The effects of different desensitizer agents on shear bond strength of orthodontic brackets after home bleaching: an *in vitro* study

**Purpose**

The aim of this study was to test the null hypothesis that no difference exists between shear bond strength values of control and bleaching plus desensitizer applied groups.

**Materials and methods**

A hundred freshly extracted human premolar teeth were randomly divided into five groups. Group I served as the control group with no bleaching application, while only bleaching was achieved in Group II. Desensitizer containing potassium nitrate–fluoride and casein phosphopeptide amorphous calcium phosphate were applied in Groups III and IV, after bleaching respectively. A bleaching agent containing amorphous calcium phosphate was used in Group V. Shear bond strength tests were carried out using a universal testing machine (Instron Corp., Norwood, MA, USA). Remnant adhesive on the teeth and brackets was examined to score the adhesive remnant index. Kruskal-Wallis and Chi-Square tests were used for statistical analysis of the data.

**Results**

Statistically significant differences were found among the groups for shear bond strength values (*p*<.001). The shear bond strength of Group III (8.0±2.2 MPa) was significantly lower than the other groups (*p*<.05). The highest shear bond strength values were found for Group I (13.6±3.7 MPa) and Group IV (12.8±4.0 MPa). No statistically significant difference was observed between Group II (10.0±2.7 MPa) and Group V (10.8±2.9 MPa). The differences between adhesive remnant index scores of the groups were not statistically significant.

**Conclusion**

Casein phosphopeptide amorphous calcium phosphate gel application showed a similar shear bond strength value to the control group, while shear bond strength values decreased after using other desensitizers.

**Keywords:** Orthodontic bonding; tooth bleaching; dentin desensitizing agents; casein phosphopeptide amorphous calcium phosphate; shear bond strength

**Introduction**

Tooth color has been considered as one of the most important indicators of wealth, beauty and prosperity since ancient times. Discoloration can negatively impact quality of life, so tooth whitening procedures have become a popular dental application in recent years. Different combinations such as honey, burned salt and vinegar or calcium hypochlorite and oxalic acid were used for tooth whitening before peroxide was found in 1884 (1). In modern dental practice, bleaching procedures with various agents have been accepted as simple, safe, effective and predictable both for dentists and patients (2).

Tooth sensitivity caused by bleaching procedures is commonly experienced as a side effect (3), which is characterized by a short, sharp pain in response to cold, hot or sweet stimulus (4). The “hypodynamic theory” is generally accepted to explain the cause of sensitivity. According to this theory chemical, thermal or physical changes create the movement of fluids in dentinal tubules and these movements generate a stimulus on the related nerve receptor causing the initiation of sensitivity or pain (5). For this reason, patients frequently benefit from desensitizing agents that can be applied easily and safely (6).

Because cosmetic dentistry showed progress, some patients with dental malocclusion are interested in both orthodontic treatment and dental bleaching (7). During initiation of fixed therapy, the enamel structure is very important for bonding success (7) and the enamel structure is affected by bleaching and desensitizing agents (6). Controversial findings have been reported in the literature about bonding success of bleached enamel. Some studies showed significant shear bond strength (SBS) decrease after dental bleaching (8, 9), while no significant difference was also found (10).

Amorphous calcium phosphate (ACP) is a biologically active material for repairing tooth structure and reducing dentin hypersensitivity. Casein phosphopeptide is a milk protein derivative used as a remineralizing agent and contains phosphoseryl sequences which are stabilized with ACP (11). Casein phosphopeptide amorphous calcium phosphate (CPP-ACP) compound prevents the dissolution of calcium and phosphate ions and provides a supersaturated solution of bioavailable calcium and phosphates (12), so CPP-ACP has also been proposed by manufacturers for the prevention and treatment of dentin hypersensitivity (13, 14).

Different desensitizer agents have been used after bleaching, however it is important to determine the appropriate agent for increased bonding success. Although the effects of different desensitizers on SBS have been reported previously, to our knowledge, no study has compared ACP, CPP-ACP and potassium nitrate–fluoride agents in orthodontic bonding success. Therefore, the aim of this in vitro study was to evaluate the effects of different desensitizers on SBS of orthodontic brackets bonded to bleached teeth. The null hypothesis of this study was “no difference exists between SBS values of control and bleaching plus desensitizer applied groups”.

Materials and methods

Sample size estimation

The experiment protocol was approved by ethical committee of Erciyes University (approval code: 2016/73), and informed consent was obtained from all patients whose teeth were extracted for orthodontic purposes. A priori power analysis was completed using G*Power Ver 3.1.9.2 (Universität Kiel, Germany) software. Based on a 1:1 ratio between groups, a sample size of 20 specimens in each group is able to provide 90% power to detect significant differences with 0.86 effect size at a significance level of α= .05. Totally 100 intact freshly extracted human permanent upper first premolar teeth were randomly allocated to five groups with 20 teeth in each.

Specimen preparation

The teeth were embedded in acrylic resin blocks with ten teeth to a block. Acrylic resin was formed 1 mm below the cervical lines of the teeth. Alginate impressions were taken from the blocks and plaster models were obtained. Nail varnish was applied on the vestibular surfaces of the teeth at a thickness of approximately 0.5 mm to 1.0 mm to provide reservoir spaces on the bleaching trays. Low-density polyethylene plates were applied to the plaster models and bleaching trays were prepared using a vacuum thermoforming machine (ProForm, Dental Resources Inc., Minn., USA) (Figure 1).

Bleaching and Bonding Procedures

The teeth surfaces were cleaned and polished with pumice and rubber cups for 10 seconds to simulate a routine clinical procedure.

Group I: This group was the control group and received no bleaching. Etching was performed with 37% orthophosphoric acid gel (3M Dental Products, Minn, USA) for 15 seconds. The etching material was removed from the teeth surfaces with air-water spray for 10 seconds, and teeth were dried for

Figure 1. Acrylic resin blocks and bleaching trays.
10 seconds. A thin uniform coat of the sealant Transbond XT primer (3M Unitek, Monrovia, USA) agent was applied to the etched surface. Premolar brackets were bonded using Transbond XT (3M Unitek Monrovia, USA) composite and light cured with a light-emitting diode curing unit (Valo, South Jordan, USA) for 20 seconds according to the manufacturer’s instructions (10 seconds per each approximal side). Teeth were stored in salivary buffer for 24 hours before the SBS test in order to obtain the highest adhesive bond strength (15).

Group II: Teeth were bleached with 22% carbamide peroxide (CP) agent (Hollywood Smiles Bleaching Pen; Onuge Oral Care Co, Henan, China) according to manufacturer’s instruction. The procedure was repeated every day for one week. Bleaching material was applied to the vestibular surfaces of the teeth with a pen applicator and spread across the surface with the pen tip brush. Then, trays were placed onto the teeth and blocks were covered with salivary buffers. Half an hour later, trays were removed and kept for later use. The surfaces of the teeth were washed with saliva and stored in the salivary buffers.

Group III: 22% CP and desensitizer agent containing potassium nitrate–fluoride (UltraEZ; Ultradent Products Inc., South Jordan, USA) was applied to the teeth consecutively. The procedure was repeated daily for 1 week. For each application, the bleaching procedure was performed in the same manner as for Group II. Then, the teeth were washed and dried with a sponge. Desensitizer material containing potassium nitrate–fluoride (UltraEZ; Ultradent Products Inc., South Jordan, USA) was applied to the vestibular surfaces of the teeth with bonding brushes. Trays were placed for the desensitizer and blocks were covered with salivary buffers. Trays were removed after 30 minutes and the teeth were covered with salivary buffers until the next application.

Group IV: CPP-ACP gel (GC Tooth Mousse gel; GC Int Corp, Tokyo, Japan) was applied to the teeth after bleaching with 22% CP. The application procedures were repeated as for Group III.

Group V: 22% CP gel containing ACP (NiteWhite ACP; Discus Dental, Culver City, USA) was used. 22% CP and ACP combination gel (NiteWhite ACP; Discus Dental, Culver City, USA) was applied to the teeth every day for a week. Bleaching material was applied to the vestibular surfaces of teeth and spread on the surface. After, trays were placed on the teeth and blocks were covered with salivary buffers. Half an hour later, trays were removed and the surfaces of teeth were washed with saliva and stored in the salivary buffers.

During the experimental procedure, blocks were stored in buffers with saturated artificial saliva (Table 1) to imitate intraoral conditions and the buffers were renewed twice a day.

At the end of the seventh day, all teeth were washed and pumiced. The bonding procedure was performed in the same manner as for the control group. Upper first premolar metal brackets were used (American Orthodontics Roth system, Master series, Sheboygan, Wisconsin, USA) for orthodontic bonding.

Shear bond strength testing

Shear strength tests were performed using an Instron Testing Machine (Instron Corp., Norwood, USA). A steel rod with one flattened and sharpened end was attached to an upper grip (movable head) connected to the load cell. The blade was positioned at the bracket-enamel interface (Figure 2) and an occluso-gingival directed load was applied. Cross-head speed was adjusted at 1 mm/min during recording the data.

After debonding, the teeth and brackets were examined to detect existence of any remnant adhesive after bracket removal. These results were scored according to the adhesive remnant index (ARI) (16). The examination of the enamel surface was performed by a blind investigator to group allocations. The ARI scores ranged from 0 to 3 (Table 2).

### Table 1. Composition of the artificial saliva

<table>
<thead>
<tr>
<th>Components</th>
<th>Per cent</th>
</tr>
</thead>
<tbody>
<tr>
<td>NaCl</td>
<td>0.08</td>
</tr>
<tr>
<td>KCl</td>
<td>0.12</td>
</tr>
<tr>
<td>MgCl₂·6H₂O</td>
<td>0.01</td>
</tr>
<tr>
<td>K₂HPO₄</td>
<td>0.03</td>
</tr>
<tr>
<td>CaCl₂·2H₂O</td>
<td>0.01</td>
</tr>
<tr>
<td>Sodium Carboxymethyl Cellulose</td>
<td>0.10</td>
</tr>
<tr>
<td>Ion-Exchanged Water</td>
<td>99.6</td>
</tr>
</tbody>
</table>

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**Figure 2.** Test apparatus used for shear bond strength testing.
Statistical analysis

Statistical analyses were performed using Statistical Package for Social Sciences, software (Version 20.0, IBM Corp.; Armonk, NY, USA). The Shapiro-Wilk W test was used to test the data for normality. Data was not normally distributed, so non-parametric tests were used. Group differences for SBS values and ARI scores were tested using Kruskal-Wallis and Chi-Square tests, respectively. A multiple comparison procedure (Student-Newman-Keuls Method) was used to isolate the group or groups that differ from the others. Confidence level was set to 95% and p<0.05 was considered statistically significant.

Results

The null hypothesis was rejected. A statistically significant difference was found between the SBS values of groups (p<0.001, Table 3). SBS values of Group I (control; 13.6±3.7 MPa) and IV (12.8±4.0 MPa) were significantly greater than other groups (p<0.05 for all comparisons). Group III had the lowest SBS value (8.0±2.2 MPa). No statistically significant difference was observed between Group II (10.0±2.7 MPa) and Group V (10.8±2.9 MPa).

Adhesive remnant index scores are presented in Table 4. No significant differences were found between groups in ARI evaluations.

Discussion

The decreasing SBS values of orthodontic brackets after bleaching were attributed to the changes in the enamel structure. Conventional acid etching with 37% phosphoric acid leads to prism core demineralization, prism sheath demineralization or both types of demineralization (17). Composite resin adheres to etched enamel by mechanical bonding through unfilled resin penetration and polymerization in these surface irregularities. However, bleached teeth lose these retentive areas which are prepared by etching and required for bonding (6,17). Titley et al. (18) observed sparse, short, poorly defined enamel tags immediately after hydrogen peroxide (HP) bleaching. In this study, bleaching with 22% CP led to a statistically significant reduction in SBS. Oskooe et al. (19) reported reduction of SBS with bleaching, while Miles et al. (9) showed

Table 2. The Adhesive Remnant Index (ARI) scores

<table>
<thead>
<tr>
<th>Index</th>
<th>Enamel Adhesive Remnant</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>No adhesive left on the tooth (%10)</td>
</tr>
<tr>
<td>1</td>
<td>Less than half of the adhesive left on the tooth</td>
</tr>
<tr>
<td>2</td>
<td>More than half of the adhesive left on the tooth</td>
</tr>
<tr>
<td>3</td>
<td>All adhesive left on the tooth (%90)</td>
</tr>
</tbody>
</table>

Table 3. Descriptive statistics and multiple comparisons of the shear bond strength values of groups

<table>
<thead>
<tr>
<th>Groups</th>
<th>N</th>
<th>25% Median</th>
<th>75% Mean</th>
<th>SD</th>
<th>Significance</th>
<th>*Multiple Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>20</td>
<td>10.0</td>
<td>13.5</td>
<td>17.0</td>
<td>13.6</td>
<td>3.7</td>
</tr>
<tr>
<td>CP</td>
<td>20</td>
<td>9.0</td>
<td>9.5</td>
<td>12.0</td>
<td>10.0</td>
<td>2.7</td>
</tr>
<tr>
<td>CP+D</td>
<td>20</td>
<td>6.5</td>
<td>7.0</td>
<td>9.0</td>
<td>8.0</td>
<td>2.2</td>
</tr>
<tr>
<td>CP+CPP-ACP</td>
<td>20</td>
<td>9.5</td>
<td>13.0</td>
<td>17.0</td>
<td>12.8</td>
<td>4.0</td>
</tr>
<tr>
<td>CP+ACP</td>
<td>20</td>
<td>9.0</td>
<td>10.0</td>
<td>12.0</td>
<td>10.8</td>
<td>2.9</td>
</tr>
</tbody>
</table>

CP: carbamide peroxide; CP+D: carbamide peroxide plus potassium nitrate–fluoride containing desensitizer; CP+ACP: amorphous calcium phosphate containing carbamide peroxide; CP+CPP-ACP: carbamide peroxide plus casein phosphopeptide-amorphous calcium phosphate

*Groups with different letters are significantly different from each other

Chi-square test revealed no significant differences between groups

Table 4. Frequency Distribution of the Adhesive Remnant Index (ARI) Scores

<table>
<thead>
<tr>
<th>Groups</th>
<th>n</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>Sig.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>20</td>
<td>1</td>
<td>2</td>
<td>7</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>CP</td>
<td>20</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>8</td>
<td></td>
</tr>
<tr>
<td>CP+D</td>
<td>20</td>
<td>4</td>
<td>3</td>
<td>6</td>
<td>7</td>
<td>NS</td>
</tr>
<tr>
<td>CP + CCP-ACP</td>
<td>20</td>
<td>2</td>
<td>2</td>
<td>6</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>CP + ACP</td>
<td>20</td>
<td>2</td>
<td>3</td>
<td>7</td>
<td>8</td>
<td></td>
</tr>
</tbody>
</table>

n: sample size; NS: non-significant; CP: carbamide peroxide; CP+D: carbamide peroxide plus potassium nitrate–fluoride containing desensitizer; CP+ACP: amorphous calcium phosphate containing carbamide peroxide; CP+CPP-ACP: carbamide peroxide plus casein phosphopeptide-amorphous calcium phosphate
decreased SBS values when bleaching was carried out with CP. In contrast, Bishara (20) showed that both in-office bleaching with 25% HP and at-home bleaching with 10% CP did not affect the SBS of orthodontic brackets to enamel.

Another reason for reduction in SBS with bleaching was accepted as residual peroxide on the enamel surface (21). Torneck et al. (22) found a significant decrease in bond strength related to the presence of residual peroxide or peroxide-related substances on the enamel surface. Neutralizing these substances with antioxidants increases the bond strength of bleached enamel (23).

Sensitivity after bleaching is a common adverse effect, and a desensitizer including potassium nitrate and fluoride ions decreases sensitivity complaints by obstructing the tubules (24), which can lead to the failing of the most important stage of bonding: adhesion between enamel and resin. Our findings on CP plus desensitizer containing potassium nitrate–fluoride indicated that using this desensitizer after CP bleaching can reduce SBS, and possible reason for this reduction may be the weakening of enamel adhesive linkage. Similar to our findings, Turkkahraman et al. (6) also reported that bleaching and desensitizer applications significantly reduce the SBS of orthodontic brackets.

Amorphous calcium phosphate and CPP-ACP compositions treat hypersensitivity of the teeth by releasing free calcium and phosphate ions in a different way to the desensitizer mechanism (12, 14). According to results of this study, SBS values of samples which used CPP-ACP were similar to the control group. SBS values were higher in the ACP bleaching group compared to bleaching plus desensitizer containing potassium nitrate–fluoride applications. An increase in SBS values with CPP-ACP was also reported by Lu et al. (25). Similarly, Oskoei et al. (19) found the same SBS values between the control group and the group that received CPP-ACP after bleaching.

In this study, CPP-ACP application after bleaching provided higher SBS than the application of bleaching agent containing ACP. This difference may be attributed to the difference in application procedures for these two materials. Saliva can enhance the effectiveness of CPP-ACP, so more effective results can be obtained if increased contact of CPP-ACP and saliva is maintained (26). ACP was applied within the CP gel, so there is no direct contact between ACP and a salivary environment, while CPP-ACP gel was applied separately after CP gel. So, remineralized enamel following CPP-ACP application after bleaching may cause a higher SBS value than ACP samples. In addition, the obliterated enamel structure with calcium and phosphate ions on the destructed core and sheath of prisms during peroxide application in the CP-containing ACP group may cause a more acid-resistant surface for etching with CP-containing ACP, compared with CPP-ACP plus CP. Machado et al. (27) found similar lowering SBS with peroxide-ACP combined gel and peroxide gel applications, compared with the control group. Although no significant difference was found between CP and CP with ACP applications in this study, a slight increase was observed with CP containing ACP gel compared with only CP application.

The evaluation of ARI scores showed that the site of bond failure is not significantly affected by the experimental procedures of this study. Reynolds (28) reported that successful clinical bonding should provide bond strength of 5.9 to 7.8 MPa, and these results have been accepted as threshold values for SBS studies. The SBS values of our study ranged between 8 to 15 MPa, meaning there is no contraindication for using ACP, CPP-ACP and the desensitizers for the elimination of sensitivity caused by bleaching in terms of bonding failure. Even though SBS values of experimental groups of this study were higher than acceptable clinical limits, using the CPP-ACP agents for the elimination of sensitivity before the fixed orthodontic therapy can be more safer than CP alone, CP containing ACP, and CP plus desensitizer containing potassium nitrate–fluoride in terms of bonding success. In the light of these findings, the clinicians will be able to recommend patients who need orthodontic treatment to use CPP-ACP agents as a desensitizer.

Artificial saliva was used to simulate the oral environment, but heat and humidity conditions in the oral cavity are highly variable. A direct correlation with in vitro design and in vivo oral conditions might be inaccurate during the interpretation of results. Further studies simulating in vivo settings which provide real heat, stress, acidity and humidity are required for more valid and reliable results.

**Conclusion**

Bleaching with 22% carbamide peroxide, 22% carbamide peroxide containing ACP and 22% carbamide peroxide bleaching plus desensitizer containing potassium nitrate–fluoride applications significantly decreased shear bond strengths of orthodontic brackets bonded to human enamel compared with the control group. CPP-ACP application after 22% carbamide peroxide bleaching and the control groups showed similar shear bond strength values. No statistically significant difference was observed in the site of bond failure for all groups. According to these findings, clinicians may recommend patients who need orthodontic treatment to use CPP-ACP after 22% carbamide peroxide bleaching as a safer desensitizer for the prospective orthodontic bonding procedure.

**Ethics Committee Approval:** Ethics committee approval was received for this study from the ethics committee of Erciyas University (Approval code: 2016-73).

**Informed Consent:** Written and verbal informed consent was obtained from patients and/or patients’ parents who participated in this study.

**Peer-review:** Externally peer-reviewed.

**Author Contributions:** NGA, AB and GK designed the study. NGA and YÜ generated the data. NGA gathered the data. NGA, AB, and GK analyzed the data. NGA wrote the majority of the original draft. NGA, AB and GK participated in writing the paper. All authors approved the final version of the paper.

**Conflict of Interest:** The authors have no conflicts of interest to declare.

**Financial Disclosure:** The authors declared that this study has received no financial support.
Farklı hassasiyet gidericilerin ortodontik braketlerin ev tipi be yazatma sonrası brakettan bağlanma dayanımı üzerinde etkileri. Amac: Bu çalışmada, ev tipi be yazatma sonrası farklı hassasiyet giderici ajan uygulanmanın, ortodontik braketlerin bağlanma dayanımı üzerine etkilerinin değerlendirilmesi ve karışımların oluşturulması amaçlanmıştır. Gereç ve Yöntem: Çalışmamızda 100 adet insan premolar diş grubu alınmıştır. Grup I hiçbir be yazatma uygulaması yapılmayan kontrol grubu olarak belirlenmiştir; Grup II de yalnız be yazatma işlemi geçirilmiştir. Grup III ve Grup IV' e be yazatma ve karışımların uygulanması olarak Instron Test Cihazı kullanılmıştır. Test sonrası diş ve bracket yüzeyinde kalan a deziv, a deziv endeksi ile skorlanmıştır. Veri analizi K ruskal-Wallis ve Chi-Kare testlerinden faydalanılmıştır. Bulgular: Gruplar arasına bağlanma dayanımı karışımlarına istatistiksel olarak anlamlı fark bulunmuştur (p<.001). Grup II' in bağlanma dayanımı değeri (8,0±2,2 MPa) diğer gruppardan anlamlı olarak düşük (p<.05). En yüksek bağlanma dayanımı değerlerinin Grup I (13,6±3,7 MPa) ve Grup IV'de (12,8±4,0 MPa) olduğu gözlenmiştir; Grup II (10,0±2,7 MPa) ve Grup V (10,8±2,9 MPa) arasında anlamlı farklık yoktur. Gruplar arası anlamlı endeks skorları arasında istatistiksel olarak anlamlı fark bulunmamıştır. Sonuç: Kazein fosfopeptit-amorfol kalsiyum fosfat içeren hassasiyet giderici bracket bağlanma değerlerini azaltmıştır. Anahat kelimeler: Ortodontik bonding; diş beyazlatma; dentin hassasiyet gidericiler; kazein fosfopeptit-amorfol kalsiyum fosfat; bracket bağlanma dayanımı

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