

# Can the Hydrogel Form of Sodium Ascorbate Be Used to Reverse Compromised Bond Strength After Bleaching?

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**Purpose:** To assess the effect of the hydrogel form of different concentrations (2.5%, 5%, and 10%) of sodium ascorbate on the shear bond strength of composite after bleaching of the enamel with 10% carbamide peroxide gel.

**Materials and Methods:** Sixty flat buccal enamel surfaces obtained from 30 bovine incisors were divided into 6 treatment groups: group I, control (nonbleached); group II, no antioxidant treatment after bleaching; group III, 10% sodium ascorbate solution after bleaching; group IV, 2.5% sodium ascorbate hydrogel after bleaching; group V, 5% sodium ascorbate hydrogel after bleaching; group VI, 10% sodium ascorbate hydrogel after bleaching. The specimens were bonded with Clearfil SE Bond, then thermocycled and subjected to the shear test until failure. Fracture analysis of the bonded enamel surfaces was examined using a stereomicroscope. Statistical analysis was carried out using Kruskal-Wallis and the Mann-Whitney U-test.

**Results:** While the samples that were not treated with antioxidant after bleaching (group I) demonstrated significantly lower shear bond strengths and the 10% sodium ascorbate gel group (group VI) demonstrated significantly higher bond strengths than the control group ( $p < 0.05$ ), no significant differences were found between the other groups and control group ( $p > 0.05$ ). Among the antioxidant groups, only the groups treated with the 10% solution and the 10% hydrogel form of sodium ascorbate (group III and VI) revealed significantly higher bond strengths than the bleached group without antioxidant (group II) ( $p < 0.05$ ). Higher scores were obtained with 10% sodium ascorbate gel (group VI) when compared with the other antioxidant-treated groups ( $p < 0.05$ ).

**Conclusion:** Within the limitations of this study, it can be concluded that the 10% hydrogel form of sodium ascorbate may be used in clinical procedures instead of its solution form. However, using sodium ascorbate hydrogel with concentrations lower than 10% may not be as reliable as using this agent in 10% concentration for reversing the compromised bond strength.

**Keywords:** sodium ascorbate hydrogel, antioxidant, bleaching.

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Vital tooth bleaching procedures are generally used in esthetic dentistry to treat discolored teeth.<sup>24</sup> Bleaching

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agents for in-office use contain high concentrations of carbamide peroxide (35% to 37%) or hydrogen peroxide (30% to 35%), while at-home whiteners consist of low concentrations of both peroxides and are employed in a custom tray under the supervision of a dentist.<sup>13,20</sup> As vital tooth bleaching has become increasingly popular, clinicians must become familiar with the products to provide optimal results and know their interactions with other dental treatments, especially the more common ones, such as adhesive restorations. Previous studies have shown that in-office or at-home whiteners adversely affect the bond strength of composite to the acid-etched enamel when bonding is performed immediately after the bleaching process.<sup>10,18,25,33,37,38</sup> It is reported that this situation is related to the presence of residual peroxide, which interferes with the resin attachment and

inhibits resin polymerization.<sup>37</sup> Some techniques have been suggested to solve the clinical problems related to the post-bleaching compromised bond strength. Cvitko et al<sup>6</sup> proposed removal of the superficial layer of enamel, Barghi and Godwin<sup>2</sup> treated the bleached enamel with alcohol before restoration, while Kalili et al<sup>15</sup> and Sung et al<sup>34</sup> suggested the use of adhesives containing organic solvents. However, the general approach is to postpone any bonding procedure for a time after bleaching, since the reduction of bond strength has been shown to be temporary.<sup>39</sup> The waiting period for bonding procedures after bleaching has been reported to vary from 24 h to 4 weeks.<sup>5,7,37,41,42</sup>

Reduced bond strength to the bleached enamel can be reversed with 10% sodium ascorbate solution before resin bonding. Lai et al<sup>19</sup> found that when sodium ascorbate was applied for 3 h to enamel after bleaching with carbamide peroxide, the reduced shear bond strength was reversed. Türkün and Kaya<sup>40</sup> shortened the application period of sodium ascorbate to 10 min and demonstrated that even a 10-min application was enough for reversing the reduced bond strength.

Sodium ascorbate solution is a neutral, biocompatible antioxidant. In clinical conditions, applying the solution form of this agent is difficult because of its flowability. Especially if more than one-tooth applications are required, using the hydrogel form of this agent with a tray may be easier and more comfortable for the patient and the clinician.

The aim of our study was to assess the effect of the hydrogel form of different concentrations (2.5%, 5% and 10%) of sodium ascorbate on the shear bond strength of composite after bleaching the enamel with 10% carbamide peroxide gel.

## MATERIALS AND METHODS

Thirty bovine incisors were used in this study. Immediately after extraction, the teeth were scraped clean of any residual tissue tags, pumiced, and washed under running tap water. The teeth were stored in distilled water at +4°C until required, a period not exceeding 1 week. The roots were removed at the cemento-enamel junction using a slow-speed diamond saw under copious water spray. The incisal part of each bovine tooth yielded 2 samples approximately 4 x 5 mm in size; hence, 60 enamel samples were obtained from all teeth using a cylindrical diamond bur and water spray cooling. The samples were mounted with the buccal surfaces upward in a plastic holder with a cold-curing acrylic resin. The buccal surfaces of samples were flush with the embedding medium to form one flat surface. The central portion of the embedded sample was ground flat with 600-grit silicon carbide paper on a water-irrigated metallurgical polishing wheel.

The 60 specimens were randomly assigned to 6 groups:

- Group I: control (nonbleached)
- Group II: no antioxidant treatment after bleaching
- Group III: 10% sodium ascorbate solution after bleaching

- Group IV: 2.5% sodium ascorbate hydrogel after bleaching
- Group V: 5% sodium ascorbate hydrogel after bleaching
- Group VI: 10% sodium ascorbate hydrogel after bleaching

### Bleaching Procedure

In all bleaching groups, a carbamide peroxide bleaching gel with a concentration of 10% (Opalescence, Ultradent; South Jordan, UT, USA; batch no: B2TTV) was applied to the enamel surfaces for 8 h per day for 7 days according to the manufacturer's instructions. The specimens were partially immersed in artificial saliva at 37°C in a glass laboratory dish, so that only the enamel surfaces coated with the bleaching gel did not contact saliva. At the end of the daily bleaching procedure, specimens were thoroughly rinsed with an air/water spray for 30 s and air dried. Specimens were stored in 250 ml of artificial saliva for the remaining hours of the day.

### Preparation and Application of the Antioxidant

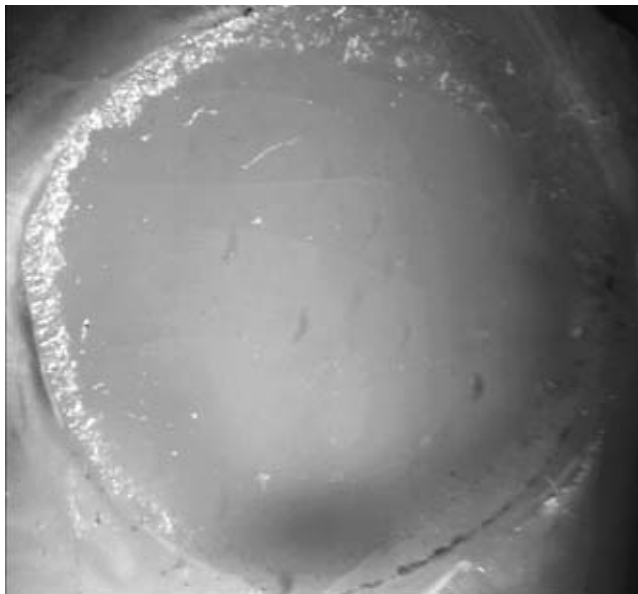
The sodium ascorbate gel formulation was prepared by using Carbopol 974P (Noveon; Cleveland, OH, USA; batch no: CC37MAB845) as a polymer. A certain amount of 0.5% Carbopol 974P was dispersed in water by stirring, ascorbic acid (Hebei Welcome Pharmaceutical; Shijiazhuang, China; batch no: 0206279) was added and dissolved, and sodium hydroxide as a neutralizer was added to thicken the gel. Gels were poured off into small bottles and purged with nitrogen to remove the oxygen. The final pH of the solution and the gels was 7.4.

Ten ml of sodium ascorbate solution was applied onto the surface of the each enamel sample for 10 min. Each minute, 1 ml of solution was dropped onto the sample with a syringe in order to keep the enamel surface wet.

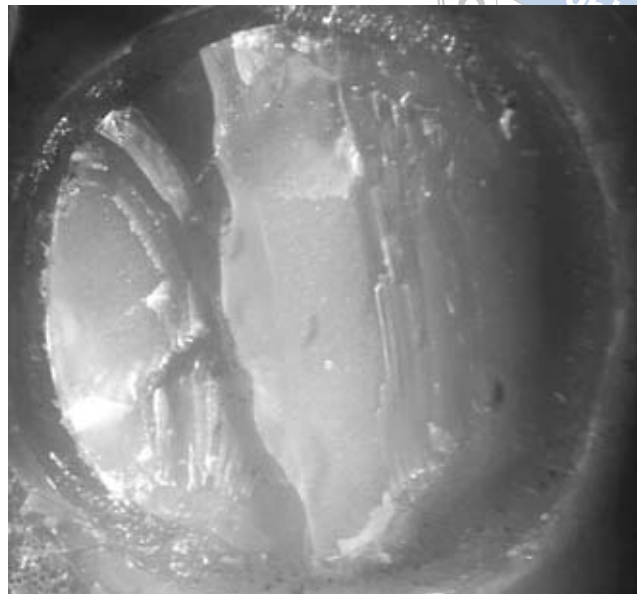
A pilot study in which 10% sodium ascorbate hydrogel was applied onto the bleached samples for 10 min, 1 h, 2 h, 4 h, and 8 h was performed in order to determine the application period of sodium ascorbate hydrogel. In this pilot study, the application of sodium ascorbate hydrogel for more than 2 h did not significantly increase the shear bond strength values; thus, in the present study, sodium ascorbate hydrogel was applied for 2 h onto the samples. After the antioxidant treatment, enamel surfaces were thoroughly rinsed with distilled water for 30 s.

### Bonding Procedure

A piece of adhesive tape with a 2-mm-diameter hole was securely adapted to the center of the flattened portion of the labial enamel to delimit the bonding surface. The self-etching adhesive system Clearfil SE Bond (Kuraray; Osaka, Japan) was applied to the delimited surface according to the manufacturer's instructions. One drop of the acidic primer (batch no: 00510A) was applied to the dentin surface for 20 s and gently air dried. A layer of bonding resin (batch no: 00716A) was applied with a brush, spread gently with air, and cured for 10 s using an Optilux 501 (Kerr/Demetron; Danbury, CT, USA) visible light-curing unit with an intensity output in excess of 450 mW/cm<sup>2</sup>. A split Teflon mold with a circular hole, 2 mm in diameter and 4



**Fig 1** The stereomicroscopic view of an adhesive failure (magnification: 50X).



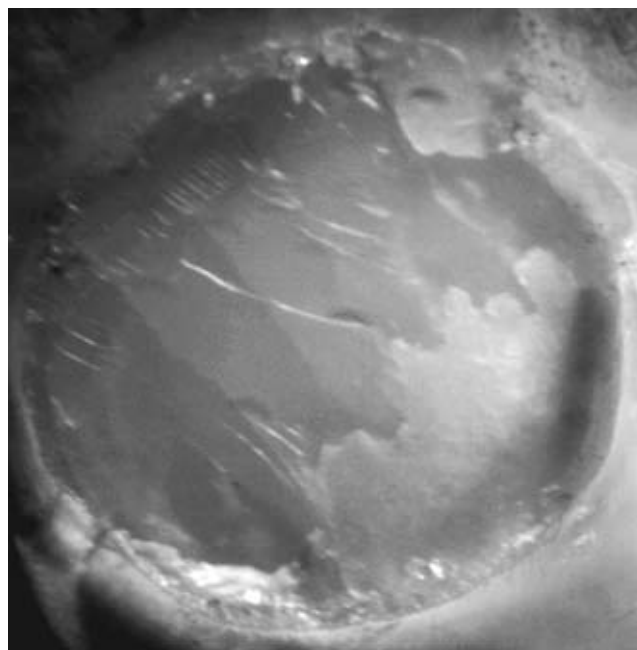
**Fig 2** The stereomicroscopic view of a cohesive failure (magnification: 50X).

mm in depth, was positioned over the hole in the adhesive tape, and was clamped into the place with a special bonding alignment apparatus. A composite resin (Clearfil AP-X, Kuraray; batch no: 00921B) was incrementally placed and cured in the mold, forming cylindrical posts perpendicular to the dentin surface. Each specimen was totally cured for 80 s. After removal from the bonding alignment apparatus and the split mold, the specimens were stored in distilled water at 37°C for 24 h. They were then subjected to 1000 thermal cycles between baths of 5°C and 55°C, with a dwell time of 30 s.

The shear bond strength was measured with a Shimadzu Universal Testing Machine (Model AG-50kNG; Shimadzu; Kyoto, Japan). A knife-edge shearing rod and a crosshead speed of 0.5 mm/min were used. The distance from the probe to the dentin surface was monitored using a spacer of two celluloid matrices. The load at failure was recorded by Labtech Notebook software version 6.3 (Omega Engineering; Stamford, CT, USA). The shear bond strengths of the specimens were calculated and expressed in MPa.

Fracture analysis of the bonded enamel surface was performed using a stereomicroscope at 50X magnification. Failures were classified as adhesive (> 75% of failure between tooth and restorative material) (Fig 1), cohesive (> 75% of the failure was within the restorative material) (Fig 2), or a mixture of the two (Fig 3).

Statistical analysis was carried out with the SPSS 13.0 software system (SPSS; Chicago, IL, USA). The shear bond strength data of the groups were statistically analyzed with the Kruskal-Wallis and Mann-Whitney U- tests. The fracture modes of the samples were compared by the chi-square test. The level of significance was determined as  $p = 0.05$  for all tests.



**Fig 3** The stereomicroscopic view of a mixed failure (magnification: 50X).

## RESULTS

Shear bond strengths in MPa (mean  $\pm$  SD), median, minimum/maximum, and interquartile range for the groups are shown in Table 1. Kruskal-Wallis analysis revealed signifi-

**Table 1 Shear bond strengths in MPa (mean  $\pm$  SD), median, min-max, and interquartile range of the groups**

Group	Mean $\pm$ SD	Median	Min-Max	Interquartile range
1	24.77 $\pm$ 3.81	23.37 <sup>a,b</sup>	20.15-32.72	5.12
2	17.89 $\pm$ 2.81	18.03 <sup>c</sup>	14.18-22.98	4.49
3	27.14 $\pm$ 8.59	26.23 <sup>a,b</sup>	14.34-44.86	11.16
4	21.21 $\pm$ 7.07	20.7 <sup>a,c</sup>	8.05-32.86	7.48
5	21.03 $\pm$ 8.28	22.6 <sup>a,c</sup>	9.13-35.23	13.63
6	33.3 $\pm$ 4.96	34.74 <sup>d</sup>	24.36-40.3	7.25

Different letters denote significant differences between the study groups ( $p < 0.05$ ).

**Table 2 The fracture modes of the tested groups**

Group	Adhesive	Mixed	Cohesive
1	4 (40%)	5 (50%)	1 (10%)
2	5 (50%)	4 (40%)	1 (10%)
3	3 (30%)	6 (60%)	1 (10%)
4	5 (50%)	5 (50%)	0
5	2 (20%)	5 (50%)	3 (30%)
6	2 (20%)	6 (60%)	2 (20%)

cant differences in bond strengths of the 6 groups ( $p = 0.005$ ).

While the samples that were not treated with antioxidant after bleaching (group II) demonstrated significantly lower shear bond strengths and the 10% sodium ascorbate gel group (VI) demonstrated significantly higher bond strengths than control group samples ( $p < 0.05$ ), no significant differences were found among the other groups and control group ( $p > 0.05$ ). Among the antioxidant groups, only the 10% solution and 10% hydrogel treated groups (III and VI) revealed significantly higher bond strengths than the no-antioxidant bleaching group (II) ( $p < 0.05$ ). Higher scores were obtained with group VI (10% sodium ascorbate gel) when compared with the other antioxidant treated groups ( $p < 0.05$ ).

The fracture modes of groups are shown in Table 2. In each group, nearly half of the samples demonstrated the mixed fracture mode. The number of adhesive failures was high in the no-antioxidant bleaching group, which revealed the lowest mean bond strength value. In contrast, the number of adhesive failures was low in the 10% sodium ascorbate gel group, which had the highest bond strength. However, no significant differences were found among the study groups in terms of fracture modes ( $p > 0.05$ ).

## DISCUSSION

Despite its different characteristics from human enamel, bovine enamel has been used in a number of studies to compare the bond strength of a restorative material to bleached and unbleached enamel surfaces, due to the difficulties in obtaining large numbers of human teeth.<sup>22,39</sup> Bovine enamel is more porous than human enamel; furthermore, it has thinner crystallites and a different structure between prisms.<sup>8,9</sup> Sydney-Zax et al<sup>35</sup> reported that the carbonate concentration in bovine enamel was higher than in human enamel. Enamel rich in carbonate has been reported to be more vulnerable to acid attack.<sup>44</sup> Lopes et al<sup>22</sup> evaluated the shear bond strength of etch-and-rinse and self-etching systems to human and bovine enamel. They concluded that both etch-and-rinse and self-etching systems performed equivalently on bovine and human enamel in terms of shear bond strength. In addition, the results of the studies performed using human teeth generally supported the results and observations recorded for bovine teeth.<sup>38,39</sup> In the view of these studies, bovine enamel samples were used in this study, as a large number of human enamel samples was difficult to obtain.

Conventional dentin bonding systems are usually composed of a three-step bonding procedure, i.e., conditioning, priming, and bonding. Two-step systems, which simplify the bonding procedure and lead to lower technique sensitivity, have also been developed. One of the two-step systems (Clearfil SE Bond) is a self-etching bonding system, which combines the etching and priming procedures into one step by a self-etching primer.<sup>28</sup> An acidic monomer in the primer can demineralize the smear layer and underlying dentin, resulting in mild etching. Although there are some studies reporting the lower demineralization ability of self-etching vs etch-and-rinse systems,<sup>11,43</sup> other authors have found similar shear bond strengths with self-etching and etch-and-rinse adhesives.<sup>3,21</sup> Moura et al<sup>27</sup> recommended the use of Clearfil SE Bond among the self-etching adhesive systems due to its superior bond strength and more retentive etching pattern. Satisfactory results were also obtained from studies which examined

the clinical performance of this adhesive.<sup>1,23,29</sup> Based on this and considering the popularity of self-etching bonding systems, Clearfil SE Bond was used in this study.<sup>26,30,40</sup>

Since many patients require the replacement of esthetic restorations immediately after bleaching, the initial reduction in enamel bond strength after bleaching is clinically very important. Studies have shown that the bond strength to enamel decreases after bleaching with carbamide peroxide in various concentrations.<sup>5,6,10,25,33,34,36</sup> Reduction in bond strength due to bleaching has been attributed to the residual oxygen which is released from the bleaching agent, and interferes with the resin infiltration into the etched enamel or inhibits the polymerization of resin.<sup>7,36,37</sup> Titley et al<sup>37</sup> reported that in the SEM evaluation of bleached specimens, large areas of the enamel surface were free of resin, and, when tags were present, they were fragmented, poorly defined, and penetrated to a lesser depth when compared with the unbleached controls. In another study, Titley et al<sup>36</sup> discovered a granular, porous aspect with a bubbly appearance at the interfaces between resin and bleached enamel with SEM examination. This coarse, porous appearance of the interfaces were postulated to be gaseous bubbling from oxidizing reactions, possibly resulting from retained peroxide in the subsurface layer of the enamel.<sup>36,40</sup> Similar to these studies, the findings of the present study also revealed that the shear bond strength of composite resin to the bleached enamel was significantly lower than that in the control group.

Although many methods have been used to reverse the compromised bond strength, in this study, emphasis was placed on neutralizing the oxygen by application of an antioxidant. Ascorbic acid and its sodium salt are well-known antioxidants with the ability to reduce oxidative compounds, especially free radicals.<sup>4,12,31</sup> Lai et al<sup>19</sup> immersed the bleached specimens in 10% sodium ascorbate solution for 3 h. In more recent studies, 10 min of antioxidant treatment was tested.<sup>16,40</sup> In all studies, sodium ascorbate treatment appeared to restore the reduced bond strength of composite to the bleached enamel samples. Sodium ascorbate allows free-radical polymerization of the adhesive resin to proceed without premature termination by restoring the altered redox potential of the oxidized bonding substrate, thus reversing the compromised bonding.<sup>18</sup> Our results concur with those of recent studies, which have shown that reduced bond strength was effectively reversed by antioxidant treatment.<sup>16,18,19,40</sup>

Application of the solution form of sodium ascorbate to the bleached tooth takes a long time, which may make it clinically unsuitable. However, if the sodium ascorbate is produced in the gel form, patients themselves can apply this gel with a bleaching tray before restoration treatment. Hydrogel is a network of polymer chains that are water insoluble, sometimes found as a colloidal gel in which water is the dispersion medium. They are superabsorbent (they can contain over 99% water) natural or synthetic polymers.<sup>14</sup> In our study, 10% sodium ascorbate gel applied for 2 h reversed the reduced bond strength. However, we found that concentrations lower than 10% were insuffi-

cient to reverse the compromised bond strength. In agreement with our findings, Kimyai and Valizadeh<sup>17</sup> reported that 10% and 20% sodium ascorbate gels were effective in reversing the compromised bond strength due to bleaching when they were applied for 3 h.

At the beginning of this study, it was expected that the hydrogel might not be as effective as the solution form, as it has lower diffusibility into the enamel. Thus, hydrogel forms of sodium ascorbate were used for a longer period than the solution form. The application time of the hydrogel was determined based on results of a pilot study in which this gel formulation was applied for 10 min, 1 h, 2 h, 4 h, and 8 h. In this pilot study, application of sodium ascorbate hydrogel for more than 2 h did not significantly increase the shear bond strength values; thus, in the present study, sodium ascorbate hydrogel was applied for 2 h onto the samples. In the current study, bond strengths after treatment with 10% sodium ascorbate hydrogel were superior to those after 10% sodium ascorbate solution application. This variation may be attributed to the differences of time of application between the solution (10 min) and of the gel (2 h), while their concentrations were the same.

In the present study, the 10% sodium ascorbate gel group demonstrated higher bond strengths than the control samples. Research has shown that for the monomer systems used and curing conditions encountered in dentistry, oxygen acts as an inhibitor of resin composite polymerization.<sup>32,45</sup> The enamel surface may include oxygen as it is in contact with room air. In addition, it has organic molecules and water including oxygen. In this way, the oxygen on the enamel surface might also inhibit polymerization at the enamel/resin interface in the unbleached samples. Bearing this in mind, the higher bond strength with 10% sodium ascorbate than in control samples might be attributed to its higher neutralization capacity of oxygen, which stems not only from the bleaching agents but also from the atmosphere or the nature of the enamel.

## CONCLUSION

Within the limitations of this study, it can be concluded that the 10% hydrogel form of sodium ascorbate may replace the solution form in clinical procedures. However, using sodium ascorbate hydrogel with concentrations lower than 10% for 2 h may not be as reliable as using this agent in 10% concentration for reversing the compromised bond strength. For the concentrations under 10%, further studies could examine increasing the application period of this gel in order to reverse the decreased bond strength.

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**Clinical relevance:** The 10% hydrogel form of sodium ascorbate effectively reversed the compromised bond strength after bleaching. Thus, this agent can be used instead of its solution form when the clinical conditions are unsuitable for the application of the solution form of sodium ascorbate.

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