

Comparative Analysis of Shear Bond Strength of Composites to the Sodium Ascorbate Hydrogel-treated Bleached Enamel Surfaces: An *In Vitro* Analysis

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ABSTRACT

Aim: To compare the shear bond strength of composites to the sodium ascorbate hydrogel-treated bleached enamel surfaces.

Materials and methods: Sixty-six extracted human maxillary first premolars were sectioned mesiodistally to obtain two nonocclusal surfaces ($n = 132$). The specimen in the experimental group ($n = 80$) were divided into four treatment groups depending upon bleaching protocols used: group I (10% FGM home bleach), group II (22% Pola Night home bleach), group III (37% light-activated in-office bleach Pola Office+), and group IV (35% Pola office in-office bleach without light activation). Specimens were subjected to the 1-minute application twice with sodium ascorbate hydrogel and were further divided into two subgroups "a" (acetone-based) and "b" (ethanol-based) depending upon the bonding agents used. The specimens of the control group ($n = 52$) were further divided into two treatment groups, that is, positive control and negative control. The specimens were tested using universal testing machine.

Results: The result of the present study revealed that the specimens bleached with 10% FGM home bleach showed the greatest shear bond strength and specimens bleached with 35% in-office bleach Pola Office showed the lowest shear bond strength.

Conclusion: A twice 1-minute application of sodium ascorbate hydrogel was effective to reverse the deleterious results of bleaching on enamel shear bond strength. Bonding agent containing acetone as a solvent (Prime and Bond NT) in this present study showed greater shear bond strength values than the bonding agent containing ethanol as a solvent (Adper Single Bond).

Statement of clinical relevance: The shear bond strength of composite restorative materials could be improved by applying sodium ascorbate gel to the bleached enamel surfaces.

Keywords: Bleaching agents, Bonding agents, Composite restorations, Sodium ascorbate hydrogel.

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INTRODUCTION

Bleaching of discolored teeth has been one of the most conservative and economical methods of improving the appearance of an individual. After bleaching, the tooth significantly becomes lighter shade when compared to the previous shade. Though, the tooth shade becomes lighter, preexisting restoration if any on the bleached tooth remains the same. The difference in the shade of preexisting restoration with bleached tooth warrants a replacement of preexisting restoration with a new restoration.¹

Bond strength to the restorative materials could be reduced after bleaching of the enamel, and it can be due to the interference of residual peroxides with resin tag formation.² Restorative procedures should be delayed to reverse the aforementioned effect, and it was also postulated that antioxidants can be used to reverse effects of bleaching agents.

Free radicals remaining in the tooth structure after bleaching react with antioxidants and thus indirectly aids in increasing the bond strength of tooth structure and restorative materials.³ Sodium ascorbate was proposed in the literature to aid in improving the bond strength of restorative materials to the bleached enamel surfaces.³

The present study aims to compare the shear bond strength of bleached enamel using different bleaching protocols with two different bonding agents after application of sodium ascorbate hydrogel. The null hypothesis states that "there would be no effect

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on shear bond strength of bleached enamel surface" after the application of antioxidant sodium ascorbate hydrogel.

MATERIALS AND METHODS

Sixty-six human maxillary first premolars extracted for periodontal reasons were collected. Institutional ethical clearance was obtained for the present study with an ethical approval number-DDCH/ADM/2016-17/1391-CD&E. Carious teeth, teeth with

cracks/ fractures, carious lesions, and developmental defects were not included. The teeth were washed under running tap water to remove blood and saliva and were rendered free of calculus and other soft tissue debris using an ultrasonic scaler. The samples were further stored in distilled water at room temperature.

Preparation of Samples

Samples were prepared by removing the roots of the teeth utilizing a high-speed diamond disk attached to the dental micromotor headpiece. Each test sample’s crown was sectioned mesiodistally in order to attain two nonocclusal enamel surfaces (Fig. 1) and thus 132 samples were obtained.

A silicone mold was prepared and test samples were mounted in acrylic resin to expose the enamel surface (Fig. 2). Test samples were further polished using 320 grit silicone carbide papers to get standard flat enamel surface.

Sample Distribution

Experimental Group

The test samples were randomly divided into four treatment groups with 20 samples in each treatment group, that is, groups I, II, III, and IV depending upon bleaching protocols used. The test groups were further divided into subgroups “a” [Fifth generation acetone-based

(Prime and Bond NT, DENTSPLY Caulk)] and ‘b’ [Fifth - generation ethanol-based (Adper Single Bond, 3M ESPE)] depending upon bonding agents used.

Control Group

The positive control group was in which the samples were not bleached and bonded with acetone-based and ethanol-based bonding agents. The negative control group was in which the samples were bleached using four different bleaching agents and any of the two bonding (acetone or ethanol) systems were used for bonding.

Bleaching Procedure

Test samples in groups I and II were treated with 10 and 22% carbamide peroxide home bleaching agents [FGM (whiteness perfect) Joinville, SC Brazil Home Bleach and Pola Night (SDI), respectively] and test samples of groups III and IV were bleached with [light-activated Pola Office + (SDI) and nonlight activated In-office bleach Pola Office (SDI), respectively] as per manufacturer’s instructions (Fig. 3).

Preparation of Sodium Ascorbate Hydrogel (Antioxidant Gel)

Sodium ascorbate solution (32%) was prepared by dissolving sodium ascorbate in pure water at normal room temperature. Carbopol gel [2.5% (wt/wt)] was prepared by mixing carbopol resin with sodium ascorbate solution. The mixture was thickened by stirring and was then neutralized adding triethanolamine (TEA), and the pH was adjusted to 7.⁴

Application of Sodium Ascorbate Hydrogel

After bleaching the test samples 32% sodium ascorbate hydrogel was applied twice for 1 minute each on enamel surfaces of the embedded specimen in the experimental group. Test samples were immersed for 10 minutes in distilled water to dissolve sodium ascorbate on enamel surface.

Bonding Procedure and Composite Placement

Test samples were etched using phosphoric acid (37%) for 15 to 30 seconds. After etching of the test samples, fifth-generation “acetone-based” (Prime and Bond NT, DENTSPLY Caulk) and “ethanol-based” (Adper Single Bond, 3M ESPE) bonding agents were applied to enamel surfaces of all test samples (Fig. 4). A plastic

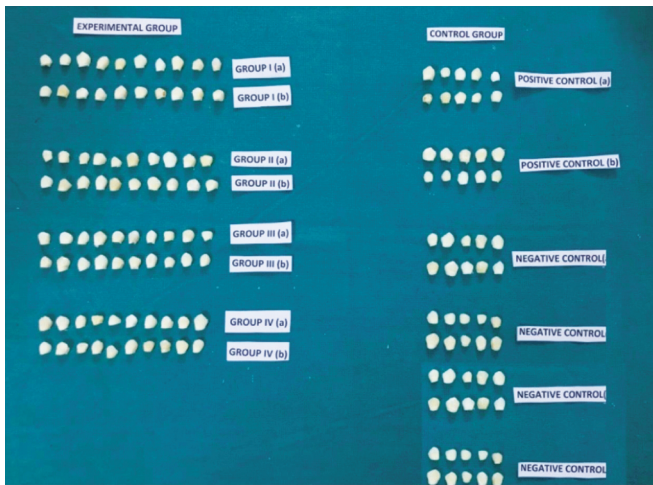


Fig. 1: Test samples for the experimental and control groups



Fig. 2: Mounting of the test sample in the acrylic block

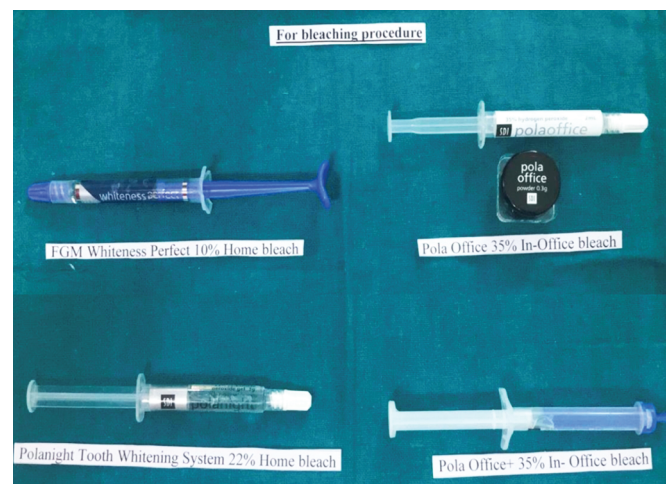


Fig. 3: Materials used for the bleaching

tube having an internal diameter of 2 mm and height of 2 mm was prepared to follow the ISO/TS 11405 specification that is, limitation of bonding area is vital for testing procedures.⁵ Plastic tube was placed on the surface of specimens before curing the adhesive and further light cured as per manufacturer's instructions. The prepared plastic tube was filled with composite material and light cured. The plastic tube was removed after the curing of the composite resin.



Fig. 4: Materials used for the composite build-up on the test samples

Preparation of the Control Group Sample

In the positive control group V ($n = 20$) specimens were not bleached and were directly bonded with acetone-based ($n = 10$) and ethanol-based ($n = 10$) bonding agents and composite build-up was done. In negative control group, test samples of group VI and VII ($n = 8$ in each group) were subjected to 10 and 22% carbamide peroxide home bleaching agents, respectively. Samples in group VIII and group IX ($n = 8$ in each group) were subjected to 37% hydrogen peroxide light-activated and 35% hydrogen peroxide nonlight-activated in-office bleaching agents, respectively using a similar technique in the experimental group. The samples were further divided based upon the bonding agents used ($n = 4$) and composite build-up done using a similar technique in the experimental group. The division of experimental and control group specimens according to the materials used are presented in Figure 5.

Shear Bond Strength Testing

Test samples were mounted and shear stress was applied at the rate of 1.5 mm/min using Universal testing machine (ASI, Delhi) with a knife edged loading head.

Statistical Analysis

The descriptive statistical analysis was done in all the experimental and control groups and means \pm standard deviation (SD) values

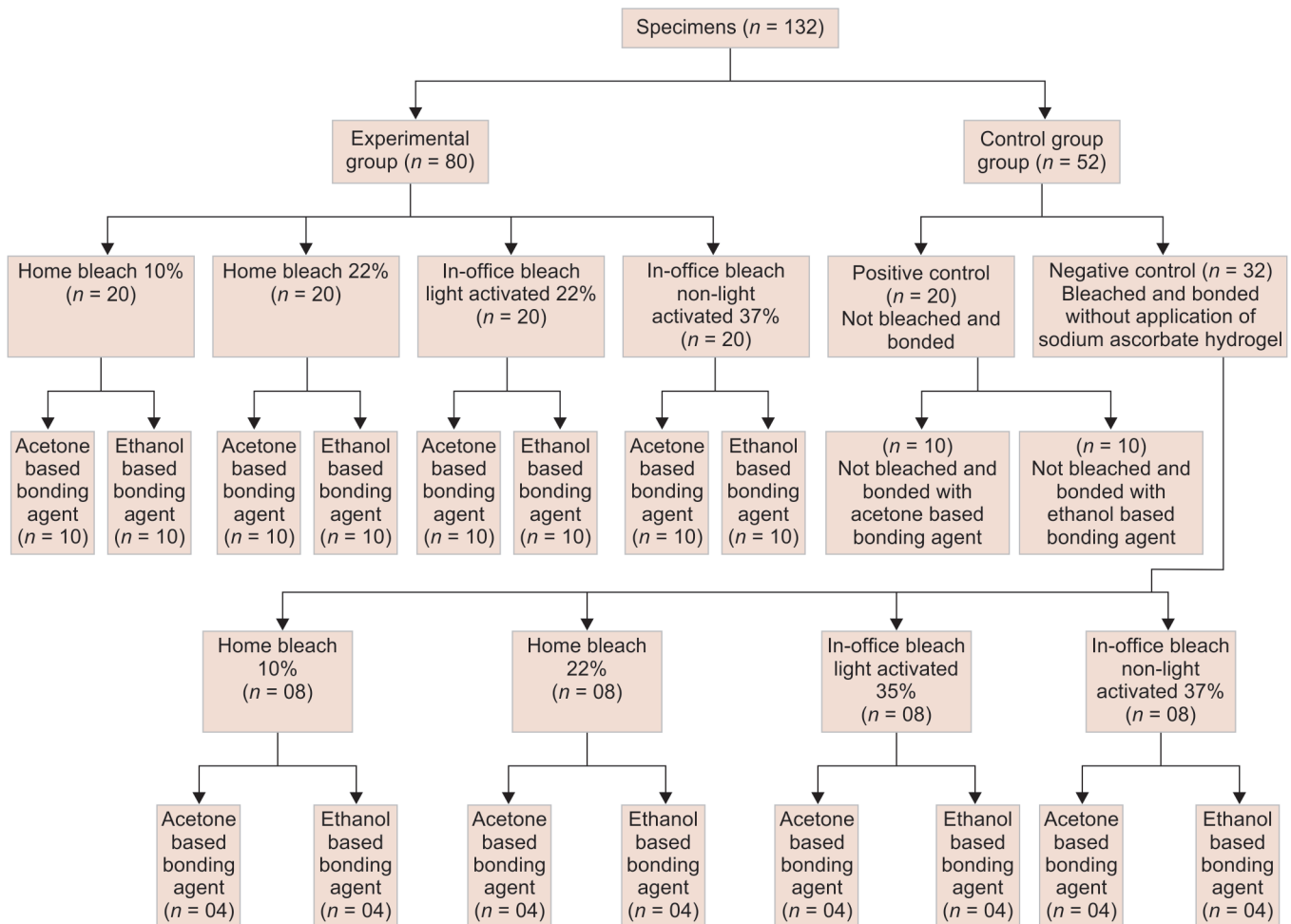


Fig. 5: Division of experimental and control groups according to the materials used

were derived. The data for each group were analyzed using Oneway analysis of variance (ANOVA) and Student *t*-test. Statistical analysis was done using "SPSS software version 21" and a *p*-value of less than or equal to 0.05 was considered as statistically significant.

RESULTS

Shear bond strength values of all the test samples were analyzed and mean ± SD values are presented in Table 1. The mean and SD of the various experimental and negative control groups are presented in Table 2. As per the result of this study, it was observed that the specimens which received sodium ascorbate hydrogel application after bleaching and later were bonded using any of the bonding systems presented higher shear bond strength (*p* < 0.001) when compared to those groups that did not receive any sodium ascorbate hydrogel treatment. Table 2 shows test samples bonded with Prime and Bond NT. Fifth-generation acetone-based bonding agent shows more shear bond strength values than the test samples bonded with Adper Single Bond, that is, ethanol-based bonding agent (*p* < 0.001) irrespective of different bleaching protocols used.

The comparison between bond strengths achieved after using acetone-based and ethanol-based bonding agents in positive control group samples is presented in Table 3. The samples prepared using acetone-based bonding agents showed higher shear strength values than the ethanol-based bonding agents (*p* < 0.001).

These intergroup comparisons of various shear bond strengths achieved with different concentrations of bleaching agents are represented in Table 4. Highest shear bond strength was observed when test samples were bleached with 10% carbamide peroxide home bleach irrespective of the bonding agent used. Similarly, the lowest shear bond strength is observed in specimens subjected to 35% hydrogen peroxide nonlight activated in-office bleach irrespective of different bonding agents used.

DISCUSSION

Bleaching agents usually contain different concentrations of carbamide peroxide/hydrogen peroxide and can cause alteration in the tooth substrate.

Oxygen-free radicals were released by the bleaching agents on to the enamel surfaces and they inhibit polymerization of the composite materials,⁴ which results in a granular and porous bubbled appearance of resin-bleached enamel interface.⁶ Thus immediately after bleaching a lower hardness values were observed on the enamel surfaces.⁴ Peroxide apatite crystals were produced in the enamel as the peroxide ions replace hydroxyl ions in apatite crystals. The replacement process can be reversed by the application of antioxidants on enamel surfaces.⁷ Buffering action of saliva requires at least 24 hours to 3 weeks to remove free radicles present on enamel surface and thus the restorative procedure need to be suspended during the recommended time.⁸ Sodium ascorbate has been traditionally used as an antioxidant considering its ability to neutralize and/or reverse the oxidizing effect of peroxide containing materials.⁹ Neutralization from antioxidant agents to free radicals is categorized into three types firstly continuous full-time prevention, second active detoxification of oxidative stress, and third passive detoxification.¹⁰ Sodium ascorbate is included in passive detoxification that can neutralize free radicals and belong to nonenzyme antioxidants.¹¹ Results of this study are in accordance with the published reports where sodium ascorbate aids in reversing the effects of bleaching agents on bonding of restorative materials.^{3,9,12-14}

Sodium ascorbate gel was used instead of solution in the present study as the gel forms have more acceptability in clinical scenarios as manipulation of solution is more difficult than gels.¹⁵ The mode of delivery both in terms of concentration and duration of application are important factors in reversing the effects

Table 1: Shear bond strength values of the experimental and control (positive and negative) group samples

S. No	Experimental group								Positive control group		Negative control group							
	Group I		Group II		Group III		Group IV		Group V	Group VI	Group VII		Group VIII		Group IX			
Subgroup (sample)	a (10)	b (10)	a (10)	b (10)	a (10)	b (10)	a (10)	b (10)	a (10)	b (10)	a (4)	b (4)	a (4)	b (4)	a (4)	b (4)	a (4)	b (4)
Mean	53.29	41.2	39.6	21.6	47.0	37.8	23.5	18.3	29.6	15.2	17	13.9	19	16.6	6	4.1	4.4	2.1
SD	4.60	1.26	1.74	1.50	2.59	2.09	2.23	3.13	2.09	1.24	1.61	2.58	1.34	1.62	1.0	1.22	0.87	1.09

Shear bond strength values were presented in the units of MPa in all the test samples

Table 2: Comparative analysis of shear bond strength between the experimental and negative control groups

S. No	Group	Mean ± SD	<i>p</i> -value
1	Experimental group using acetone-based bonding agents (G Ia + G IIa + G IIIa + G IVa)	40.8 ± 11.63	< 0.001*
2	Negative control group using acetone-based bonding agents (G VIa + G VIIa + G VIIIa + G IXa)	11.6 ± 6.77	
3	Experimental group using ethanol-based bonding agents (G Ib + G IIb + G IIIb + G IVb)	29.7 ± 10.24	< 0.001*
4	Negative control group using ethanol-based bonding agents (G VIb + G VIIb + G VIIIb + G IXb)	9.2 ± 6.56	

**p* value of < 0.05 was considered as statistically significant



Table 3: Comparative analysis of shear bond strength values in positive control groups using acetone-based and ethanol-based bonding agents

S. No	Group	Mean \pm SD	p-value
1	Positive control group using acetone-based bonding agent (G Va)	29.6 \pm 2.09	<0.001
2	Positive control group using ethanol-based bonding agent (G Vb)	15.2 \pm 1.23	

*p value of <0.05 was considered as statistically significant

Table 4: Comparative evaluation of shear bond strength of experimental samples using acetone-based and ethanol-based bonding agents

S. No	Type of bonding agent	Group	Mean \pm SD	p-value
1	Acetone-based bonding agent	G Ia	53.2 \pm 4.6	<0.001
		G IIa	39.6 \pm 1.74	
		G IIIa	47 \pm 2.59	
		G 1Va	23.5 \pm 2.23	
2	Ethanol-based bonding agent	G Ib	41.2 \pm 1.25	<0.001
		G IIb	21.6 \pm 1.5	
		G IIIb	37.8 \pm 2.09	
		G 1Vb	18.3 \pm 3.12	

*p value of <0.05 was considered as statistically significant

of bleaching agents. The regularly used concentrations of sodium ascorbate range from 2.5 to 35%.

Different concentrations (2.5, 5, and 10%) of sodium ascorbate were tested and agents with concentration less than 10% are not sufficient to reverse the effects of bleaching agents.⁷ It was also reported that the amount of sodium ascorbate required to neutralize the effects of bleaching agents is proportional to the concentration of bleaching agents used.¹⁶⁻¹⁸

In the present study, the bleached enamel was exposed to sodium ascorbate as per recommendations in the past literature (two applications of 1 minute each).¹⁹ The methodology used in the present study was different from past literature, where longer duration of applications (10 minutes to 6 hours) were tested.²⁰ It was reported that the frequency of applications is more vital than the contact time of sodium ascorbate.¹⁹ The reaction of oxide-free radicals and antioxidants reach the maximum in 1 minute and thus increasing the duration of contact time of sodium ascorbate cannot remove more free radicals.¹⁹

A commonly used in-office bleaching system with or without the light activation and home bleaching systems with different concentrations were used, and it was found that specimen bleached with 10% FGM home bleach, that is, group I showed greater shear bond strength as compared to other experimental and control groups. The possible explanation could be that the application of sodium ascorbate immediately after use of carbamide peroxide increased the length of resin tag formation and the present study results are in accordance with the past literature.^{2,15,21-25} Sodium ascorbate is, however, less effective in teeth bleached with greater concentrations of carbamide peroxide with higher concentrations of oxygen being released.^{2,26}

It was reported that use of 35% hydrogen peroxide is more deleterious for resin tag formation than 10% carbamide peroxide. Ten percent carbamide peroxide releases 3% hydrogen peroxide and 7% urea; this is 10 times lower than 35% concentration of carbamide peroxide.^{2,11} Therefore, the results of our present study were in concurrence with the available scientific data, which states lower bond strength values when the greater concentration of hydrogen peroxide is used and antioxidant agents were not able to neutralize the effects of bleaching agents on bond strength.⁶ Bleached test samples in

the negative control group, which were bleached and immediately restored using composite resin without any antioxidant treatment reported the least bond strength values and the presumable reason was that the residual oxygen released by bleaching agents on the enamel surface inhibit the polymerization of bonding agents.¹⁰

Specimens of positive control group, that is, specimens that were not bleached and were directly bonded with acetone and ethanol-based bonding agent also showed the statistically significant difference in their bond strength value, with higher bond strength in samples bonded with acetone-based bonding agent when compared to specimens bonded with the ethanol-based bonding agent. The reason behind the above results may be because acetone plays an important role to displace the water from tooth surface and results in better bond strength which was in accordance with the past literature.²⁷

Light sources have greater potential for whitening teeth because they produce necessary heat for activating the hydrogen peroxide.²⁷ The use of the light source for activation of bleaching agents is optional, and the effect of light sources is at times controversial. Some believe that it is very effective in bleaching the tooth structure, while others report only certain lights are effective.²⁸ Various factors determine the controversial results such as baseline color of the teeth, concentration of bleaching agents used, and duration of the treatment.²⁹⁻³⁶

In the present study, specimens of group III bleached with 37% hydrogen peroxide in-office bleaching system Pola office+ with light activation showed greater bond strength value in comparison to specimens of group IV bleached with 35% hydrogen peroxide in-office bleaching system Pola office without light activation. The possible explanation could be due to better polymerization and release of less oxygen-free radicals from the bleaching agents after exposure to light source resulting in greater bond strength values. This may be justified by the fact that the light-activation system produces good instant bleaching effects than the nonlight-activation systems with less concentrated hydrogen peroxide. Considering the above findings and results, the hypothesis of the present study was rejected where the application of sodium ascorbate antioxidant hydrogel significantly increased the shear bond strength of bleached enamel surfaces.

LIMITATIONS

Though every effort was taken to mimic the oral conditions, thermocycling of the test samples was not done in the present study. Secondly, the influence of dynamic or cyclic load testing was not in the present study which may limit the recommendation present study results to the clinical practice. Thus, the results of this *in vitro* study need to be cautiously implemented in the clinical scenario, and *in vivo* and randomized controlled trials are indicated to reinforce the present study results.

CONCLUSION

A twice 1-minute application of sodium ascorbate hydrogel was effective in neutralizing the deleterious effects of bleaching agents on enamel shear bond strength. Bonding agent containing acetone as a solvent (Prime and Bond NT) showed greater shear bond strength values than the bonding agent containing ethanol as a solvent (Adper Single Bond). As the concentration of bleaching agents increases from 10 to 37%, the shear bond strength decreases except for the specimens bleached with a 35% light-activated in-office bleaching system.

CLINICAL SIGNIFICANCE

The use of sodium ascorbate gel on the bleached enamel surfaces with acetone-based bonding agents improved the shear bond strength of the restorative composite materials.

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