

# Effect of Delayed Bonding and Antioxidant Application on the Bond Strength to Enamel after Internal Bleaching

Halil İbrahim Kılınc, DDS, PhD,<sup>1</sup> Tuğrul Aslan, DDS, PhD,<sup>2</sup> Kerem Kılıç, DDS, PhD,<sup>1</sup> Özgür Er, DDS, PhD,<sup>3</sup> & Gökmen Kurt, DDS, PhD<sup>4</sup>

<sup>1</sup>Department of Prosthodontics, Erciyes University, Faculty of Dentistry, Kayseri, Turkey

<sup>2</sup>Department of Restorative Dentistry and Endodontics, Erciyes University, Faculty of Dentistry, Kayseri, Turkey

<sup>3</sup>Department of Endodontics, Trakya University, Faculty of Dentistry, Edirne, Turkey

<sup>4</sup>Department of Orthodontics, Yeni Yüzyıl University, Faculty of Dentistry, İstanbul, Turkey

## Keywords

Internal bleaching; enamel; antioxidant; bonding.

## Correspondence

Halil İbrahim Kılınc, Department of Prosthodontics, Faculty of Dentistry, Erciyes University, Kayseri, Turkey. E-mail: hikilinc@erciyes.edu.tr

*This study was presented at the 38th Annual Conference of the European Prosthodontic Association & 21st Scientific Congress of the Turkish Prosthodontic and Implantology Association in İstanbul, Turkey on 25–27 September 2014.*

*The authors deny any conflicts of interest.*

Accepted January 26, 2015

doi: 10.1111/jopr.12303

## Abstract

**Purpose:** This study evaluated the effect of delayed bonding and antioxidant application (AA, 10% sodium ascorbate) after internal bleaching (35% carbamide peroxide) on the shear bond strength of an adhesive cement to enamel.

**Materials and Methods:** Eighty-four human maxillary central incisors were endodontically treated. The control group remained unbleached with no AA. Experimental groups were all internally bleached. The buccal enamel was finished and polished with metallographic paper to a refinement of #600, in order to obtain a 5-mm<sup>2</sup> flat bonding area. An adhesive cement (Clearfil Esthetic) was placed into a plastic tube with internal diameter of 3 mm and a 3-mm height and cured on the enamel. Bonding occurred either immediately after bleaching (group Im), a 7-day delay (group 7), or a 14-day delay (group 14), and half the specimens were treated with antioxidant application (groups Im-AA, 7-AA, and 14-AA). Shear bond strength testing was performed on a universal testing machine, and data were analyzed with ANOVA and Fisher test (5%).

**Results:** Delaying of bonding is a useful factor for enhancing shear bond strength ( $p < 0.05$ ), whereas AA only enhanced shear bond strength after 7 days delayed bleaching ( $p < 0.05$ ). The highest bond strength was noted in groups 7-AA (20.51 ± 4.5 MPa), 14 (19.82 ± 4.6), 14-AA (20.27 ± 4.4), and control (20.51 ± 5.1), which were not significantly different from each other.

**Conclusions:** After internal bleaching, adhesive cementation to enamel is recommended only when delayed 14 days, or delayed 7 days with sodium ascorbate application.

The nonesthetic sight of discolored anterior teeth is a leading concern for a person's appearance, and modern dentistry has alternatives for this problem. Considering the needs of the patient and to achieve esthetic results, whitening may be recommended before restoring teeth with bonded restorative appliances.<sup>1</sup> Clinicians should evaluate the etiology of discoloration when bleaching is selected as a treatment. Intrinsic staining of nonvital teeth can effectively be removed by internal bleach.<sup>2</sup> Hydrogen peroxide, sodium perborate, sodium percarbonate, and carbamide peroxide can be used as bleaching agents to penetrate enamel and dentin and solubilize or decolorize the chromogens.<sup>3</sup>

On the other hand, clinicians should consider the effects of the selected bleaching protocol on possible future dental treatments.<sup>4</sup> Additional esthetic resin-bonded restorations, such

as laminate or full-ceramic veneers, are often required because the results of the bleaching treatments are not always esthetically acceptable.<sup>5</sup> However, because of the bleaching, the bond strength of adhesive cement to dentin or enamel is reduced when the teeth are restored with adhesive-bonded veneers, and some studies have identified the presence of residual peroxide formation, which interferes with resin polymerization.<sup>6–10</sup>

Various protocols have been suggested to resolve this clinical problem.<sup>11–13</sup> The general approach is to postpone any bonding procedure by 24 hours to 4 weeks because some studies have reported that tooth bonding is insufficient within this timeframe.<sup>8,14,15</sup> In contrast, recent studies have proven that such a delay is unnecessary,<sup>9,10,16</sup> and even 10-minute applications of 10% sodium ascorbate on the bleached tooth surface is sufficient to reverse bond strength reduction.<sup>17</sup>

The salt of ascorbic acid, sodium ascorbate, is a potent antioxidant that can eliminate free radicals in biological systems, including oral environments, because of its nontoxicity. Therefore, sodium ascorbate is commonly used in the food industry as a safe agent.<sup>10,18</sup> Its efficacy for reversing compromised bonding has been proven by several studies.<sup>8-10,16-18</sup> The effect of vital bleaching on bonding was evaluated in all these studies; however, the effect of internal bleaching of endodontically treated teeth on bonding was evaluated in only a few of them. Therefore, the purpose of this study is to investigate the effect of delayed bonding and 10% sodium ascorbate treatment as application of antioxidant (AA) after internal bleaching with carbamide peroxide on the shear bond strength of an adhesive cement to enamel.

## Materials and methods

Eighty-four similarly sized, intact human maxillary central incisors extracted for periodontal reasons were collected and stored in normal saline. Any remnant periodontal ligaments on the teeth were removed with a curette. The pulp chamber was accessed from the lingual surface using a water-cooled spherical diamond drill (Dentsply/Maillefer, Ballaigues, Switzerland) in a high-speed handpiece. The pulp was removed with endodontic instruments, and the root canals were prepared with ProTaper rotary instruments (Dentsply/Maillefer) up to the ProTaper F3 instrument. Irrigation was performed using 5 ml of 2.5% NaOCl between each instrument during the cleaning and shaping of the root canal. The final irrigation was performed using 5 ml of 17% ethylenediaminetetraacetic acid (EDTA) for 1 minute, followed by copious irrigation with distilled water. After drying with paper points, the root canal treatments were performed with a cold lateral condensation technique using gutta-percha (Dentsply Maillefer, Petropolis, Brazil) and resin sealer (AH Plus; Dentsply DeTrey GmbH, Konstanz, Germany). Then, root canal filling material was shortened 2 to 3 mm subgingivally from the cemento-enamel junction. A composite resin base was laid on the root canal filling material.

Teeth were randomly divided into seven groups of 12 teeth each and stored in distilled water ( $37^{\circ}\text{C} \pm 1^{\circ}$ ) in individual, closed bottles. The groups were separated according to the AA and the delaying time after bleaching (Table 1). In the three bleached groups, bonding procedures were performed immediately and after 7 and 14 days. In the other three bleached groups, the luting procedures were performed immediately and after 7 and 14 days following AA for 10 minutes. AA was not performed on the last unbleached specimens in order to have a control group.

Thirty-seven percent carbamide peroxide bleaching gel (Whiteness SuperEndo; Dentscare, Ltda, Joinville, Brazil) was inserted into the pulpal cavities of the teeth, which were washed and dried before bleaching. After a piece of absorbent paper was placed on the gel, the access cavities were sealed with glass ionomer cement (Fuji IX GP; GC Corp., Tokyo, Japan). The teeth comprising each group were stored in distilled water ( $37^{\circ}\text{C} \pm 1^{\circ}$ ) in individual, closed containers. After 3 days, the bleaching agent was replaced with a fresh solution. This exchange was performed twice more for each bleaching

**Table 1** Experimental design of this study

Groups	n	Condition	Delaying time	Application of sodium ascorbate
C	12	Unbleached	–	Without
Im	12	Bleached	–	Without
Im-AA	12	Bleached	–	With
7	12	Bleached	7 days	Without
7-AA	12	Bleached	7 days	With
14	12	Bleached	14 days	Without
14-AA	12	Bleached	14 days	With

group. After 9 days, the internal bleaching of all experimental specimens was completed. The control groups were not bleached but were restored with composite directly after cleaning the remnants of the endodontic treatment out of the access cavities.

After restoring the access cavities of the bleached teeth with composite resin, all teeth were completely embedded in self-cured acrylic resin to be made into separate molds ( $20 \times 15 \times 10 \text{ mm}^3$ ). Before the bonding procedure, the buccal surface of each tooth was ground and polished with metallographic paper to a refinement of #600 in the polishing machine (TegraPol 11; Struers, Ballerup, Denmark) so as to obtain a  $5 \text{ mm}^2$  flat enamel area.

Using a syringe, a 10% sodium ascorbate solution was applied to the AA groups. Drops of the sodium ascorbate were agitated on the enamel surfaces with a microbrush for 10 minutes. Then specimens were washed and gently dried. The groups without AA were not subjected to any procedure at this time.

A dual-cure resin cement (Clearfil Esthetic Cement; Kuraray, Tokyo, Japan) was used as an adhesive for this study. According to its bonding procedure, flattened enamel surfaces were etched with K-Etchant Gel for 10 seconds, washed, and gently dried. Then equal amounts of ED Primer II Liquids A and B were mixed and applied to the enamel surfaces of the specimens for 30 seconds. The surfaces were dried gently. Clearfil Esthetic Cement pastes A and B were mixed and placed into a plastic tube with an internal diameter and height of 3 mm, and then pressed on the flat surfaces of the specimens during the initial curing for 10 seconds. Excess adhesive cement was removed using a dental probe. Then, resin cement was polymerized from each direction with an LED curing device (light intensity:  $1000 \text{ mW/cm}^2$ ; Elipar FreeLight 2 LED Curing Light; 3M ESPE, St. Paul, MN) for 40 seconds. The plastic tube was left in place for the remainder of the experiment.

After 24-hour storage at 100% humidity, the shear bond strength test was performed with a universal testing machine (Instron, Canton, MA) in which the load was applied to the enamel/cement interface using a knife-edged loading head at a 1 mm/min constant speed. The maximum load at failure was recorded in newtons (N) and converted to megapascals (MPa). The enamel surfaces of the specimens were evaluated using a scanning electron microscope (SEM) (JLeo 440, Cambridge, UK) at  $50\times$  and  $1000\times$  magnifications.

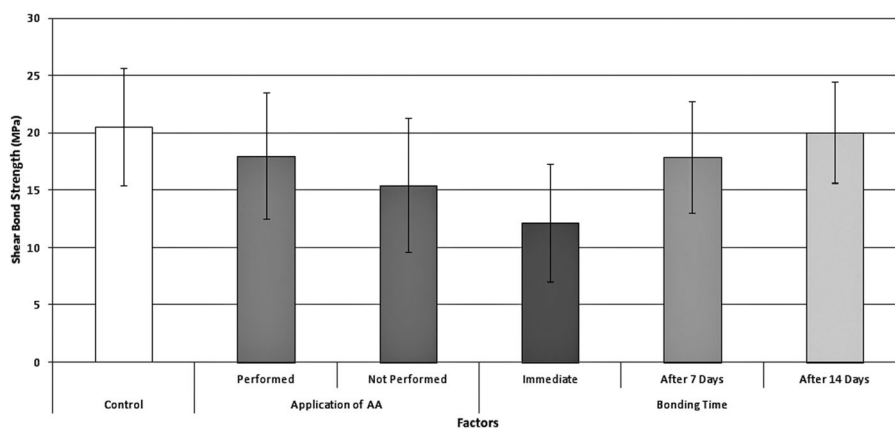


Figure 1 Mean and standard deviations of the factors of the study.

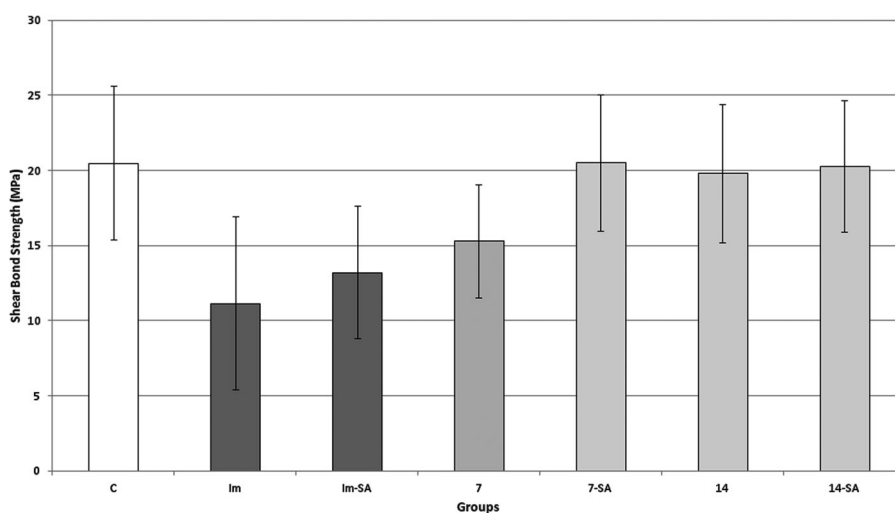


Figure 2 Mean and standard deviations of the groups of the study.

Table 2 Fracture types

Fracture types	Groups						
	C	Im	Im-AA	7	7-AA	14	14-AA
Adhesive	10	10	10	10	9	9	10
Mixed	0	0	0	0	1	1	0
Cohesive	0	0	0	0	0	0	0

**Statistical analysis**

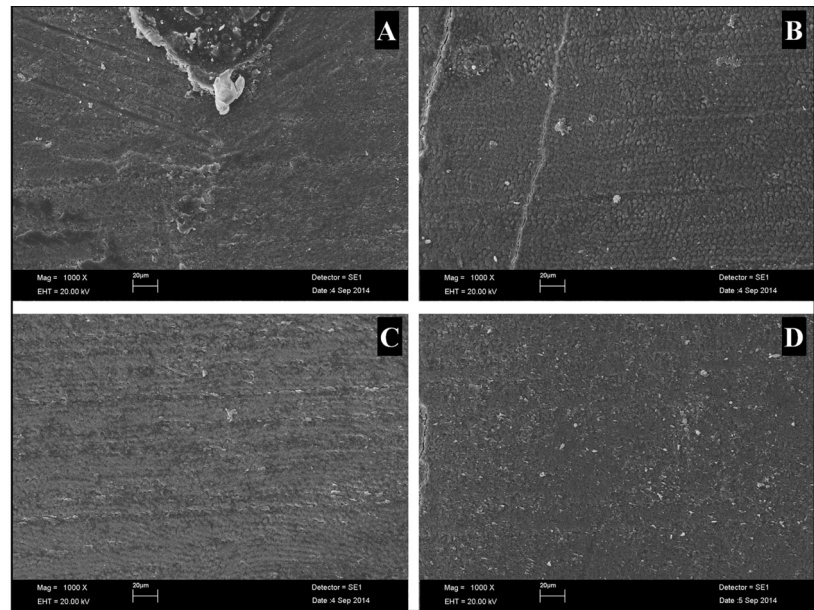
First, data were evaluated with the Kolmogorov-Smirnov normality test ( $\alpha = 0.05$ ). The mean of the normal distributed shear bond strengths of the factors analyzed was computed with two-way ANOVA. Another one-way ANOVA was used to compute the relationships among all groups. The Fisher least significant difference (LSD) test was used as a post hoc test. All analyses

were performed using SPSS 20.0 (IBM Corporation Software Group; New York, NY) at a 5% level of significance.

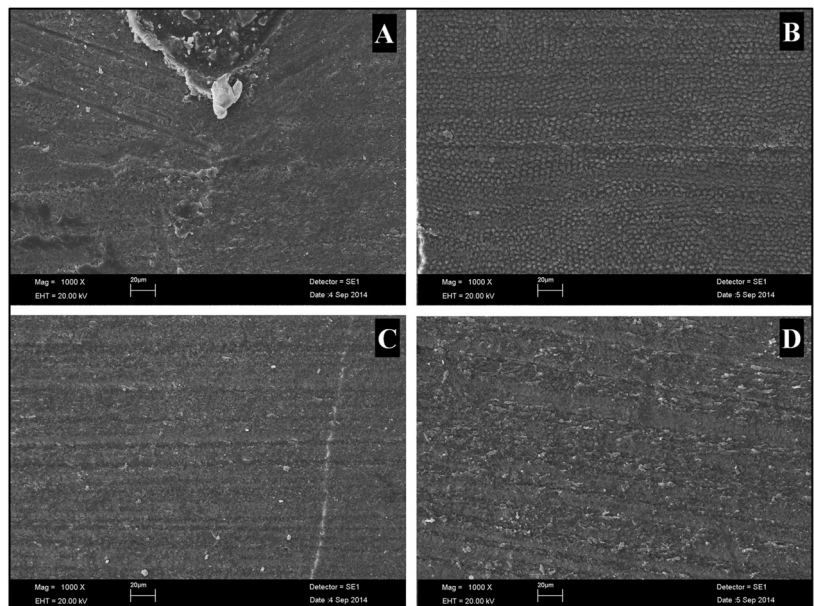
**Results**

According to the results of the two-way ANOVA, different delaying times ( $p < 0.0001$ ) and AA ( $p = 0.023$ ) affected shear bond strength without interaction ( $p = 0.209$ ). Bonding without AA ( $15.43 \pm 5.9$ ) showed lower bonding strengths than with AA and control groups ( $p < 0.05$ ). The shear bond strengths of the control group ( $20.51 \pm 5.1$ ) and the groups bonded with AA ( $18.00 \pm 5.5$ ) did not significantly differ from each other ( $p \geq 0.05$ ) (Fig 1). Moreover, the immediate bonding group ( $12.20 \pm 5.1$ ) showed lower bonding strength values than delayed bonding and the control groups ( $p < 0.05$ ). Bonding after 7 ( $17.91 \pm 4.9$ ) and 14 ( $20.05 \pm 4.4$ ) days did not show significant differences from the control group ( $20.51 \pm 5.1$ ) or from each other ( $p \geq 0.05$ ) (Fig 1).

According to the one-way ANOVA, a statistically significant difference was observed within the groups ( $p < 0.0001$ ).



**Figure 3** SEM pictures after bonding test of the control (A), Im (B), 7 (C), and 14 (D) groups.



**Figure 4** SEM pictures after bonding test of the control (A), Im-AA (B), 7-AA (C), and 14-AA (D) groups.

In the Fisher LSD post hoc ( $\alpha = 0.05$ ) evaluation (Fig 2), group Im ( $11.17 \pm 5.8$ ) showed significantly lower shear bond strength values than all groups except group Im-AA ( $13.23 \pm 4.4$ ). Group Im-AA significantly differed from the other groups except for group 7 ( $15.31 \pm 3.7$ ), which showed significantly lower values than the rest of the groups. The 7-AA ( $20.51 \pm 4.5$ ), 14 ( $19.82 \pm 4.6$ ), 14-AA ( $20.27 \pm 4.4$ ), and C ( $20.51 \pm 5.1$ ) groups did not show significantly different shear bond strengths among each other.

According to SEM observations, there was adhesive breakage in the majority of specimens of in all groups. Almost no mixed or cohesive fracture was observed (Table 2). Therefore, there was no need to perform a statistical interpretation.

## Discussion

Considering the significant dehydration that occurs in endodontically treated teeth, office bleaching often produces only short-term success. Internal bleaching is an uncomplicated and relatively low-risk intervention for improving the esthetic appearance.<sup>2</sup> Whichever bleaching method is chosen, the results are not always satisfactory for all patients, and alternate or further prosthodontic rehabilitations may be required.<sup>5,17</sup>

Many studies have evaluated the effects of vital bleaching of dentin/enamel or nonvital bleaching of dentin, on bond strength.<sup>7-10,14,16,18,19</sup> However, those studies did not evaluate the effect of the bleaching when it is applied to the pulp cavity

and its effect on enamel bonding. Laminate veneers have been considered a conservative approach because their boundaries are limited in enamel. In such situations, bonding to nonvital bleached enamel is also important for the survival of the restoration. There appears to be no available literature that considers this issue.

According to previous studies, the bleaching process has several important local adverse effects, and perhaps the most important is the changing ratio between the organic and inorganic components of dental hard tissues.<sup>20-22</sup> Breakdowns of the bleaching process, which are known as free radicals,<sup>20,23</sup> or the delayed release of oxygen, affect resin infiltration into etched enamel and dentin or block polymerization of resins, resulting in the reduction of bond strength.<sup>5,24,25</sup> The immediate bond strength values in this study, which are lower than other delayed bonding times, verify this reduction.

This study has shown that these degradations are not permanent, like previous studies.<sup>26</sup> In some studies, different suspension times have been found useful to solve this problem.<sup>2,14,15,27</sup> The results obtained in the present study after postponing the bonding for 14 days are compatible with previous studies. Moreover, according to other studies, the application of antioxidants can be an option.<sup>2,8,9,27</sup> Ascorbic acid has been known as the most popular antioxidant, and its sodium salt reduces oxidative compounds, especially free radicals.<sup>2,8,14,15,27</sup> In the present study, the results of bonding tests and the SEM findings after delaying bonding for 7 days also showed improvement in groups with sodium ascorbate, with less enamel reflection due to lower resin thickness (Figs 3, 4).

Gökce *et al* studied external bleached enamel, and showed that 7 days postponement or the application of sodium ascorbate were effective methods for neutralizing the effects of bleaching.<sup>28</sup> However, in the present study, right after the last appointment of internal bleaching the application of sodium ascorbate did not solve the enamel bonding problem. We believe that this may be due to the characteristics of internal bleaching, as the internal bleaching agents affect dental hard tissues (primarily dentin, then enamel) more than the vital bleaching agent. Internal bleaching with thrice-placed bleaching agent may have caused more delayed oxygen release than vital bleaching, and 10-minute application of the sodium ascorbate was not able to reduce the oxidative compounds in the internal bleached enamel. In another study Hansen *et al*<sup>29</sup> evaluated the effectiveness of the short-term application of 35% sodium ascorbate to counteract the effects of a 7-day 35% hydrogen peroxide bleaching regimen on bond strength. The authors of this study showed that the application of 35% sodium ascorbate in a clinically relevant timeframe was not effective at reversing bleaching effects on bond strength. Hansen *et al*<sup>29</sup> emphasized that bonding procedures should be delayed following tooth bleaching. The SEM observations in the present study also showed this finding. Immediate bonding of the internal bleached group produced enamel surfaces free of resin in large areas, and resin tags are poorly defined, fragmented, or penetrated to less depth than the control group, as in previous studies.<sup>28,30</sup> Also, with immediate bonding, the application of sodium ascorbate did not show effective differentiation with SEM, as with bond strength (Figs 3, 4). On the other hand, the 14-day delay groups did not show any differences in bond strength and SEM observa-

tions, because the enamel condition was reversed, as in the unbleached control group specimen (Figs 3, 4).

The findings of the present study seem to have important clinical relevance for internally bleached enamel bonding; however, prospective clinical studies and further *in vitro* studies using mechanical fatigue tests must be conducted to support this clinical argument. Moreover, additional studies could evaluate the effects of the application of sodium ascorbate to internal bleached enamel for durations greater than 10 minutes.

## Conclusions

Within the limitations of this study, the following conclusions were drawn:

1. Immediate bonding on the internal bleached enamel is not recommended, even when 10% sodium ascorbate is used as an antioxidant.
2. Postponing bonding for 7 days is not sufficient to reverse the effects of nonvital bleaching. In such situations, either the application of 10% sodium ascorbate or further postponement to 14 days might be reasonable solutions for reversing the effects of internal bleaching.

## References

1. Lutz F: State of the art of tooth-colored restoratives. *Oper Dent* 1996;21:237-248
2. Zimmerli B, Jeger F, Lussi A: Bleaching of nonvital teeth. A clinically relevant literature review. *Schweiz Monatsschr Zahnmed* 2010;120:306-320
3. Dahl JE, Pallesen U: Tooth bleaching—a critical review of the biological aspects. *Crit Rev Oral Biol Med* 2003;14:292-304
4. Christensen GJ: Bleaching teeth: practitioner trends. *J Am Dent Assoc* 1997;128(Suppl):16S-18S
5. Josey AL, Meyers IA, Romaniuk K, *et al*: The effect of a vital bleaching technique on enamel surface morphology and the bonding of composite resin to enamel. *J Oral Rehabil* 1996;23:244-250
6. Spyrides GM, Perdigao J, Pagani C, *et al*: Effect of whitening agents on dentin bonding. *J Esthet Dent* 2000;12:264-270
7. Wilson D, Xu C, Hong L, *et al*: Effects of different preparation procedures during tooth whitening on enamel bonding. *J Mater Sci Mater Med* 2009;20:1001-1007
8. Uysal T, Ertas H, Sagsen B, *et al*: Can intra-coronally bleached teeth be bonded safely after antioxidant treatment? *Dent Mater J* 2010;29:47-52
9. Turkun M, Celik EU, Kaya AD, *et al*: Can the hydrogel form of sodium ascorbate be used to reverse compromised bond strength after bleaching? *J Adhes Dent* 2009;11:35-40
10. Lai SC, Tay FR, Cheung GS, *et al*: Reversal of compromised bonding in bleached enamel. *J Dent Res* 2002;81:477-481
11. Barghi N, Godwin JM: Reducing the adverse effect of bleaching on composite-enamel bond. *J Esthet Dent* 1994;6:157-161
12. Sung EC, Chan SM, Mito R, *et al*: Effect of carbamide peroxide bleaching on the shear bond strength of composite to dental bonding agent enhanced enamel. *J Prosthet Dent* 1999;82:595-599
13. Cvitko E, Denehy GE, Swift EJ, Jr., *et al*: Bond strength of composite resin to enamel bleached with carbamide peroxide. *J Esthet Dent* 1991;3:100-102
14. Unlu N, Cobankara FK, Ozer F: Effect of elapsed time following bleaching on the shear bond strength of composite resin to

- enamel. *J Biomed Mater Res B Appl Biomater* 2008;84:363-368
15. Cavalli V, Reis AF, Giannini M, et al: The effect of elapsed time following bleaching on enamel bond strength of resin composite. *Oper Dent* 2001;26:597-602
  16. Kimyai S, Valizadeh H: The effect of hydrogel and solution of sodium ascorbate on bond strength in bleached enamel. *Oper Dent* 2006;31:496-499
  17. Turkun M, Kaya AD: Effect of 10% sodium ascorbate on the shear bond strength of composite resin to bleached bovine enamel. *J Oral Rehabil* 2004;31:1184-1191
  18. Lai SC, Mak YF, Cheung GS, et al: Reversal of compromised bonding to oxidized etched dentin. *J Dent Res* 2001;80:1919-1924
  19. May LG, Salvia AC, Souza RO, et al: Effect of sodium ascorbate and the time lapse before cementation after internal bleaching on bond strength between dentin and ceramic. *J Prosthodont* 2010;19:374-380
  20. Haywood VB: History, safety, and effectiveness of current bleaching techniques and applications of the nightguard vital bleaching technique. *Quintessence Int* 1992;23:471-488
  21. Tong LS, Pang MK, Mok NY, et al: The effects of etching, micro-abrasion, and bleaching on surface enamel. *J Dent Res* 1993;72:67-71
  22. Attin T, Kielbassa AM, Schwanenberg M, et al: Effect of fluoride treatment on remineralization of bleached enamel. *J Oral Rehabil* 1997;24:282-286
  23. Goldstein RE, Garber DA, Goldstein CE, et al: Esthetic update: the changing esthetic dental practice. *J Am Dent Assoc* 1994;125:1447-1456
  24. Torneck CD, Titley KC, Smith DC, et al: Adhesion of light-cured composite resin to bleached and unbleached bovine dentin. *Endod Dent Traumatol* 1990;6:97-103
  25. Perdigao J, Francci C, Swift EJ, Jr., et al: Ultra-morphological study of the interaction of dental adhesives with carbamide peroxide-bleached enamel. *Am J Dent* 1998;11:291-301
  26. Lai S, Tay FR, Mak YF, et al: Reversal of compromised bonding in carbamide peroxide-bleached enamel. *J Dent Res* 2002;81:A223-A223
  27. May LG, Salvia ACR, Souza ROA, et al: Effect of sodium ascorbate and the time lapse before cementation after internal bleaching on bond strength between dentin and ceramic. *J Prosthodont* 2010;19:374-380
  28. Gokce B, Comlekoglu ME, Ozpinar B, et al: Effect of antioxidant treatment on bond strength of a luting resin to bleached enamel. *J Dent Res* 2008;36:780-785
  29. Hansen JR, Frick KJ, Walker MP: Effect of 35% sodium ascorbate treatment on microtensile bond strength after nonvital bleaching. *J Endod* 2014;40:1668-1670
  30. Titley KC, Torneck CD, Ruse ND: The effect of carbamide-peroxide gel on the shear bond strength of a microfil resin to bovine enamel. *J Dent Res* 1992;71:20-24