agents on repair bond strength of a prosthodontic microfilled composite. *J Oral Rehabil*, 29(7), 644-648.
- Imbery, T., Gray, T., DeLatour, F., Boxx, C., Best, A., & Moon, P. (2014). Evaluation of Flexural, Diametral Tensile, and Shear Bond Strength of Composite Repairs. *Operative Dentistry*, 39(6), E250-E260.



- Joulaei, M., Bahari, M., Ahmadi, A., & Savadi Oskoee, S. (2012). Effect of Different Surface Treatments on Repair Micro-shear Bond Strength of Silica- and Zirconiafilled Composite Resins. J Dent Res Dent Clin Dent Prospects, 6(4), 131-137.
- Jung, M., Sehr, K., & Klimek, J. (2007). Surface texture of four nanofilled and one hybrid composite after finishing. Oper Dent, 32(1), 45-52.
- Kakaboura, A., Fragouli, M., Rahiotis, C., & Silikas, N. (2007). Evaluation of surface characteristics of dental composites using profilometry, scanning electron, atomic force microscopy and gloss-meter. *J Mater Sci Mater Med*, 18(1), 155-163.
- Ko, H. C., Han, J. S., Bächle, M., Jang, J. H., Shin, S. W., & Kim, D. J. (2007). Initial osteoblast-like cell response to pure titanium and zirconia/alumina ceramics. *Dent Mater*, 23(11), 1349-1355.
- Kupiec, K. A., & Barkmeier, W. W. (1996). Laboratory evaluation of surface treatments for composite repair. *Oper Dent*, 21(2), 59-62.
- Loomans, B. A., Cardoso, M. V., Opdam, N. J., Roeters, F. J., De Munck, J., Huysmans,M. C., & Van Meerbeek, B. (2011). Surface roughness of etched composite resin in light of composite repair. *J Dent*, 39(7), 499-505.
- Loomans, B. A., Cardoso, M. V., Roeters, F. J., Opdam, N. J., De Munck, J., Huysmans, M. C., & Van Meerbeek, B. (2011). Is there one optimal repair technique for all composites? *Dent Mater*, 27(7), 701-709.
- Lovell, L. G., Newman, S. M., & Bowman, C. N. (1999). The effects of light intensity, temperature, and comonomer composition on the polymerization behavior of



dimethacrylate dental resins. J Dent Res, 78(8), 1469-1476.

- Lucena-Martín, C., González-López, S., & Navajas-Rodríguez de Mondelo, J. M. (2001). The effect of various surface treatments and bonding agents on the repaired strength of heat-treated composites. *J Prosthet Dent*, 86(5), 481-488.
- Lung, C. Y., & Matinlinna, J. P. (2012). Aspects of silane coupling agents and surface conditioning in dentistry: an overview. *Dent Mater*, 28(5), 467-477.
- Mamanee, T., Takahashi, M., Nakajima, M., Foxton, R. M., & Tagami, J. (2015). Initial and long-term bond strengths of one-step self-etch adhesives with silane coupling agent to enamel-dentin-composite in combined situation. *Dent Mater J*, 34(5), 663-670.
- Matinlinna, J. P., Lassila, L. V., Ozcan, M., Yli-Urpo, A., & Vallittu, P. K. (2004). An introduction to silanes and their clinical applications in dentistry. *Int J Prosthodont*, 17(2), 155-164.
- Mendes, L. T., Loomans, B. A. C., Opdam, N. J. M., Silva, C. L. D., Casagrande, L., & Lenzi, T. L. (2020). Silane Coupling Agents are Beneficial for Resin Composite Repair: A Systematic Review and Meta-Analysis of In Vitro Studies. *J Adhes Dent*, 22(5), 443-453.
- Miletic, V. J., & Santini, A. (2008). Remaining unreacted methacrylate groups in resinbased composite with respect to sample preparation and storing conditions using micro-Raman spectroscopy. *J Biomed Mater Res B Appl Biomater*, 87(2), 468-474.



- Mohamad, D., Rj, Y., & Mann, A. B. (2007). Post-polymerization of dental resin composite evaluated with nanoindentation and micro- Raman spectroscopy.
- Morresi, A. L., D'Amario, M., Capogreco, M., Gatto, R., Marzo, G., D'Arcangelo, C., & Monaco, A. (2014). Thermal cycling for restorative materials: does a standardized protocol exist in laboratory testing? A literature review. *J Mech Behav Biomed Mater*, 29, 295-308.
- Németh, K. D., Told, R., Szabó, P., Maróti, P., Szénai, R., Pintér, Z. B., Lovász, B. V., Szalma, J., & Lempel, E. (2023). Comparative Evaluation of the Repair Bond Strength of Dental Resin Composite after Sodium Bicarbonate or Aluminum Oxide Air-Abrasion. *Int J Mol Sci*, 24(14).
- Nishigawa, G., Maruo, Y., Irie, M., Maeda, N., Yoshihara, K., Nagaoka, N., Matsumoto, T., & Minagi, S. (2016). Various effects of sandblasting of dental restorative materials. *PloS one*, *11*(1), e0147077.
- Obici, A. C., Sinhoreti, M. A. C., Frollini, E., Correr Sobrinho, L., & Consani, S. (2004). Degree of conversion of Z250 composite determined by fourier transform infrared spectroscopy: comparison of techniques, storage periods and photo-activation methods. *Materials Research*, 7, 605-610.
- Ozcan, M., Barbosa, S. H., Melo, R. M., Galhano, G. A., & Bottino, M. A. (2007). Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. *Dent Mater*, *23*(10), 1276-1282.

Padipatvuthikul, P., & Mair, L. H. (2007). Bonding of composite to water aged composite



with surface treatments. Dent Mater, 23(4), 519-525.

Papacchini, F., Dall'Oca, S., Chieffi, N., Goracci, C., Sadek, F. T., Suh, B. I., Tay, F. R., & Ferrari, M. (2007). Composite-to-composite microtensile bond strength in the repair of a microfilled hybrid resin: effect of surface treatment and oxygen inhibition. *J Adhes Dent*, 9(1), 25-31.

- Peutzfeldt, A. (1997). Resin composites in dentistry: the monomer systems. *European Journal of Oral Sciences*, *105*(2), 97-116.
- Rathke, A., Tymina, Y., & Haller, B. (2009). Effect of different surface treatments on the composite-composite repair bond strength. *Clin Oral Investig*, 13(3), 317-323.
- Rinastiti, M., Ozcan, M., Siswomihardjo, W., & Busscher, H. J. (2010). Immediate repair bond strengths of microhybrid, nanohybrid and nanofilled composites after different surface treatments. *J Dent*, 38(1), 29-38.
- Rinastiti, M., Özcan, M., Siswomihardjo, W., & Busscher, H. J. (2011). Effects of surface conditioning on repair bond strengths of non-aged and aged microhybrid, nanohybrid, and nanofilled composite resins. *Clin Oral Investig*, 15(5), 625-633.
- Ritter, A., Sulaiman, T., Altitinchi, A., Baratto-Filho, F., Gonzaga, C., & Correr, G.
 (2020). Effect of Tribochemical Coating on Composite Repair Strength. *Operative Dentistry*, 45(6), E334-E342.
- Rosales-Leal, J. I., Osorio, R., Holgado-Terriza, J. A., Cabrerizo-Vílchez, M. A., & Toledano, M. (2001). Dentin wetting by four adhesive systems. *Dent Mater*, 17(6), 526-532.



- Sarkar, N. K. (2000). Internal corrosion in dental composite wear. *J Biomed Mater Res*, 53(4), 371-380.
- Shahdad, S. A., & Kennedy, J. G. (1998). Bond strength of repaired anterior composite resins: an in vitro study. *J Dent*, 26(8), 685-694.
- Sideridou, I., Tserki, V., & Papanastasiou, G. (2002). Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins. *Biomaterials*, 23(8), 1819-1829.
- Söderholm, K. J., & Roberts, M. J. (1991). Variables influencing the repair strength of dental composites. *Scand J Dent Res*, 99(2), 173-180.
- Staxrud, F., & Dahl, J. E. (2011). Role of bonding agents in the repair of composite resin restorations. *Eur J Oral Sci*, 119(4), 316-322.
- Suzuki, S., Ori, T., & Saimi, Y. (2005). Effects of filler composition on flexibility of microfilled resin composite. J Biomed Mater Res B Appl Biomater, 74(1), 547-552.
- Teixeira, E. C., Bayne, S. C., Thompson, J. Y., Ritter, A. V., & Swift, E. J. (2005). Shear bond strength of self-etching bonding systems in combination with various composites used for repairing aged composites. *J Adhes Dent*, 7(2), 159-164.
- Turssi, C. P., Ferracane, J. L., & Ferracane, L. L. (2006). Wear and fatigue behavior of nano-structured dental resin composites. *J Biomed Mater Res B Appl Biomater*, 78(1), 196-203.

Valandro, L. F., Özcan, M., Bottino, M. C., Bottino, M. A., Scotti, R., & Bona, A. D.



(2006). Bond strength of a resin cement to high-alumina and zirconia-reinforced ceramics: the effect of surface conditioning. *Journal of Adhesive Dentistry*, 8(3).

- Valente, L. L., Sarkis-Onofre, R., Gonçalves, A. P. R., Fernández, E., Loomans, B. A. C., & Moraes, R. R. d. (2016). Repair bond strength of dental composites: systematic review and meta-analysis. *International Journal of Adhesion and Adhesives*, 69, 15-26.
- Yesilyurt, C., Kusgoz, A., Bayram, M., & Ulker, M. (2009). Initial repair bond strength of a nano-filled hybrid resin: effect of surface treatments and bonding agents. J Esthet Restor Dent, 21(4), 251-260.
- Yoshihara, K., Nagaoka, N., Sonoda, A., Maruo, Y., Makita, Y., Okihara, T., Irie, M., Yoshida, Y., & Van Meerbeek, B. (2016). Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives. *Dent Mater*, 32(10), 1218-1225.



Abstract (In Korean)

다양한 표면 처리에 대한 나노필러 복합 레진 간 수리 결합 강도 연구

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(지도교수 노 병 덕)

본 연구의 목적은 나노필러형 복합레진의 수리 접착 강도에 미치는 다양한 표면 처리 효과를 평가하기 위한 연구이다. 135 개의 A3 body 색상의 나노필러형 복합레진(Filtek Z350XT)블록을 제작하였다. 양성 대조군을 제외한 모든 시편은 10,000 번의 열 순환 처리 과정을 거쳤다. 열 순환 처리 후 모든 시편은 표면 처리 유형에 따라 하나의 음성 대조군과 세 가지 기계적 표면 처리 (카바이드 버, 다이아몬드 버, 샌드블라스트)로 구분되었다. 그 후 각 그룹에서 절반은 실란



처리를, 남은 절반의 시편은 실란 처리를 하지 않았다. 이후, 처리된 표면에 플라스틱 몰드를 사용하여 동일한 유형의 복합레진(Z350XT)을 2mm 두께로 적용시킨 후 전단 접착 강도 시험을 시행하였다. 파절 양상은 광학현미경 하에서 관찰하였으며, 기계적 처리만 이루어진 시편의 각 표면을 주사전자현미경을 통해 관찰하였다.

나노필러형 복합레진의 중합율을 측정하기 위해서 60 개의 A3 body 색상의 나노필러형 복합레진 (Filtek Z350XT, n=30)을 추가로 제작하였다. 마이크로필러형 복합레진 (Metafil CX, n=30)은 대조군으로 사용되었다. 열 순환 처리 전과 후의 사용 가능한 C=C 결합의 수를 확인하기 위해 푸리에 변환 적외선을 이용한 복합레진 중합율 측정을 시행하였다.

또한, 접착 강도와의 상관 관계 분석을 위해 표면 거칠기를 측정하였다. 40개의 A3 body 색상의 나노필러형 복합레진 (Filtek Z350XT) 블록을 추가 제작하였다. 이후 열 순환 처리 및 기계적 표면 처리 후 표면 거칠기 측정을 하였다.

통계 분석으로는 Tukey's post hoc test 를 이용한 일원분산 분석, Bonferroni post hoc test 를 이용한 반복 측정 일원분산 분석, 독립 표본 T 검정, 그리고 Mann-Whitney U 검정을 시행하였다. 접착 강도와 표면 거칠기 간의 상관 관계는 Pearson 의 상관 분석을 이용해 평가되었다. 모든 검정의 유의 수준은 *p* < 0.05 로 설정하였다.



나노필러형 복합레진의 높은 중합률과 열 순환 처리 후 사용 가능한 C=C 결합이 제한적임을 확인하였다. 따라서 나노필러형 복합 레진의 수리 접착 강도의 대부분은 미세 기계적 구조에 의존한다. 가장 높은 표면 거칠기는 샌드블라스트로 표면 처리한 그룹에서 관찰되었다. 또한, 접착 강도와 표면 거칠기 간에 중간 정도의 양의 상관 관계가 관찰되었다 (*p* = .003; r = 0.452). 가장 높은 평균 전단 접착 강도는 SA-S 그룹에서 나타났다. 기계적 처리를 시행한 모든 그룹은 음성 대조군에 비해 유의하게 높은 접착 강도를 보였다. 화학적 처리만 시행한 그룹 (S 그룹)은 음성 대조군과 유의한 차이가 없었다. 가장 높은 평균 접착 강도를 보인 SA-S, SA 그룹에서 응집성 파절 양상의 비율이 다른 그룹에 비해 높았다.

열 순환 처리 후 사용 가능한 C=C 결합은 제한적이며, 기계적 처리를 시행한 모든 그룹에서 음성 대조군에 비해 개선된 접착 강도를 나타내었다는 점에서 복합레진 수리 전에는 반드시 기계적 처리가 필요하다. 특히 샌드블라스트와 실란 적용을 함께 시행할 경우, 높은 수준의 접착 강도를 기대할 수 있을 것으로 생각된다.

핵심 되는 말 : 나노필러형 복합레진; 복합레진 수리; 원자힘현미경; 유니버설 접 착제; 접착결합강도; 주사전자 현미경; 중합율; 표면 거칠기; 푸리 에 변환 적외선

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