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RESEARCH ARTICLE

The effect of sodium ascorbate application and intracoronal bleaching on the shear bond strength of composite resin

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ABSTRACT

An increase in the frequency of 35% hydrogen peroxide (HP) application during intracoronal bleaching leads to a higher accumulation of free radical residues, which can negatively affect the bonding of composite resin. The removal of these free radicals can be facilitated by the application of 35% sodium ascorbate (SA) combined with 0.4% surfactant. An additional frequency of SA application is required to counteract the increased free radical residues. This study aimed to evaluate the effect of the frequency of 35% SA combined with 0.4% surfactant and different intracoronal bleaching frequencies on the shear bond strength of composite resin. Forty-eight premolar teeth were divided into two main groups of 24 specimens each. Group I received two applications of 0.01 mL HP 35%, while Group II received three applications. After each bleaching session, the specimens were stored in a closed tube for five days per session, then washed and dried. Each group was further divided into three subgroups. Subgroup A was left untreated for seven days; Subgroup B received two applications of 35% sodium ascorbate combined with 0.4% surfactant; and Subgroup C received three applications. All specimens were restored with composite resin and immersed in pH 7 artificial saliva in an incubator for seven days. Shear bond strength was tested using a Universal Testing Machine at a crosshead speed of 0.5 mm/min. Two-way ANOVA revealed a significant effect of sodium ascorbate application frequency and different intracoronal bleaching frequencies on composite resin shear bond strength ($p < 0.01$). The results indicate that the shear bond strength of composite resin subjected to three applications of 35% sodium ascorbate combined with 0.4% surfactant is statistically comparable to those obtained with two applications of the same formulation.

Keywords: hydrogen peroxide; sodium ascorbate; surfactant; shear bond strength

INTRODUCTION

Tooth discoloration is an aesthetic issue that can significantly impact an individual's self-confidence. One of the common causes of tooth discoloration is pulp necrosis. Discoloration occurs due to the decomposition of bacterial metabolic products and pulp necrosis byproducts that infiltrate the dentinal tubules. Intracoronal bleaching is considered one of the most conservative treatment options for addressing tooth discoloration.[1] This procedure involves the use of oxidizing agents such as hydrogen peroxide (5-38%), which are placed inside the pulp chamber for 3-7 days and can be repeated two to four times until the desired results are achieved.[1-3]

Hydrogen peroxide works by generating free radicals that break the double bonds in the chromophore molecules of organic pigments within the tooth structure. This process transforms the pigments into simpler and lighter molecules, resulting in a whitening effect of the teeth.[4,5] The effectiveness of bleaching is influenced by several factors, including the initial tooth color, patient age, gender, the type of bleaching agent used, and application methods such as enamel prophylaxis, contact time, and application frequency.[6-8]

Despite its effectiveness, intracoronal bleaching has several side effects, such as tooth structure damage, cervical resorption, and compromised resin composite bonding. Higher concentrations of bleaching agents can increase the risk of these side effects.[9] One approach to minimize these negative impacts is to perform resin composite restoration after a waiting period of 1-4 weeks post-bleaching. This waiting period is necessary to eliminate residual free radicals that may interfere with the bonding between the resin composite and tooth structure.[1,9]

Antioxidants such as sodium ascorbate can eliminate residual free radicals and enhance the bonding strength of resin composites. The application of 35% sodium ascorbate twice, with an interval of 5-10 minutes, has been proven effective in restoring bonding strength to enamel post-bleaching.[10,11] However, sodium ascorbate has limited penetration due to its high water solubility but low lipid solubility. Therefore, the addition of surfactants is required to improve its efficacy.[12,13]

Surfactants are substances that enhance the penetration of active agents by reducing the surface tension of liquids.[14] Surfactant molecules contain hydrophilic and hydrophobic groups, enabling them to interact with liquids and eliminate free radicals.[15] Previous research by Yulianasari et al. demonstrated that a combination of 35% sodium ascorbate with 0.4% surfactant resulted in better bond strength of resin composites compared to using sodium ascorbate without surfactant or with lower surfactant concentrations.[16] Similar findings were reported by Anindita, who found that the application of 35% sodium ascorbate with 0.4% surfactant provided higher shear strength of resin composites.[17]

Shear bond strength is a fundamental parameter for evaluating the integrity of the adhesive interface between restorative materials and dental substrates. It is defined as the maximum stress that a bonded assembly can endure before debonding under shear loading conditions.[18] This parameter reflects the quality of adhesion and the mechanical stability of restorations under functional stress, and its evaluation includes adhesive, cohesive, and mixed failure modes that provide insight into the mechanisms of bond failure.

The frequency of bleaching procedures has been reported to influence the accumulation of residual free radicals, which can adversely affect tooth structure and bonding performance. In clinical practice, multiple bleaching cycles are often required to achieve optimal esthetic results. However, studies by Anindita et al (2023) and Yulianasari et al (2022) were limited to a single application of 35% hydrogen peroxide followed by a single application of 35% sodium ascorbate combined with 0.4% surfactant, leaving uncertainty regarding the optimal antioxidant application frequency, particularly under repeated bleaching conditions. Based on this background, the present study aims to evaluate the effect of different frequencies of 35% sodium ascorbate combined with 0.4% surfactant application on the shear bond strength of resin composites under varying intracoronal bleaching frequencies.

MATERIALS AND METHODS

This *in vitro* study has received ethical approval from the Research Ethics Commission of the Faculty of Dentistry - RSGM UGM Prof. Soedomo, Universitas Gadjah Mada, Yogyakarta with approval number 104/UN1/KEP/FKG-RSGM/EC/2024. The research subjects used in this study consisted of 48 premolar teeth. The specimens were randomly divided into two groups based on bleaching frequency (two bleaching sessions and three bleaching sessions), and each of these groups was further divided into three subgroups (7-day delay; application of 35% sodium ascorbate combined with 0.4% surfactant for two or three times). This resulted in six experimental groups, with each group consisting of eight teeth. Group IA (two bleaching sessions + 7-day delay), Group IB (two bleaching sessions + two applications of sodium ascorbate combined with surfactant), Group IC (two bleaching sessions + three applications of sodium ascorbate combined with surfactant), Group IIA (three bleaching sessions + 7-day delay), Group IIB (three bleaching sessions + two applications of sodium ascorbate combined with surfactant), and Group IIC (three bleaching three sessions + three applications of sodium ascorbate combined with surfactant).

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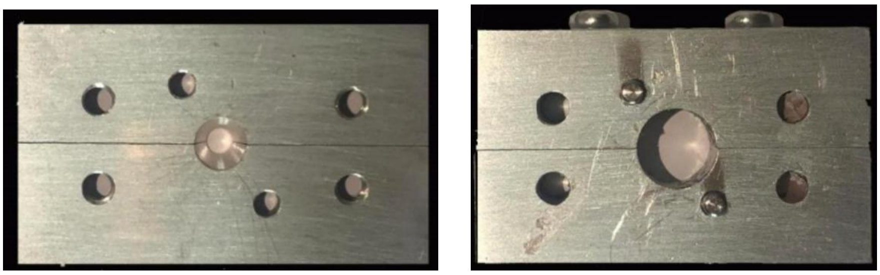
The 35% sodium ascorbate antioxidant gel combined with 0.4% surfactant was prepared following the protocol established at the Faculty of Pharmacy, Universitas Gadjah Mada. Ten milliliters of distilled water were placed in a water bath at 50 °C. Once cooled, 3.5 grams of sodium ascorbate powder (Sigma-Aldrich, USA) was added, followed by the addition of 0.04 mL of surfactant (Tween® 80, Sentra Chemical Indonesia) using a micropipette and stirred until homogeneous. The gel was then stored in a refrigerator at 4 °C until use.

A composite resin molding device (Mold A) was fabricated from metal for shaping the composite resin specimens. The mold is made of metal, with dimensions of 50 mm in length, 50 mm in width, and 4 mm in height. The center of the mold



(A) (B)

Figure 1. (A) Molding tool A for molding composite resin, (B) Molding tool B for tooth fixation

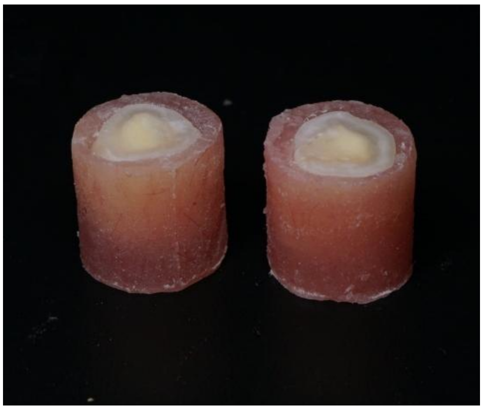
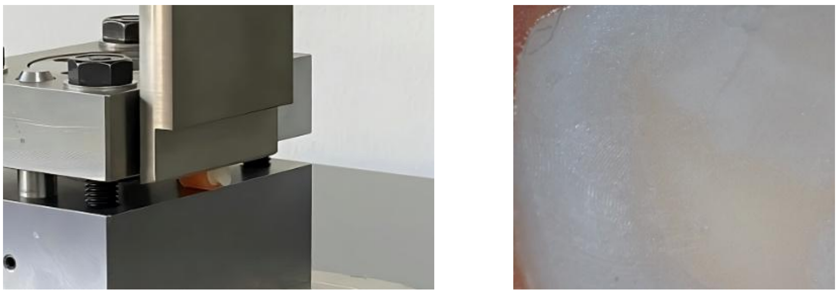


Figure 2. Tooth that has undergone acrylic resin fixation



(A) (B)

Figure 3. (A) Shear bond strength test using Universal Testing Machine. (B) Observation results of composite resin bond failure

has a truncated-conical hole, with a base diameter of 3 mm and a peak diameter of 6 mm. The mold can be split into two parts, allowing it to be easily opened once the composite resin has set.

Fabrication of the tooth fixation mold (Mold B), used for acrylic resin impressions in tooth fixation, is made from metal with dimensions of 50 mm in length, 50 mm in width, and 10 mm in height. The center of the mold has a cylindrical hole with a 10 mm diameter. This mold can be split into two parts, allowing it to be easily opened once the acrylic resin has hardened.

Forty-eight premolar teeth, post-orthodontic extraction, meeting the inclusion criteria, were disinfected by immersion in 0.5% chloramine-T solution for 24 hours. Calculus and debris on the teeth were cleaned, after which the teeth were soaked with distilled water and stored in a refrigerator at 4 °C until use, in accordance with ISO 29022 standards.

Measurements were made at a point 2 mm apical from the Cementoenamel Junction (CEJ) and 4 mm coronal from the CEJ using a caliper, marked with a marker, and then cut using a double-faced diamond disc bur. The teeth were slowly embedded and placed into acrylic resin with the occlusal surface facing up, aligned with the surface of the acrylic resin, prior to the resin setting. The teeth embedded in the acrylic resin were removed from Mold B once the resin had hardened. This procedure was repeated until all the teeth were fixed.

All 48 specimens were randomly divided into two groups, after which they were subjected to bleaching using 0.01 ml of 35% H_2O_2 (Opalescene Endo, Ultradent Products, South Jordan, USA). Each sample was then placed in a sterile plastic container and incubated at 37 °C for 120 hours. The samples were washed with 10 ml of distilled water and dried for 10 seconds using an air spray. The bleaching process was repeated twice for Group I and three times for Group II.

Group I (two bleaching sessions) consisted of 24 specimens divided into 3 subgroups: A, B, and C. Group A (control) specimens were immersed in artificial saliva and stored in the incubator at 37 °C for seven days before the restorative procedure. Group B was treated with 0.01 ml of 35% sodium ascorbate combined with 0.4% surfactant for five minutes, washed with 15 ml of distilled water, dried for five seconds, and then applied with the same sodium ascorbate-surfactant combination (two applications) before the restorative procedure. Group C received the same treatment as Group B, but the procedure was repeated three times before the restorative procedure.

For Group II (three bleaching sessions), the same treatment as Group I was applied to subgroups IIA, IIB, and IIC, followed by the restorative procedure. The restorative procedure for each sample was performed using bulk-fill composite resin restoration (Beautifill, Bulk Restorative, Shofu Inc, Japan). Bonding material (Single Bond Universal, 3M, USA) was applied to each dentin surface of the specimens and polymerized for ten seconds. A mold was placed on the samples and filled with composite resin, then polymerized for 20 seconds. All specimens were then immersed in artificial saliva and stored in the incubator at 37 °C for 24 hours.

Shear bond strength testing was performed on each sample using a Universal Testing Machine (Tensilon RTI-1225), and bonding failure was observed using a stereomicroscope with 20x magnification (Olympus DF Plapo 1X, Japan) at the Integrated Research Laboratory, Faculty of Dentistry, Universitas Gadjah Mada. Data were analyzed using two-way ANOVA and LSD tests.

RESULTS

The mean shear bond strength values of composite resin are presented in Table 1. The data were subsequently subjected to a normality test using the Shapiro-Wilk test ($\alpha = 0.05$) and a homogeneity of variance test using Levene's test ($\alpha = 0.05$). The results of these tests are summarized in Table 2. Both normality and homogenity test results indicated p-values greater than 0.05, indicating that the data were normally distributed and homogeneous.

Based on these findings, the data met the requirements for parametric statistical analysis using two-way analysis of variance (ANOVA). The results of the two-way ANOVA test are presented in Table 3. The data were then tested using the Post Hoc Least Significant Difference (LSD) test to determine the pairs of groups with differences in the shear bond strength of composite resin. The results of this test can be seen in Table 4.

Table 1. The mean values and standard deviations of the shear bond strength of composite resin after intracoronal bleaching with 35% H2O2, followed by the application of 35% sodium ascorbate combined with 0.4% surfactant (in MPa).

Bleaching application	n	Application of sodium ascorbate combined with surfactant		
		7-day immersion (A)	Application of 2x SA (B)	Application of 3x SA (C)
		x̄ ± SD	x̄ ± SD	x̄ ± SD
2x Bleaching (I)	8	10.57 ± 0.17	11.37 ± 0.20	11.27 ± 0.33
3x Bleaching (II)	8	10.29 ± 0.22	10.45 ±0.13	10.54 ± 0.15

Table 2. The results of the normality and homogeneity tests for the shear bond strength of composite resin after intracoronal bleaching with 35% H2O2, followed by the application of 35% sodium ascorbate combined with 0.4% surfactant

Group		p value	
		Shapiro-Wilk	Levene's test
IA	2x bleaching and 7-day immersion	0.890	
IB	2x bleaching and 2x SA 35% + surfactan 0.4%	0.453	
IC	2x bleaching and 3x SA 35% + surfactan 0.4%	0.619	0.264
IIA	3x bleaching and 7-day immersion	0.796	
IIB	3x bleaching and 2x SA 35% + surfactan 0.4%	0.205	
IIC	3x bleaching and 3x SA 35% + surfactan 0.4%	0.754	

Table 3. The results of the two-way ANOVA for the shear bond strength of composite resin after intracoronal bleaching with 35% H2O2, followed by the application of 35% sodium ascorbate combined with 0.4% surfactant

Treatment	Degrees of freedom	F	p
Frequency of sodium askorbat	2	27.060	0.001*
Frequency of bleaching	1	112.091	0.001*
Frequency of sodium askorbat *frequency of bleaching	2	9.465	0.001*

The two-way ANOVA results showed that the frequency of sodium ascorbate application had a significant effect on the shear bond strength of composite resin after bleaching with 35% hydrogen peroxide ($p = 0.001$; $p < 0.05$). Similarly, the frequency of intracoronal bleaching also demonstrated a significant effect on shear bond strength ($p = 0.001$ ($p < 0.05$). In addition, a significant interaction was observed between sodium ascorbate application frequency and bleaching frequency, indicating that the combined effects of these two variables influenced the shear bond strength of composite resin following bleaching.

Post Hoc LSD test indicate that there was no significant difference in the frequency of sodium ascorbate application combined with surfactant between groups IB and IC ($p = 0.316$) or between groups IIB and IIC ($p = 0.439$). Table 4 also shows that there was no significant difference between the group subjected to three bleaching sessions followed by a 7-day immersion (IIA) compared to

Table 4. The results of the Post Hoc LSD test for the shear bond strength of composite resin after intracoronal bleaching with 35% H2O2, followed by the application of 35% sodium ascorbate combined with 0.4% surfactant

Group pairings treatment	Mean Difference	p
IA-IB	-0.798	0.001*
IA-IC	-0.691	0.001*
IA-IIA	0.286	0.010*
IA-IIB	0.121	0.257
IA-IIC	0.038	0.714
IB-IC	0.107	0.316
IB-IIA	1.085	0.001*
IB-IIB	0.92	0.001*
IB-IIC	0.837	0.001*
IC-IIA	0.978	0.001*
IC-IIB	0.812	0.001*
IC-IIC	0.73	0.001*
IIA-IIB	-0.165	0.125
IIA-IIC	-0.247	0.024*
IIB-IIC	-0.082	0.439

Table 5. Classification of bonding failure for each research group

	Bonding failure	Number	Persentage
Group IA	Adhesive failure	2	25%
	Cohesive failure	0	0%
	Mixed failure	6	75%
Group IB	Adhesive failure	1	12.5%
	Cohesive failure	0	0%
	Mixed failure	7	87.5%
Group IC	Adhesive failure	2	25%
	Cohesive failure	0	0%
	Mixed failure	6	75%
Group IIA	Adhesive failure	3	37.5%
	Cohesive failure	0	0%
	Mixed failure	5	62.5%
Group IIB	Adhesive failure	2	25%
	Cohesive failure	0	0%
	Mixed failure	6	75%
Group IIC	Adhesive failure	1	12.5%
	Cohesive failure	0	0%
	Mixed failure	7	87.5%

the group subjected to three bleaching sessions followed by two applications of sodium ascorbate combined with surfactant 0.4% (IIB) ($p = 0.125$). However, a significant difference was found when the three-bleaching group with a 7-day delay (IIA) was compared with the group receiving three applications of sodium ascorbate combined with 0.4% surfactant (IIC) ($p = 0.024$). The observation of the bonding failure areas in the research specimens is presented in Table 5.

Analysis of bonding failure areas in the research specimens was conducted to determine whether the bonding failure was adhesive, cohesive, or mixed failure. Across all six experimental groups, mixed failure was the most frequently observed mode, accounting for 77.08% of all specimens.

DISCUSSION

The mean shear bond strength of resin composite in the group with three bleaching applications was lower compared to the group with two bleaching applications. The lowest value was found in the group with three bleaching applications without sodium ascorbate, with a shear strength of 10.287 ± 0.219 MPa, while the highest value was obtained in the group with two bleaching applications and two applications of 35% sodium ascorbate combined with 0.4% surfactant, at 11.372 ± 0.204 MPa. These results indicate that higher bleaching application frequencies lead to a reduction in the shear bond strength of restorations. This finding aligns with the findings of Karadas et al. (2019), who stated that increased bleaching frequency and duration cause chemical and morphological changes to the dentin surface, thereby reducing bond strength.[19]

The two-way ANOVA results also showed a significant effect of the application frequency of 35% sodium ascorbate combined with 0.4% surfactant on the shear bond strength of resin composites. This result is consistent with studies by Coppla et al (2019) and Nugraheni et al (2018), which found that repeated applications of sodium

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ascorbate enhance the effectiveness of free radical removal post-bleaching. Higher frequencies of sodium ascorbate application increase the availability of antioxidants, thereby improving the ability to eliminate free radicals.[10,13]

The frequency of 35% sodium ascorbate applications significantly influenced the shear bond strength of resin composites. Applying sodium ascorbate more than once within a short period effectively eliminates free radicals within the dentinal tubules, restoring the bonding strength of resin composites. Post-bleaching free radicals interfere with resin composite polymerization, leading to the formation of low-quality polymers. Sodium ascorbate works by donating electrons to neutralize free radicals, which are then oxidized into dehydroascorbate.[12,20]

The two-way ANOVA results also indicated that the frequency of bleaching agent application significantly affects the shear strength of resin composites. Bleaching agents alter the organic structure of dentin, disrupting the diffusion of adhesive materials into the interfibrillar spaces of collagen fibers.[20] Collagen denaturation post-bleaching interferes with the formation of the hybrid layer, which is essential for the long-term durability of the bond between resin composites and teeth.[21] The decrease in pH during bleaching also reduces microhardness and increases tooth porosity, decreasing the length and number of resin tags.[21,22]

Post Hoc test results showed no significant difference in shear bond strength between groups receiving two (IB; IIB) and three (IC; IIC) applications of 35% sodium ascorbate combined with 0.4% surfactant. Nugraheni et al. (2018) stated that more than one application of sodium ascorbate is required to eliminate residual free radicals after two bleaching applications. These results suggest that to fully remove residual free radicals after two or three applications of 35% hydrogen peroxide, at least two applications of 35% sodium ascorbate combined with 0.4% surfactant are necessary.

In the present study, the contact time of 35% sodium ascorbate combined with 0.4% surfactant on post-bleaching teeth was 5 minutes. This duration was selected based on findings by Nascimento (2019), which demonstrated that the reaction between hydrogen peroxide and sodium ascorbate occurs rapidly. A five-minute contact time with the antioxidant is sufficient to eliminate free radicals post-bleaching, allowing restorative procedures to be performed earlier.[23]

Post hoc test results also revealed significant differences between groups subjected to two and three bleaching applications. This finding is consistent with the study by Ghaleb et al. (2020), which reported that increasing the number of bleaching applications leads to greater accumulation of free radicals. Consequently, resin composite shear bond strength was lower following three bleaching applications compared with two.[24]

Post Hoc test results also revealed significant differences between groups subjected to two and three bleaching applications. This finding aligns with research by Ghaleb et al (2020), which stated that the greater the number of bleaching applications, the more free radicals are generated. These results indicate that the shear bond strength of resin composites is lower with three bleaching applications compared to two.[24]

The recommended clinical procedure is to allow a waiting period of 1-3 weeks post-bleaching before restoration, since optimal bonding strength and color stability are typically achieved after 2-3 weeks. In this study, the shear bond strength of resin composites after application of 35% sodium ascorbate combined with 0.4% surfactant was higher than that of the control group, indicating that a seven-day waiting period alone is insufficient to completely eliminate free radicals.[1,22]

The highest shear bond strength of the resin composite was observed in group IB, which underwent two bleaching applications followed by two applications of 35% sodium ascorbate combined with 0.4% surfactant, yielding a value of 11.372 ± 0.204 MPa. This value falls within the recommended dentin bond strength range of 10–50 MPa.[25] Although Single Bond Universal Adhesive exhibits an optimal bond strength of approximately 30 MPa, the maximum values achieved in this study were lower than the manufacturer's stated performance.

The frequency of 35% sodium ascorbate application has also been reported to influence calcium loss and modify the surface structure of dentin previously exposed to 35% hydrogen peroxide, which may contribute to variations in adhesive outcomes.[26] Several methodological variables, including the adhesive system used, specimen preparation procedures, storage environment, and loading conditions, may also have affected the bond strength results.[25] Furthermore, the operator-dependent technique used during adhesive application is an additional factor that influences the overall bonding performance.[27]

However, the effectiveness of residue removal may vary depending on the rinsing volume, duration, and technique used.The technique for cleaning residues and applying sodium ascorbate is crucial for optimizing the antioxidant's ability to eliminate free radicals. The method of cleaning sodium ascorbate residues from the dentin surface can influence the shear bond strength test results of resin composites.[16] In this study, sodium ascorbate residues were cleaned using 15 ml of distilled water for 30 seconds, followed by air-drying. This procedure has been reported to create a suitable dentin surface for bonding procedures.[24] However, the effectiveness of residue removal may vary depending on the rinsing volume, duration, and technique used.

The sodium ascorbate application technique in this study was performed passively by dripping 35% sodium ascorbate combined with 0.4% surfactant without agitation. Passive application may be less effective than agitation, which could enhance the penetration of the antioxidant.[9,16] As a result, most specimens experienced mixed failure, a combination of adhesive and cohesive failure. Adhesive failure occurs at the substrate or adhesive interface, while cohesive failure occurs within the substrate or material. Stress distribution at the bonding interface is influenced by the location and geometry of the testing device, which determines the bond strength.[21]

Limitations of this study lies in the method of sodium ascorbate (SA) application and residue removal. The cleaning of sodium ascorbate residues was performed using 15 mL of distilled water for 30 seconds, followed by air-drying. While this approach has been reported to provide a clinically acceptable dentin surface for bonding procedures,[24] alternative methods such as longer immersion in distilled water, reduced rinsing volume, or pumice prophylaxis have also been suggested to improve residue removal.[16] Moreover, the application of sodium ascorbate in this study was carried out passively without any agitation. Previous studies indicated that active application through agitation may enhance sodium ascorbate penetration and improve the neutralization of residual free radicals on the dentin surface.[21] Passive application, as used in this study, has been associated with less effective outcomes in restoring resin bond strength following bleaching procedures.[9] Therefore, the absence of active agitation during sodium ascorbate application and the standardized rinsing protocol may limit the generalizability of the results, particularly in clinical scenarios requiring optimal antioxidant activity.

CONCLUSION

Within the limitations of this study, it can be concluded that shear bond strength of composite resin applied with three applications of 35% sodium ascorbate combined with 0.4% surfactant is the same as the shear bond strength applied with two applications of 35% sodium ascorbate combined with 0.4% surfactant. Additionally, teeth bleached three times exhibited lower shear bond strength compared to teeth bleached two times.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this paper.

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