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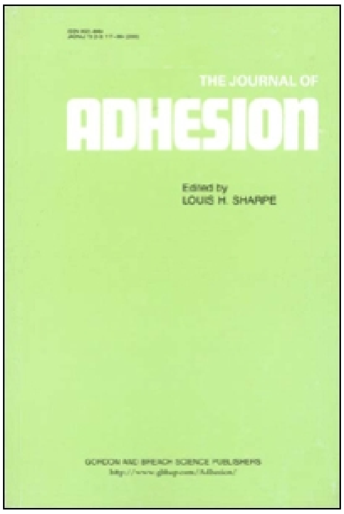

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Microtensile Bond Strength of Self-Adhesive Resin-Based Dental Cement to Bleached Enamel

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Microtensile Bond Strength of Self-Adhesive Resin-Based Dental Cement to Bleached Enamel

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This study investigated the effectiveness of bleaching agents on the micro tensile bond strength (μTBS) of self-adhesive luting cement and enamel. A total of 126 samples were prepared from the labial surfaces of permanent human maxillary central incisors and assigned into three groups with 42 samples each as: control, enamel bleached with 10% hydrogen peroxide (HP), and enamel bleached with 10% carbamide peroxide (CP). Rely X Unicem® (3M, ESPE) was used as self-adhesive resin cement. The total testing period for bleaching was selected as 14 days and the bleaching agents were applied 8 hours a day. The statistical one-way ANOVA model and Tukey HSD multiple comparison test ($\alpha = .01$) were used to assess the differences. The control group demonstrated the highest value (18.245 MPa) while HP and CP showed the much lower values of 11.150 and 14.222 MPa, respectively. Bleaching agents affect the μTBS negatively. Failure analysis of the fracture surfaces demonstrated that almost all samples showed adhesive failures at the cement-enamel interface.

Keywords: Bleaching; Carbamide peroxide; Hydrogen peroxide; Micro-tensile bond strength; Self- adhesive resin cement

INTRODUCTION

Indirect adhesive procedures constitute a substantial portion of contemporary esthetic restorative treatments. Resin cements are

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increasingly used for luting all ceramic, metal, or composite indirect, restorations like tooth-colored inlays, onlays, veneers, crowns and adhesive bridges; fiber and metal posts, and orthodontic brackets due to their excellent mechanical properties, better bond strengths, and improved esthetics compared with other traditional luting cements such as glass ionomer, zinc phosphate, and polycarboxylate resin [1–5].

Dental luting cements form the link between a fixed restoration and the supporting tooth structure [6]. The longevity of indirect adhesively cemented restorations is directly related to the adhesive effectiveness between dental tissues and resin cements [2,7,8]. The bonding capacity of adhesive luting agents is influenced by various factors related to the material itself like monomer composition, filler content, curing mode, curing efficiency, etc. [3,9]. and on the type of the adherent surfaces like enamel, dentin, alloys, ceramics, composites, etc. [2,8,10,11]. Most composite luting agents require that the tooth surface be modified and sometimes followed by the application of dental adhesives prior to luting of the restoration, although some have self-adhesive properties. Depending on the treatment of dental tissues, resin cements can be classified as total-etch, self-etch, and self-adhesive resin cements. Total-etch resin cements require the use of phosphoric acid followed by multi- or two-step total-etch adhesive before the application of the resin cement. Self-etch resin cements use an acidic primer, which is not rinsed away, to modify the dental tissues before bonding. Self-adhesive resin cements are able to bond to dental tissues without previous application of a bonding adhesive [12–15].

Bleaching is known to stimulate patients towards acceptance of additional esthetic dental procedures. When patients have their teeth bleached, they are often interested in veneers, replacement of old restorations, diastema closures, or other esthetic procedures [16]. In addition, bleaching may be indicated before an esthetic composite resin restoration placement to obtain a more pleasing final shade for the case [17]. Furthermore, when the results obtained from bleaching treatment are not esthetically acceptable, resin-bonded veneers are often required [18,19].

Numerous studies have addressed the question of whether various bleaching procedures affect the bond strength of composite resins to enamel specimens prepared from bovine or human teeth. When the bonding procedure is carried out immediately and up to 1 week after vital bleaching with hydrogen peroxide or carbamide peroxide-based bleaching agents, a reduction in enamel bond strength has been shown in previous research [18–24].

Based on the literature, previous studies generally centered on the bonding efficiency of different composite resin cements or different adhesive systems after bleaching applications; little or no information was found about self-adhesive resin cements. The question as to whether the bond strength of this resin cement can be affected following the application of different vital bleaching agents, when immediate bonding is needed, is still unclear. As a result, this study was designed to compare and investigate the bonding effectiveness of self-adhesive luting cement to enamel that has been bleached with 10% carbamide peroxide (CP) or 10% hydrogen peroxide (HP), using a standard microtensile bond strength (μTBS) test set-up.

MATERIALS AND METHODS

Twenty-one freshly-extracted, caries-free, permanent human maxillary central incisors with intact crowns were selected for the study and stored in 0.5% chloramine T solution at 4°C immediately after extraction. None of the extracted teeth had been stored for longer than 3 months [25]. In this study, the age difference among the collected teeth was ignored. Each tooth was cleaned with pumice with a profilaxis cup at low speed for 20 seconds.

1. Bleaching Application

Two different bleaching agents were used in this study. The 10% HP solution was prepared by diluting the 30% HP solution in 0.1 M phosphate buffer solution (PBS) and the 10% CP solution was prepared by dissolving carbamide peroxide powder (Sigma, St. Louis, MO, USA) in 0.1 M PBS [26]. For the experimental groups, the 14 teeth were immersed in bleaching solutions (seven teeth for HP and seven teeth for CP) for 8 hours in an incubator at 37°C in a 95% air, 5% CO_2 atmosphere. The procedure was repeated on 14 consecutive days. The teeth were incubated in artificial saliva between bleaching cycles. The bleaching agent was changed every day after the bleaching cycle was completed [27]. Teeth without bleaching agent application were prepared as controls.

2. μTBS Testing

The labial surfaces of the teeth were used as the substrates for testing. Preparation of tooth surfaces was carried out by first preparing a flat surface in enamel on a stationary disk using SiC sandpaper (Forcipol 1 Grinding and Polishing Machine, Metkon, Bursa, Turkey) and water cooling. The enamel samples were then divided into the following groups with seven samples each: 1) intact enamel + Rely X Unicem® (Lot number: 238351, 3 M ESPE, Seefeld, Germany); 2) enamel bleached with HP + Rely X Unicem; 3) enamel bleached with CP + Rely X Unicem.

A microhybrid composite resin (Filtek Z250®, 3 M ESPE, Seefeld, Germany) block samples of 8 × 4 × 6 mm were prepared and their bonding surfaces were blasted using 50 μm Al_2O_3 for 10 seconds, rinsed with distilled water, and air dried. Next, a silane coupling agent was applied to the restoration intaglio surface (Rely X Ceramic Primer®, 3 M ESPE, Seefeld, Germany), allowed to evaporate for 1 minute, and air dried for 30 seconds [13].

The composite resin blocks were cemented on the enamel according to the experimental groups. Rely X Unicem was used, according to the manufacturer's instructions, without any pre-treatment steps, i.e., without etching, priming, or bonding. Rely X Unicem was processed in a capsule system (Aplicap®, activated, and mechanically triturated with a mixing unit (triturator/amalgamator) for 15 s, as recommended by the manufacturer. Then the resin cement was applied to both the prepared surface of the resin block and enamel before seating. The composite resin blocks were placed under pressure (200 g), while the excess cement was carefully removed from the margins with disposable small brushes. All of the samples tested were halogen light-cured (Hilux 200 Curing Light, Benlioglu Dental, Turkey) for four 80-second periods at right angles to each other. Light intensity output was monitored with a radiometer (Hilux Curing Light Meter, Benlioglu, Turkey) throughout the bonding procedures to maintain at least 600 mW/cm^2. The restored samples were stored in distilled water at 37°C for 24 hours. Following storage in distilled water, samples were sectioned parallel to the adhesive interface to obtain slabs with a thickness of 1.0 ± 0.1 mm. Each of the slabs was then rotated 90 degrees and again cut perpendicular to the adhesive interface (Fig. 1) to obtain 1.0 ± 0.1-mm^2 beam samples [28] using a precision low-speed diamond saw (Microcut 175, Metkon, Bursa, Turkey). The inner beams from each experimental group were selected. Samples with either inappropriate dimensions or uneven enamel interfaces that could not be aligned perpendicular to the tensile load, or instantly fractured in the event that they did not fit to the jig properly before the microtensile testing, were discarded; thus, the total number of samples for each group was 42. The samples were tested individually by attaching them to a microtensile jig (Fig. 1) using cyanoacrylate glue (Zapit®, Dental Ventures of America, Inc, Corona, CA, USA). The 1.0 × 1.0-mm sticks were then submitted to a tensile load using

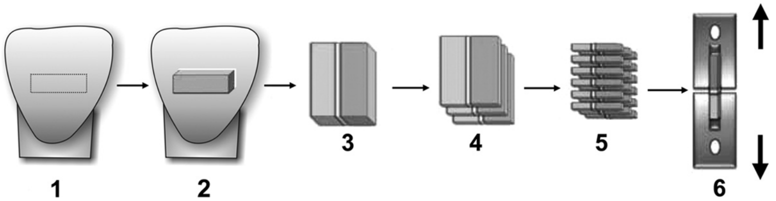

FIGURE 1 Microtensile sample preparation. 1) Flat enamel was selected on the labial surface. 2) Resin blocks were cemented. 3) Initial sectioning. 4) Sectioning parallel to the adhesive interface to obtain slabs with a thickness of 1.0 ± 0.1 mm. 5) Final sectioning to obtain 1.0 ± 0.1 mm^2 beam samples. 6) Samples were mounted in microtensile jig.

a universal testing machine (Model 3367, Instron Corp, Norwood, MA, USA) with a 10 N load cell and tested in microtensile strength at cross-head speed of 1 mm/mm until fracture. Microtensile bond strength calculations were made using the following equation: $s = L/A$, where s is the bond strength (MPa), L = test load (N), and A = area (mm^2). Microtensile bond strength data were statistically analyzed with one-way analysis of variance (ANOVA) and the Tukey HSD multiple comparisons test ($\alpha = .01$). The SPSS 11.0 software (SPSS, Inc., Chicago, IL, USA) was used for statistical analysis. Fractured samples were examined with a scanning electron microscope to determine the mode of failure. Failure modes were classified as adhesive, cohesive, or mixed.

RESULTS

Mean μTBS values and the standard deviations of each experimental group are summarized in Table 1. The ranking of the μTBS values

TABLE 1 Mean μTBS and Failure Mode Distribution by Sample, Per Group (n = 42)

Mean μTBS, in Mpa	Control 18.245 (0.391)^{a*}	HP 11.150 (0.846)^{b*}	CP 14.222 (0.505)^{c*}
Failure mode			
Adhesive	32	41	38
Cohesive	1	0	0
Mixed	11	1	4

*Different letters (*a*, *b*, *c*) indicate statistically significant differences for means between groups ($p < 0.01$).

TABLE 2 Summary of Tukey HSD Test

		MTBS Tukey HSD[a]		
		Subset for alpha = .01		
1) control, 2) HP, 3) CP	N	1	2	3
2	42	11.1500		
3	42		14.2229	
1	42			18.2450
Sig.		1.000	1.000	1.000

Means for groups in homogeneous subsets are displayed.
[a] Uses harmonic mean sample size = 42.000.

(MPa) among test groups was as follows: control > CP > HP. The statistical one-way ANOVA model containing all possible interactions yielded significant differences between control, HP, and CP groups. The post-hoc TUKEY test (Table 2) showed that the control group demonstrated the highest value (18.245 MPa) while HP and CP showed much lower values of 11.150 and 14.222 MPa, respectively. After HP and CP treatments, μTBS significantly

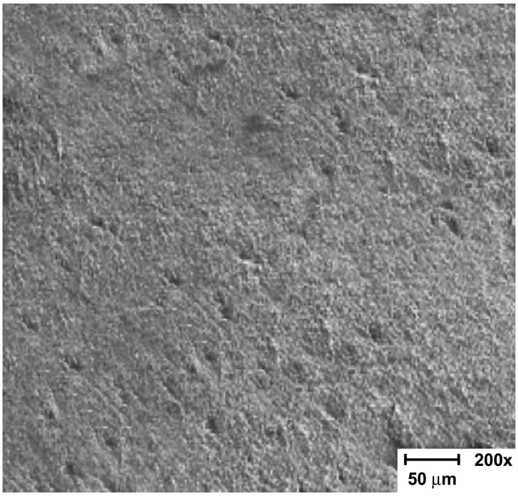

FIGURE 2 Adhesive failure from HP group.

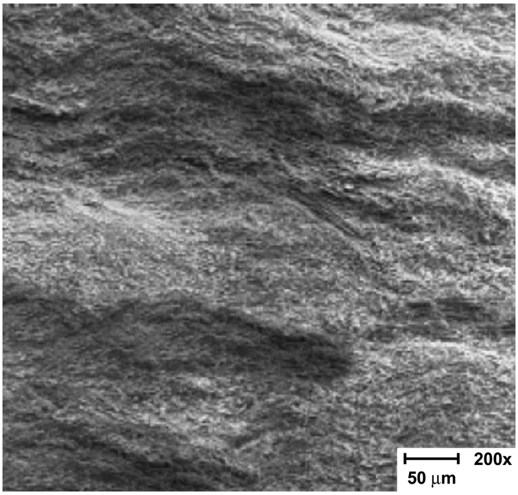

FIGURE 3 Cohesive failure from control group.

decreased ($p < 0.01$). Bond strength also statistically decreased following HP application compared with CP ($p < 0.01$). The fracture mode was predominantly adhesive for all groups (Table 1, Figs. 2–4).

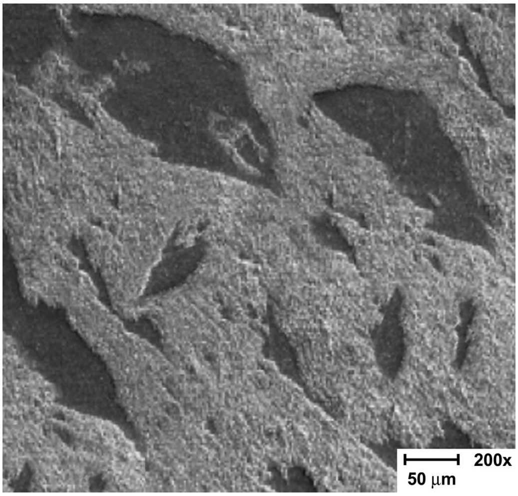

FIGURE 4 Mixed failure from CP group.

DISCUSSION

The use of a self-adhesive resin-based luting agent is becoming more popular today as an increasing number of dental restorations are routinely bonded. The present paper comparatively tested the μTBS of self-adhesive resin cement when it used was after different bleaching treatments.

Enamel bleaching is quite often carried out prior to placement of resin-bonded restorations [29]. Some of the possible side effects of vital bleaching are enamel surface alterations [30,31] and mineral loss [32]. Bonds to enamel/dentin may be altered following bleaching because of the presence of hydrogen peroxide [33]. It was suggested that the enamel and dentin organic matrix was altered by the hydrogen peroxide [34]. These changes may affect the formation of a strong and stable bond between the resin cement applied and the enamel surface. Spyrides *et al.* [35] evaluated the effect of three bleaching regimens (35% hydrogen peroxide, 35% carbamide peroxide, and 10% carbamide peroxide) on dentin bond strength. They found decreased bond strengths when the bleached teeth were immediately bonded. On the other hand, there was a significant increase in bond strength when bonding was delayed for a week. The results were similar for both the 35% carbamide peroxide and the 35% hydrogen peroxide. Dishman *et al.* [22], studying 25% hydrogen peroxide bleaching, found a decrease in the number of resin tags in the enamel and suggested that bleaching caused polymerization inhibition, which affected bond strength. Lai *et al.* [36] also suggested that the reduction in resin-enamel bond strength could be due to the delayed release of oxygen, which affects polymerization.

Hydrogen peroxide, in various concentrations, is the primary material currently used by the profession in the bleaching process. Carbamide peroxide-based systems with 10 to 15% carbamide peroxide release 3 to 5% H_2O_2. Carbamide peroxide ultimately breaks down into water, oxygen, and urea and hydrogen peroxide into water and oxygen [33,37,38]. Mechanical properties of polymer materials depend on their degree of polymerization. The lower degree of polymerization contributes to the lower bond strength for the treated specimens. To explain the lower degree of polymerization, it is important to note that oxygen inhibits polymerization of the resin adhesive system [39,40].

The agents used in this study were based on home-bleaching procedures using 10% hydrogen peroxide or carbamide peroxide. We used bleaching agents in solution rather than in a gel form, which is how this agent is normally delivered for mouthguard bleaching. This was done with the purpose of eliminating most variations that may exist between commercial products due to differences in composition, thickness, pH, and decomposition by-products [26]. The pH was 6.5 in the current study because popular commercial preparations have an average pH of 5 to 6.5 [41]. Since home bleaching comprises daily applications of 5 to 8 hours for 2 to 5 weeks [42,43], in this study, the total testing period was selected as 14 days and the bleaching agents were applied only 8 hours a day in order to simulate the clinical situation as closely as possible. Since the solutions were self-prepared, using the commercial products would alter the results.

One of the limitations of the *in vitro* studies is lack of saliva [44]. In this study, bleaching agents were left in contact with teeth 8 hours without the dilution or activation effects of saliva. It could be claimed that a mass of carbamide peroxide gel placed on a tooth in contact with a restorative material would have no effect as the gel would not be activated [44]. Similarly, in clinical applications of bleaching products, the concentration of hydrogen peroxide has been shown to be reduce a dramatically due, in part, to the effects of saliva [45]. Since bleaching treatments usually, but not always, are restricted to the anterior dentition, maxillary central incisors were chosen in this study. The type of teeth may also alter the results.

In terms of the overall bond strength values obtained in this study, the results were in part comparable with the results obtained by other investigators in similar studies [2,3,13,46]. Duarte *et al.* [13]. investigated μTBS of self-adhesive and self-etch resin cements to intact and etched enamel and reported that Rely X Unicem® exhibited a 13.03 MPa μTBS value. However, acid etching of enamel before the application of Rely X Unicem resulted in 32.92 MPa. Also, Hikita *et al.* [2]. reported a μTBS value of 19.6 MPa for enamel and 15.9 MPa for dentin, following the application of Rely X Unicem. On the other hand, our control group containing intact enamel + Rely X Unicem exhibited a μTBS value of 18.245 MPa (Table 1). Differences between μTBS values may be due to the type of teeth used and the experimental set-up. Al-Assaf *et al.* [46] reported a bond strength value of 4.47 MPa after testing the tensile bond strength of Rely X Unicem following thermal cycling using metallic rods that were bonded to dentin. μTBS of Rely X Unicem to different regions of dentin yielded 8.2, 5.7, and 5.5 MPa for superficial, deep, and cervical dentin regions, respectively [3].

Oxygen has a profound effect on the polymerization mechanism. It is known that hydrogen peroxide released from carbamide peroxide, due to its low molecular weight, can penetrate enamel to reach the dental pulp [47], and that there is a continuous leaching of the hydrogen peroxide that is retained in the bleached enamel [48]. In this study, bleaching applications (HP or CP) may result in oxygen accumulation within the enamel and oxygen might cause the inhibition of polymerization. In addition to the effect of oxygen, water content of the enamel structure might also account for the decrease of μTBS values compared with the control group. The presence of water might also originate from the decomposition of hydrogen peroxide. Carbamide peroxide dissociates into 3% HP, as mentioned before [43]. This means that both HP and CP bleaching agents have the same active agent. The results of the current study also indicated that increasing peroxide concentration led to a decrease in bond strength ($\alpha = 0.01$) compared with the control group (Table 1). In comparison, the effects of the two bleaching agents at 10% showed that bonding was less negatively influenced in the presence of CP than with HP. Failure analysis of the μTBS fracture surfaces (Figs. 2–4) demonstrated that when bonding following HP or CP application, almost all samples (~97 and 90%, respectively) showed adhesive failures at the cement-enamel interface (Table 1). Higher relative oxygen concentrations or water within the bleached tissues may also cause these types of failures due to poor resin infiltration, attachment, and polymerization. Although delayed resin bonding of up to 3 weeks post-bleaching has been recommended [21,22,33,36,42,43], every effort must be made to facilitate maximum retention of restorations under varying treatment conditions. Therefore, it would be valuable to investigate by further studies using different concentrations of carbamide peroxide and hydrogen peroxide, along with different time intervals of testing bond strength of self-adhesive resin-based cement.

CONCLUSIONS

In this study, the effect of bleaching agents on μTBS of self-adhesive luting cement and enamel was investigated. The results showed that both CP and HP treatments resulted in a decrease in the μTBS of self-adhesive luting cement. When immediate bonding after bleaching treatments is needed, it should be considered that CP would be more reliable than HP.

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