



Article Effect of a Desensitizing Agent on Shear Bond Strength of Ceramic Bracket on Previously Bleached Teeth

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Abstract: This study aims to analyze the effect of desensitizing agents on the shear bond strength (SBS) of ceramic brackets after in-office bleaching procedures. Twenty-seven extracted premolars were equally divided into three groups (n = 9). Group 1 served as the control; bleaching was performed with 37% hydrogen peroxide. In group 2, bleaching and the application of a fluoride-containing desensitizing agent (CPP-ACFP) were performed. In group 3, bleaching and the application of a nonfluoride-containing desensitizing agent (CPP-ACP) were performed. Ceramic brackets were bonded with composite resin. SBS was tested using a universal testing machine. The site of bonding failure was measured using the adhesive remnant index (ARI) score. There was a statistically significant difference in mean SBS values (p < 0.05). Group 1 showed the lowest SBS value (6.32 ± 4.83 MPa), which differed significantly with groups 2 and 3 (p < 0.05). There was no significant difference between group 2 (15.36 \pm 4.67 MPa) and group 3 (12.19 \pm 6.81 MPa) (p > 0.05). The ARI score did not show a significant difference. The conclusion is that the application of fluoride-containing and non-fluoride desensitizing agents increases the shear bond strength of ceramic brackets on bleached teeth with composite resin cementation. The application of both desensitizing agents had no impact on the results of the ARI score, with the highest ARI score in each group being 4, indicating that less than 10% of the adhesive substance remains on the enamel.

Keywords: shear bond strength; desensitizing agent; in-office bleaching; ceramic; bracket

1. Introduction

Bleaching is a common procedure used to enhance the appearance of a smile in society, along with the growing interest in aesthetic and cosmetic dentistry. Orthodontics is another therapy that is gaining favor in the aesthetic sector since it is utilized to promote dentofacial harmony, improve occlusion, and alleviate malocclusion [1]. Orthodontic tooth movements may be accomplished with many different types of orthodontic appliances, such as labial or buccal brackets, lingual brackets, and clear aligners [2,3]. Nowadays, orthodontists frequently face patients who are dissatisfied not only with the alignment, but also with the color of their teeth due to the rising demand for adult orthodontics [4]. When it comes to treating discolored teeth, bleaching is thought of as a more conservative option because it does not require any invasive procedures like extensive tooth tissue-removing prosthodontic preparation procedures [5].

Bleaching itself is defined as the chemical degradation of chromogens and occurs through a complex chemical reaction [6]. Free radicals, oxygen molecules, and peroxide anions are the chemical byproducts of the hydrogen peroxide breakdown process that are used to whiten teeth. These compounds are capable of forming stable bonds with other organic and inorganic molecules, including dark-pigmented chromophores. Then, via a reduction–oxidation reaction, these free radicals attach to and degrade the chromophore, modifying its optical structure and diminishing its ability to absorb light [7].

Although bleaching treatment has numerous benefits, there are concerns among clinicians, especially orthodontists, about how it affects the surface of the enamel and



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). how well adhesive resin bonds to previously bleached enamel. Prior studies report that bleaching can decrease the shear bond strength of orthodontic brackets [8–10]. Bleaching agents may impact the structure by decomposing organic and inorganic components, and they may also impact the morphology of enamel by causing porosity, cavities, depression, deeper enamel, loss of as prismatic layers, and a rough surface [11]. In-office bleaching temporarily reduced enamel microhardness and increased surface roughness [12].

Patients frequently mention dentin hypersensitivity as a side effect of bleaching therapy, in addition to tooth morphological changes. Some researchers theorize that postbleaching hypersensitivity is caused by the direct activation of nerve receptors in the tooth's structure by hydrogen peroxide, resulting in increased pain [7,13]. Utilizing a desensitizing agent is one of the methods that have been employed to reduce post-bleaching hypersensitivity. Desensitizing agents have two distinct modes of action. The degeneration of dentinal tubules, which prevents the passage of dentinal fluids and promotes remineralization, is required for one process. The second one reduces the sensory excitability of nociceptor by inhibiting pulp nerve activity [14]. Desensitizing agents include both fluoride and non-fluoride-containing substance, such as casein phosphopeptide-amorphous calcium phosphate fluoride (CPP-ACFP) and casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) [15].

Along with the need for orthodontic and cosmetic treatment, the popularity of ceramic brackets has grown and is becoming a popular choice, particularly for adult patients who desire orthodontic treatment without compromising aesthetics. Ceramic brackets were introduced to the dental profession in an effort to meet patients demands for aesthetics and were first utilized in clinical settings in 1987. Ceramic brackets are made of aluminum oxide [16]. They offer numerous benefits over stainless steel brackets, including biocompatibility, good aesthetics, resistance to chemical and temperature changes, and good adhesive strength [17]. Furthermore, dental ceramic also has limitations, such as fractural strength and wear resistance [18].

There is currently no research on the effect of post-bleaching desensitizing agents on the shear strength of enamel and bracket attachment. Bond strength levels between 5.9 MPa and 7.8 MPa are considered clinically acceptable, which is suggested by Reynolds [19]. These values are believed to be optimal for providing pressure and force to the teeth during mastication. The attachment strength value range is essential for clinical practice because it determines how readily the bracket will detach if it is below the normal range and how difficult it will be to debond if it is above the normal range, which could result in enamel damage.

Bonding delays of 1 to 3 weeks are recommended after bleaching to minimize clinical concerns associated with bleaching-mediated decreased bond strength [20]. On the other hand, patients who require orthodontic treatment place an important priority on their appearance. Some of them have discolored teeth and wish to have them bleached before having orthodontic treatment at the same meeting. Consequently, the purpose of this study is to investigate the impact of desensitizing agents on the shear bond strength of ceramic brackets following in-office bleaching procedures. The hypothesis of this study was that both fluoride and non-fluoride desensitizing agents increase the shear bond strength of ceramic brackets on previously bleached teeth. Fluoride and non-fluoride desensitizing agents will not result in a different ARI score following application.

2. Materials and Methods

2.1. Sample Preparation

Under the guidelines of the "ISO 29022:2013" standard, the samples were subjected to a series of shear-bond-strength tests. A total of 27 maxillary premolars (n = 27) that were extracted for orthodontic purposes and had no defect, a full and healthy crown, no cavities,

no fillings, and no fractures or cracks were chosen. Sample size determination in this study was calculated using the Federer formula, which is described as follows:

$$(t-1)(n-1) \ge 15$$

n: number of samples in each group;t: number of experimental groups.

This study used 3 groups (control group, a fluoride-containing desensitizing agent group, and a non-fluoride-containing desensitizing agent group).

$$(3 - 1) (n - 1) \ge 15$$

 $n - 1 \ge 15/2$
 $n - 1 = 7.5$
 $n = 8.5 \approx 9$

All of the samples were trimmed in the root area so that the distance from the cementoenamel junction to the base was 7 mm using a low-speed handpiece (W & H, Bürmoos, Austria) and diamond bur (Meisinger, Neuss, Germany).

Each selected tooth was then mounted in self-curing acrylic (Hillon, S Court Limited, Lancshire, UK) into a silicone mold ($25 \text{ mm} \times 25 \text{ mm} \times 13 \text{ mm}$) up to the cementoenamel junction based on its vertical axis. The teeth were then subjected to a prophylactic procedure, polished with a rubber cup and pumice using a low-speed handpiece, rinsed with water, and dried with air. All specimens were then stored for 24 h in a saline solution (Otsuka, Malang, Indonesia) prior to the next procedure.

Then, samples were randomly assigned to one of three groups (n = 9): group 1 (control group, CG), bleached teeth; group 2 (fluoride group, FG), bleached teeth and a fluoride-containing desensitizing agent (CPP-ACFP) applied; and group 3 (non-fluoride group, NFG), bleached teeth and a non-fluoride-containing desensitizing agent (CPP-ACP) applied. The enamel surfaces of all specimens were bleached using 37% hydrogen peroxide (Pola Office, SDI, Bayswater, Australia) as per the manufacturer's instructions. One cycle of bleaching lasted 20 min and was repeated up to three times, for a total bleaching time of 60 min. The samples were then rinsed with distilled water and desiccated with a dental chip blower.

Group 2 (Tooth Mousse Plus, GC, Tokyo, Japan) and group 3 (Tooth Mousse, GC, Tokyo, Japan) were then given a desensitizing agent. As per the manufacturer's instructions, a desensitizing agent was applied to the enamel surface of each sample with a microbrush for three minutes before being rinsed with distilled water and dried with a dental chip blower.

A total of 27 ceramic upper premolar brackets (20/40TM, American Orthodontics, Washington Ave, Sheboygan, WI, USA) with a surface area of 10.84 mm² were utilized in this study. The enamel's surface was etched with 37% phosphoric acid for 20 s (Hexa Etch, Bogor, Indonesia). A TransbondTM XT-bonding (3 M, Minnesota, USA) (TransbondTM XT) was applied to the bracket base, which was then placed on the labial enamel surface and firmly pressed into place to expel excess adhesive. The excess adhesive was removed, and a 10 s light cure was applied to all surfaces using a quartz tungsten halogen light-curing unit (Woodpecker, Guilin, China) with a light intensity of 450 mW/cm². Before the subsequent procedure, all specimens were stored for 24 h in a saline solution.

2.2. Shear-Bond-Strength Test

The shear-bond-strength (SBS) test was conducted in a universal testing machine (Pearson Panke Equipment Ltd., London, UK) at a speed of 1 mm/min. To allow force transmission in the occlusogingival direction, the chisel was placed parallel to the surface of the tooth bracket interface. The measured data were recorded in Newton unit. In order to translate the data to megapascals (MPa), data were divided by the bracket base area (10.84 mm²).

2.3. Mode of Failure

After bracket debonding, the entire sample enamel surface was examined under a stereomicroscope (SZX16, Olympus, Tokyo, Japan) with $10 \times$ magnification to determine residual adhesive. Remained adhesive was assessed according to the adhesive remnant index (ARI) classification by Bishara and Trulove [20] with the following criteria:

Score 1. All of the adhesive remains on the tooth;

Score 2. More than 90% adhesive remains on the tooth;

Score 3. More than 10% but less than 90% of the adhesive remains on the tooth;

Score 4. Less than 10% of the composite remains on the tooth;

Score 5. No composite remains on the tooth.

2.4. Scanning Electron Microscope (SEM) and EDX Evaluation

One sample from each group was chosen for bracket debonding in order to determine tooth surface morphology using a scanning electron microscope (Phenom Desktop ProXL Nanoscience Instruments, Phoenix, AZ, USA) with $10,000 \times$ magnification. This evaluation was followed by an EDX test to identify the elements of calcium and phosphorus on the surface of the enamel after bleaching and the application of desensitizing agent.

2.5. Data Analysis

To examine the data, SPSS statistical software was employed. The Shapiro–Wilk tests indicated that the data were normally distributed (p > 0.05). One-way analysis of variance (ANOVA) was used to analyze the shear-bond-strength data in order to determine whether there was a statistically significant difference between the shear bond strengths of the various groups. Finally, Tukey's multiple-comparison test (p < 0.05) was used to compare the mean SBS values. The ARI scores were evaluated by Kruskal–Wallis analysis to determine the differences between the groups.

3. Results

3.1. Shear Bond Strength

Table 1 contains the mean and standard deviation for the SBS in the three study groups. Highest SBS was found in fluoride group (15.36 ± 4.67 MPa) and the lowest SBS was found in control group (6.32 ± 4.83 MPa). Data were subjected to Shapiro–Wilk test and showed normal distribution (p > 0.05). Levene's test of homogeneity of variance found a value of 1.648 with a significance level of p > 0.05, indicating that the data had the same variation.

Table 1. Descriptive statistics and results of the ANOVA and Tukey's tests comparing SBS of three groups.

Crown	-	SBS (MPa)	One Way ANOVA Sig *	<i>p</i> -Value	
Gloup	11		One way ANOVA Sig	Group 2	Group 3
1	9	6.32 ± 4.83	0.011	0.003 *	0.044 *
2	9	15.36 ± 4.67		-	0.261
3	9	12.19 ± 6.81		-	-

* Significant difference between groups (*p* < 0.05); group 1 = control group; group 2 = fluoride group (CPP-ACFP); group 3 = non-fluoride group (CPP-ACP).

The ANOVA test indicated that there were statistically significant differences (p = 0.011) among the groups. Tukey's post-hoc test revealed that the fluoride (group 2) and non-fluoride-based (group 3) desensitizing agent exhibited a statistically significant increase in its SBS compared to the control groups (p > 0.05), while there was no significant difference in the SBS value between group 2 and group 3 (p < 0.05) (Table 1).

3.2. Adhesive Remnant Index

The ARI scores for all groups are listed in the Table 2. The result of Kruskal–Wallis test showed that no significant difference of ARI score among group (p > 0.05). Most frequent ARI score was 4 in all group.

Group	n	1	2	3	4	5	р
1	9	0	1 (11.1%)	3 (33.3%)	4 (44.4%)	1 (11.1%)	0.998
2	9	0	0	4 (44.4%)	5 (55.5%)	0	
3	9	0	1 (11.1%)	3 (33.3%)	4 (44.4%)	1 (11.1%)	

Table 2. Frequency distribution of the ARI in all groups and Kruskal-Wallis test results.

Group 1 = control group; group 2 = fluoride group (CPP-ACFP); group 3 = non-fluoride group (CPP-ACP).

3.3. Scanning Electron Microscope and EDX Analysis

The results of the SEM are displayed in Figure 1. Figure 1a (control group) shows a large number of big crater-like porosities (red arrow). The porosities are surrounded by structures that tend to be thin and irregular. In Figure 1b (fluoride group), only a few are of a shallow porosity and are surrounded by a thick, white circular structure (red arrow). In Figure 1c (non-fluoride group), several shallow pores are seen with an irregularly thick structure surrounding them (red arrow).



Figure 1. Scanning electron microscope examination of enamel surface after bracket debonding using 10,000× magnification. (a) Control group; (b) fluoride group (CPP-ACFP); (c) non-fluoride group (CPP-ACP). Red arrow outlined the characteristic of surface morphology.

Figure 2 and Tables 3–5 show the results of EDX analysis of the enamel surface after debonding the showed amount of minerals in each group. It showed that in the control group, the amount of phosphorous was lower than in the other two groups.



Figure 2. EDX analysis of enamel surface after bracket debonding. (**a**) Control group; (**b**) fluoride group (CPP-ACFP); (**c**) non-fluoride group (CPP-ACP).

Element Number	Element Symbol	Element Name	Atomic Concentration	Weight Concentration
8	О	Oxygen	40.32	32.75
20	Ca	Calcium	14.59	29.69
6	С	Carbon	24.65	15.03
15	Р	Phosphorus	8.43	13.26
7	Ν	Nitrogen	10.92	7.77
14	Si	Silicon	0.56	0.80
11	Na	Sodium	0.40	0.47
17	Cl	Chlorine	0.13	0.23

Table 3. Elements analysis for EDX results of group 1.

Table 4. Elements analysis for EDX results of group 2.

Element Number	Element Symbol	Element Name	Atomic Concentration	Weight Concentration
8	О	Oxygen	36.23	32.03
6	С	Carbon	44.02	29.22
20	Ca	Calcium	10.64	23.57
15	Р	Phosphorus	6.87	11.77
14	Si	Silicon	1.87	2.90
11	Na	Sodium	0.28	0.35
17	Cl	Chlorine	0.08	0.16

Table 5. Elements analysis for EDX results of group 3.

Element Number	Element Symbol	Element Name	Atomic Concentration	Weight Concentration
8	О	Oxygen	46.89	40.56
20	Ca	Calcium	10.96	23.75
6	С	Carbon	33.86	21.98
15	Р	Phosphorus	7.44	12.45

Element Number	Element Symbol	Element Name	Atomic Concentration	Weight Concentration
11	Na	Sodium	0.40	0.49
14	Si	Silicon	0.26	0.39
17	Cl	Chlorine	0.20	0.38

Table 5. Cont.

4. Discussion

The results of this investigation show that both fluoride and non-fluoride desensitizing agents increase the shear bond strength of ceramic brackets with composite resin cementation, implying that the null hypothesis was accepted. The highest SBS was found in the fluoride group, while the lowest SBS was found in the control group. The process of bleaching works by causing hydrogen peroxide to form free radicals that interact with chromogen macromolecules that stain the teeth and break them down into smaller molecules [21]. The reduction in post-bleaching shear strength is primarily attributable to changes in the enamel surface, such as enamel roughness and loss of prismatic formations, changes in the composition of the organic content of enamel, loss of calcium, and reductions in microhardness [22,23]. Remaining free radicals on the enamel may prevent the resin's penetration and polymerization, which would reduce the shear bond strength [20].

In this study, there was no time gap between bleaching and the bracket-bonding procedure. When the action was carried out, the possibility of residual free radicals was still high. High levels of free radicals persisting in the control group inhibited the infiltration and polymerization of the composite resin, resulting in the lowest shear bond strength compared to the other groups. The results of this study were supported by scanning electron microscope examination, which revealed substantial demineralization following bleaching in the control group, as evidenced by many porosity characteristics and irregular enamel prism structures. Demineralization is caused by residual free radicals that interact with organic components of the enamel, making the surface of the enamel rough. Hydrogen peroxide that is in contact for a long time can damage the organic matrix, which results in a decrease in the mechanical properties of the teeth [24].

An increase in shear bond strength occurred in both the fluoride and non-fluoride group because desensitization can cause remineralization on post-bleaching enamel, resulting in an improvement in the enamel structure, which results in better attachment of the mechanical bond between the bracket and the enamel [25]. The SEM images of the fluoride and non-fluoride groups support the results of this study by showing remineralization in both groups, marked by a less porous appearance than the control group. The depth of the pores in the fluoride and non-fluoride groups was also shallower than the pores in the control group, which looked deeper and darker in color. The structures around the porous in the fluoride and non-fluoride groups looked more uniformly white than that in the control group, which was surrounded by structures that tended to be thin and irregular.

The shear bond strength was lowest in the control group (bleaching without the administration of desensitizing agent). The findings of this investigation are consistent with the findings of prior investigations. Chauhan et al.'s research using the intracoronal bleaching method with three different types of bleaching materials revealed that the control group with no bleaching application had the highest shear-bond-strength value of the ceramic bracket when compared to other groups that were given bleaching action [9]. Rahul et al.'s research also revealed a decrease in the SBS of the ceramic bracket after in-office bleaching, whereas home bleaching had no significant effect on the SBS of the bracket-bonding process was recommended 24 h after bleaching [8]. In accordance with this investigation, Gungor et al., found that bleaching drastically lowered the SBS of the brackets [4].

The findings of this study contradict those of Abdulkareem and Al-Mulla, who found no difference in the shear strength of ceramic brackets after intracoronal bleaching. This is due to the ceramic bracket having zirconia particles coating the base of the bracket, which provides an undercut that strengthens the micromechanical bond. In addition, the translucent nature of ceramic brackets facilitates polymerization by light curing, and their high hardness prevents the peeling effect during the debonding procedure, resulting in high shear bond strength [26]. Other research by Oztas et al., also showed that home bleaching with 20% carbamide peroxide did not affect the shear strength of ceramic brackets [27].

Shear bond strength rises as a result of remineralization in both fluoride and nonfluoride groups. Remineralization can strengthen the bond and enhance the enamel's microscopic structure, but it has no effect on how etching creates microporous surfaces. These findings are consistent with those of Khargekar et al., who found that applying a fluoride-containing desensitizing agent before bracket bonding with composite resin produced stronger bracket bonds than the control group (etching application only) [28]. According to research by Kecik et al., non-fluoride desensitization boosted shear bond strength. On the enamel surface, non-fluoride desensitization serves as a buffer by supplying calcium and phosphate ions, preventing demineralization and enabling remineralization [29]. Research by Amuk et al., showed that the shear strength of the metal bracket after home bleaching increased after the application of non-fluoride desensitization. This study used an artificial salivary buffer solution. Saliva can increase the effectiveness of non-fluoride desensitization so that maximum remineralization occurs after the administration of non-fluoride desensitization [30].

Different results were reported by Da Rocha et al., who stated that fluoride desensitization can reduce shear bond strength. Fluoride can generate fluorapatite crystals on the enamel, which are tougher than hydroxyapatite, so teeth with higher fluoride content should be more resistant to etching and can interfere with the bracket-bonding process and decrease shear bond strength [31]. Research conducted by Ray et al., also showed that non-fluoride desensitization in the form of 26% potassium oxalate reduced shear bond strength. Oxalate is able to form calcium oxalate crystals that interfere with adhesion between resin and enamel [32].

The shear bond strength of the fluoride and non-fluoride groups was not significantly different. This shows that the two desensitizing materials have the same ability to increase the post-bleaching shear bond strength of the ceramic bracket with composite resin cementation. In both groups, there was remineralization caused by the desensitizing agent. Based on the SEM images, both groups showed remineralization, although the fluoride group showed a higher probability of remineralization because the pores were shallower and fewer and surrounded by a regular white structure, compared to the porous structure in the non-fluoride group, which was still surrounded by an irregular structure. These two images are different from the SEM image of the control group, which shows large numbers of deep pores surrounded by irregular structures. According to Thakkar et al., samples that received fluoride desensitization experienced remineralization at a higher average rate than the control group. The depth of lesions that previously received demineralization treatment was measured using a stereomicroscope. Remineralization was higher in the fluoride group due to fluoride's presence as an amorphous localization of calcium phosphate fluoride on the enamel surface, providing a reservoir to slowly release these ions [33]. Kristanti et al., reported that the application of fluoride-based desensitizing agents resulted in a decrease in mineral content that was lesser in post-bleaching enamel than desensitizing agents without fluoride. This relates to the development of fluorapatite, which is more durable and acid-resistant than hydroxyapatite [34].

In this study, the application of fluoride and non-fluoride desensitizing materials was carried out in a relatively short time (3 min) in accordance with the product usage instructions recommended by the manufacturer, so it can be estimated that the function of the fluoride reservoir in fluoride desensitization did not function optimally and the resulting remineralization between the two materials was still relatively the same. The SEM images from the fluoride and non-fluoride groups were also similar, with both displaying a shallow porous appearance surrounded by a whiter structure, indicating that fluoride

and non-fluoride desensitizing agents can cause remineralization and improve enamel conditions after bleaching. This is why there is no significant difference in shear strength between the fluoride and non-fluoride groups.

The ARI scores in the three groups did not differ significantly. In each group, the highest ARI score was 4, indicating that less than 10% of the adhesive substance remained on the enamel. Although the shear bond strength rose after the administration of fluoride and non-fluoride desensitizing agents, the ARI score was not altered. In this study, the amount of shear bond strength is unrelated to the ARI score; it is merely used to determine where the connection failed, whether it was on the adhesive, the bracket and adhesive, or the enamel and adhesive. The highest ARI score in this study indicates that the attachment failure was in the enamel and adhesive complex, which is more beneficial because it reduces the chance of enamel damage during debonding because the less residuals on the enamel, the less time it takes for the instrument to clean residual adhesive on the enamel.

According to Reynolds (1975), a clinically acceptable range for shear bond strength is 5.9 MPa–7.8 MPa, and the bond strength of orthodontic brackets must be sufficient to withstand masticatory and orthodontic forces. Excessive bond strength is not preferred since it might harm the enamel during bracket debonding [19,35]. The average shear strength in the control group (6.32 MPa) is still within the clinically acceptable range although the value is lower than the other groups, but the standard deviation of the control group is relatively large based on this; desensitizing materials can be considered to increase the shear bond strength so that the brackets could withstand orthodontic and masticatory force. The mean of shear bond strength of the fluoride group (15.36 MPa) and the nonfluoride group (12.19 MPa) was higher than the clinical range. As a result, enamel fracture during debonding is also a potential outcome.

Proper debonding techniques are necessary to decrease the danger of enamel fracture when debonding ceramic brackets [36]. Bora et al., proposed that debonding with a ligature cutter is the procedure that causes the least harm to enamel and is therefore recommended [37]. Subramani and Bollu wrote that the conventional debonding method using debonding pliers and a diamond bur that is recommended by the bracket manufacturer's directions is still recommended in order to minimize the occurrence of enamel damage [38].

5. Conclusions

The application of both desensitizing agents, including those that are fluoride and non-fluoride, increases the shear bond strength of ceramic brackets on previously bleached teeth. The use of both desensitizing agents had no effect on the ARI score values, with the highest ARI score in each group being 4, suggesting that less than 10% of the adhesive substance remained on the enamel.

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