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Tensile bond strength of brackets after antioxidant treatment on bleached teeth

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SUMMARY Various studies have reported a significant reduction in tensile bond strength of brackets when bonding is carried out immediately after bleaching. The purpose of this investigation was to determine the effect of an antioxidant agent on the tensile bond strength values of metal brackets bonded with composite resin to human enamel after bleaching with carbamide peroxide (CP).

A total of 80 extracted premolar teeth were randomly divided into three bleaching groups of 10 per cent CP and an unbleached control group. The specimens in group 1 were bonded immediately after bleaching; group 2 were stored in an artificial saliva solution for 7 days after bleaching; group 3 were treated with 10 per cent sodium ascorbate, immediately before bonding, whereas the unbleached specimens in group 4 had no treatment before bonding. Tensile bond strengths were established in MPa. To evaluate the amount of resin left on the enamel surfaces after debonding, the adhesive remnant index (ARI) scores were used. The tensile bond strength data were analyzed with the Kruskal–Wallis test and pairwise comparisons were made by the Mann–Whitney U test at a significance level of $P < 0.05$.

The brackets bonded immediately after bleaching revealed significantly lower tensile bond strengths than those of unbleached enamel ($P = 0.000$). No statistically significant differences in tensile bond strength were noted when the delayed-bonding ($P = 6.000$) and antioxidant-treated ($P = 0.2757$) groups were compared with the control group. The antioxidant treatment immediately after bleaching was effective in reversing the tensile bond strength of brackets.

Introduction

Vital tooth bleaching is a safe and well-accepted procedure for the treatment of surface and intrinsic staining of teeth (Matis *et al.*, 1998). At-home whitening systems are effective for treating the superficial enamel layers, such as food staining, mild uniform yellow, orange, or light brown discolourations, and also for very mild cases of tetracycling staining, fluorosis or enamel mottling (Bishara *et al.*, 1993). The first report on a patient-applied at-home bleaching system using carbamide peroxide (CP) was reported by Haywood and Heymann (1989). Since then, various whitening systems have been developed and peroxide compounds at different concentrations are currently being used to bleach enamel (Larson, 1990; Cavalli *et al.*, 2004).

Bleaching of discoloured teeth can be performed before or after fixed orthodontic appliance treatment. An *in vitro* study on clinical colour differences when bleaching was applied after debonding of brackets was carried out by Hintz *et al.* (2001). It required at least two to four weeks of continuous bleaching to achieve a significant difference. Conversely, when patients have previously had their teeth bleached, they often become aware of orthodontic problems and want to be treated. Nevertheless, numerous studies have revealed that if an in-office or an at-home system is used prior to adhesive restorations or before application of resin bonded fixed appliances, the bonding strength to tooth structures is significantly reduced (Titley *et al.*, 1991; Stokes *et al.*, 1992; Garcia-Godoy *et al.*, 1993; Toko and Hisamitsu, 1993; Miles *et al.*, 1994; Ben-Amar *et al.*, 1995; Lai *et al.*, 2002). To eliminate clinical problems related to post-bleached compromised bond strength, some techniques have been suggested. Cvitko *et al.* (1991) proposed the removal of a superficial layer of enamel, Barghi and Godwin (1994) pre-treated the bleached enamel with alcohol, while Kalili *et al.* (1991) and Sung *et al.* (1999) advised the use of adhesives containing organic solvents. However, the general suggestion is to reschedule any bonding procedure after the last bleaching session, since the reduction of composite resin bond strength to freshly bleached enamel has been shown to be temporary (Torneck *et al.*, 1991; McGuckin *et al.*, 1992; Miles *et al.*, 1994). The waiting period for bonding procedures after bleaching has been reported to vary from 24 hours to four weeks (Titley *et al.*, 1992; Dishman *et al.*, 1994; Miles *et al.*, 1994; van der Vyder *et al.*, 1997; Cavalli *et al.*, 2001; Uysal *et al.*, 2003).

Compromised bonding to bleached enamel being due to inhibition of polymerization of resin-based materials is attributed to residual oxygen. In a recent *in vitro* study on extracted and sandblasted human third molars, Lai *et al*. (2002) found that when sodium ascorbate, an antioxidant, was applied for 3 hours to enamel after bleaching with CP, the shear bond strength of the composite was reversed.

CP used in bleaching treatments is a biological oxidant. CP actualizes the bleaching process by oxidizing the macromolecules of stains quickly and breaks them into smaller fragments, consequently diffusing them across dental surfaces (Goldstein and Kiremidjian-Schumacher, 1993; Rose and Bode, 1993). If the bond strength decreases on enamel treated with CP as a result of the oxidizing action, it may be reversed by applying a biocompatible and neutral antioxidant such as sodium ascorbate before applying the resin composite.

The aim of this study was to determine whether the tensile bond strength of standard metal orthodontic brackets, immediately bonded to CP-bleached enamel tissues increases after application of an antioxidant agent.

Materials and methods

Preparation of specimens

Eighty sound human maxillary and mandibular premolars extracted for orthodontic reasons were collected. All the teeth used in this study were extracted over the course of three months. They had undamaged buccal enamel, no caries and no pre-treatment with any chemicals. Following extraction, residue on the teeth was removed and washed away with tap water. They were then stored in a solution of 0.1 per cent thymol. Before the experiment, the roots of all the teeth were separated using a water-cooled diamond bur. The coronal pulps were removed and again washed with water. The pulp chamber was filled with light body elastomeric impression material (Xantopren VL Plus, Heraus Kulzer Gmbh & Co, KG, Dormagen, Germany) to avoid penetration of the self-curing acrylic monomers into the pulp chamber. The teeth were then embedded in standardized $15 \times 18 \times 29$ mm^3 polyethylene moulds using self-curing resin with the labial surfaces exposed, and stored in distilled water at +4°C until required. Prior to the bleaching procedure, the enamel surfaces were polished with oil- and fluoride-free fine pumice and water using a brush and a slow-speed handpiece, rinsed again, and dried with an air syringe. The method specified for each experimental group was then followed.

Experimental groups

The samples were randomly divided into three bleaching groups of 10 per cent CP ($n = 60$) and a control group ($n = 20$). Group 1 consisted of specimens bonded immediately after bleaching ($n = 20$). The teeth in group 2 were immersed in artificial saliva solution for 7 days after bleaching ($n = 20$), while group 3 specimens were treated with an antioxidant agent, 10 per cent sodium ascorbate, just before bonding ($n = 20$). Specimens in the control group were not bleached ($n = 20$), and were only immersed in artificial saliva solution for 7 days before bonding (Table 1).

Bleaching procedure

In all three bleaching groups, a commercial 10 per cent CP at-home bleaching gel (Rembrandt Xtra-Comfort, Den-Mat, Santa Maria, California, USA) was applied to the enamel surfaces of the embedded teeth for 8 hours in a day according to the manufacturer's instructions. The specimens were partially immersed in an artificial saliva solution at 37°C in a glass laboratory beaker so that only the enamel surfaces coated with the bleaching gel were not in contact with the saliva. After completion of the daily bleaching procedure, the specimens were thoroughly rinsed with a compressed air/water syringe for 30 seconds, air-dried, and stored in 250 ml of artificial saliva solution. The procedure was continued for 7 days.

Artificial saliva immersion

The specimens in group 2 were immersed in 250 ml of artificial saliva solution at 37°C for 7 days immediately after the bleaching process whereas those in group 4 were only immersed in the artificial saliva for 7 days without prior bleaching. The artificial saliva solution had an electrolyte composition similar to that of human saliva. It was composed of 1 g sodium carboxymethylcellulose, 4.3 g xylitol, 0.1 g potassium chloride, 5 mg calcium chloride, 40 mg potassium phosphate, 1 mg potassium thiocyanate and 100 g distilled deionized water. The artificial saliva solution was changed twice daily during the consecutive 7-day time period. After the specimens were removed from the artificial saliva, the enamel surfaces were rinsed with an air/water syringe for 30 seconds and the brackets were bonded.

Application of antioxidant

Group 3 specimens were treated as follows: 10 ml of 10 per cent sodium ascorbate was dripped on the enamel surfaces of the embedded teeth following the bleaching process and agitated with a sterile brush. After 10 minutes, it was washed

Table 1 Study groups and treatment methods before bonding.

Groups	Treatment method
1	10 per cent carbamide peroxide (CP)-bleaching
2	10 per cent CP-bleaching + immersion in artificial saliva solution for 7 days
3	10 per cent CP-bleaching + 10 per cent sodium ascorbate treatment
4	Immersion in artificial saliva solution for 7 days (control)

with distilled water and dried. Thereafter the brackets were bonded.

Bonding of brackets

Stainless steel, standard edgewise, twin brackets with a mesh base (Forestadent, Pforzheim, Germany) were used in this study. O-rings that fit into the slots were soldered to the brackets before bonding. The brackets were bonded to specimens at 24°C room temperature using a chemically cured composite resin (Concise Orthodontic Bonding System, 3M Unitek, CA, USA). For all specimens, etching liquid (1 drop) was applied to the labial surface of the enamel for 30 seconds. Any remaining demineralized tooth particles and etching liquid were removed with an air/water syringe applied for 10 seconds. The teeth were then dried for 10 seconds with oil-free compressed air. Equal amounts (1 drop) of enamel bond resin A and B were mixed for 5 seconds and applied to the etched surfaces in a thin coat. Immediately after, the bonding paste was prepared by placing equal portions of paste A and B, mixed for 15 seconds and applied to the bracket base in sufficient volume that the base was fully covered. The brackets were then seated and positioned firmly in the middle third of the labial enamel surface. Excess resin was removed using a scaler. After bonding, the specimens in all groups were stored in distilled water at 37°C for 24 hours. They were then subjected to 500 thermal cycles in two thermally controlled baths of streaming tap water maintained at 5°C and 55°C, with a dwell time of 30 seconds at each temperature.

Analysis of tensile bond strength

The tensile bond strengths of the samples were measured with a Universal Testing Machine (Lloyd Instruments plc, Fareham, Hampshire, UK). The specimens were securely clamped and aligned towards the shearing hook by means of the movable plateau of a vice (Figure 1). The slot of the bracket was perpendicular to the plunger of the testing machine. Metal o-rings ensured a pure strength force during mechanical loading and prevented deformation of the bracket wings. The crosshead speed of the 500 N load cell (NLC 500 N, Lloyd Instruments plc) was set at 1 mm/minute. The load at failure was recorded and processed using the Dapmat Software System (version 2.31, Hampshire, UK). The data for the applied load were standardized by dividing the force-to-failure with the entire surface area of the bracket base and expressed in MPa (bracket base dimensions were provided by Forestadent).

Location of adhesive failure

A stereo microscope (Olympus Co, Tokyo, Japan) at ×16 magnification was used to analyse the bonded enamel surfaces and bracket bases. The adhesive remnant index (ARI; Årtun and Berglund, 1984) was used to classify failure patterns observed in debonded specimens.

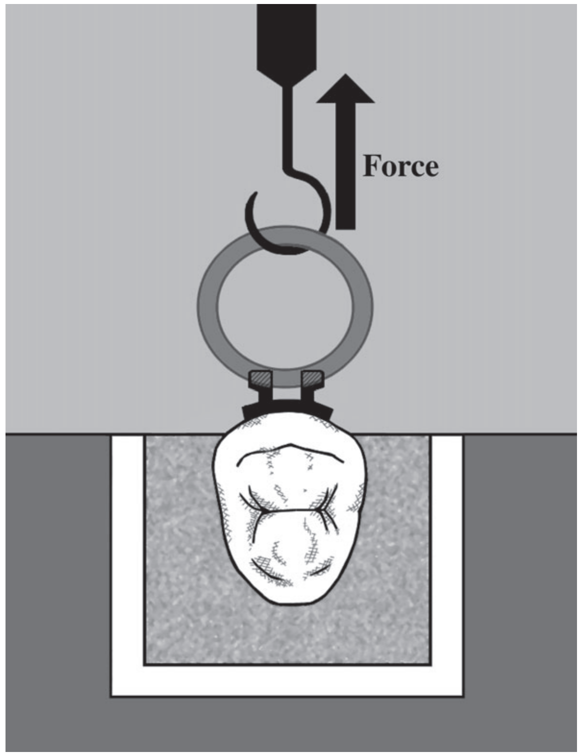

Figure 1 Schematic diagram of the test assembly for determining tensile bond strength.

Statistical analysis

The tensile bond strength data of the groups were subjected to a test of normality. With respect to this, a non-parametric test (Kruskal–Wallis) was used to determine the significance between the groups. The ARI scores were evaluated by means of chi-square analysis. The level of significance was established as $P < 0.05$ for all statistical tests. To determine the differences between the groups, a Mann–Whitney U test with Bonferroni correction was performed. The predetermined level of significance ($P < 0.05$) was reset as $P < 0.0083$ after Bonferroni-correction. Statistical analyses were processed with the SPSS 10.0 software system (SPSS Inc., Chicago, Illinois, USA).

Results

Tensile bond strengths in MPa (Mean ± SD), medians and quartiles for the groups are shown in Table 2. A Kolmogorov–Smirnov test demonstrated a non-normal distribution ($P = 0.023$). The Kruskal–Wallis test showed that there were statistically significant differences in bond strength among the four groups ($P = 0.000$). The tensile bond strength of brackets in group 1 was significantly lower than unbleached enamel ($P = 0.000$). For the bleaching groups, when groups 2 and 3 were compared with the control group, there was no statistically significant difference in tensile bond strength values ($P = 6.000$ and $P = 0.2757$, respectively).

This showed that both antioxidant treatment and artificial saliva immersion were significantly effective in increasing the tensile bond strength of brackets to bleached enamel. When the antioxidant-treated and saliva-immersed groups were compared with each other, no statistically significant difference was observed ($P = 0.2945$).

Examination of the tooth surfaces and bracket bases after failure indicated that resin may adhere either to the bracket bases or to the tooth surfaces. To assess the amount of resin left on the enamel surfaces after debonding, the ARI was used (Table 3). Chi-square analysis of the ARI scores revealed that there was a statistical difference between the groups ($P = 0.0000127$). Enamel tear-out due to mechanical loading was observed in only one specimen in group 4.

Discussion

Generally, adhesive capacity has been evaluated by *in vitro* testing, with shear and tensile tests being the most widely used (Pashley *et al.*, 1995). Testing the bond strength by tensile loading produces more adhesive failures, which may favour the evaluation of the true bond strength (Della Bona and van Noort, 1995).

Home-bleaching that uses 10–22 per cent CP is a procedure applied by the patients. Studies have shown that the bonding strength of enamel decreases after bleaching with CP in various concentrations (Cvitko *et al.*, 1991; Stokes *et al.*, 1992; Titley *et al., 1992;* Garcia-Godoy *et al.*, 1993; Miles *et al.*, 1994; Ben-Amar *et al.*, 1995; Josey *et al.*, 1996; Sung *et al.*, 1999; Cavalli *et al.*, 2001). Turkun and Kaya (2004) investigated the effect of different concentrations of CP on the shear bond strength of resin composite to bleached bovine enamel and demonstrated a significant decrease in shear bond strength caused by 10, 16 and 20 per cent CP. That study also showed the reduction in tensile bond strength of brackets post-bleaching in contrast to a control group.

Several investigations have attempted to clarify the decrease in enamel bond strength caused by CP after bleaching. It has been suggested that weak bonding surfaces and staining susceptibility are related to enamel surface morphology with varying degrees of surface roughness and structural changes by loss of prismatic formation (Ben-Amar *et al.*, 1995; Josey *et al.*, 1996; Cavalli *et al.*, 2004). Further, changes in the organic substance, the loss of calcium, and decrease in microhardness are potential causes of a reduction in tensile bond strength (McCracken and Haywood, 1996; Hegedus *et al.*, 1999). It has also been claimed that residual oxygen, which is released from the bleaching agent, interferes with the resin infiltration into the etched enamel or inhibits the polymerization of resin (Rueggeberg and Margeson, 1990; Kalili *et al.*, 1991; Titley *et al.*, 1991, 1992; Dishman *et al.*, 1994). In a scanning electron microscopic (SEM) study of bovine incisors, Titley *et al.* (1991) found that the appearance of the interfaces between resin and bleached enamel were considerably diverse. Specimens, which were exposed to 35 per cent hydrogen peroxide, exhibited large areas of enamel surface that were free of resin. Dishman *et al.* (1994) applied 25 per cent hydrogen peroxide and observed that the composite bond was compromised through a decrease in the number and poor quality of existing resin tags. Other SEM examinations of interfaces between resin and bleached enamel, showed an

Table 2 Descriptive statistics of tensile bond strengths (MPa) for each group.

Group	*n*	Quartile 1	Median	Quartile 3	Min	Max	Mean	SD
1	20	8.458	9.235	10.733	4.86	13.57	9.43	2.55
2	20	12.788	17.100	17.970	9.57	19.80	15.60	3.35
3	20	13.558	14.870	16.068	12.17	16.77	14.67	1.65
4	20	13.985	16.315	17.588	10.86	19.22	15.94	2.58

Quartile 1: 25 percentile; Median: 50 percentile; Quartile 3: 75 percentile; SD: standard deviation; $P < 0.0083$ (after Bonferroni correction).

Table 3 Comparisons of adhesive remnant index (ARI) of groups in different conditions.

	ARI scores*			
Condition	*n*	1	2	3
Bleached	20	10	6	4
Bleached + artificial saliva immersion	20	0	6	14
Bleached + 10 per cent sodium ascorbate treatment	20	4	6	10
Not bleached (control)	20	0	2	18

*ARI scores: 1 = less than half of the adhesive left on the tooth surface; 2 = half of the adhesive or more left on tooth surface; 3 = all adhesive left on tooth surface, failure between adhesive and bracket base.

association of gap density in adhesion fields with the lowest mean bond strengths (McGuckin *et al.*, 1992; Titley *et al.*, 1992). The coarse and porous appearance of the interfaces postulated to be gaseous bubbling from oxidizing reactions, possibly resulted from retained peroxide in the subsurface layer of the enamel (Turkun and Kaya, 2004).

Previous investigations have demonstrated that immersion of *in vitro* specimens in artificial saliva, distilled water or even saline results in a complete reversal of the reduced enamel bonds (Torneck *et al.*, 1991; Titley *et al.*, 1992; Miles *et al.*, 1994; Josey *et al.*, 1996; Cavalli *et al.*, 2001). The results of the present study are in agreement with those findings, assuming that the immersion process is removing the residual oxygen from the bleaching material. Human saliva is supposed to have similar action after bleaching in the oral environment. The delay period after bleaching required to return the bonding strength to a pre-bleached level is still debated, but the commonly suggested post-bleaching time period is 7 days before bonding (Torneck *et al.*, 1991; McGuckin *et al.*, 1992; Miles *et al.*, 1994; Spyrides *et al.*, 2000). The present investigation confirmed that a period of 7 days after bleaching is sufficient to obtain adequate tensile bond strength for clinical conditions.

The major aim of this study was to re-establish or at least repair the temporary reduction in bond strength by neutralizing the residual oxygen through the application of an antioxidant. The method of antioxidant treatment for bleached teeth before direct bracket bonding would possibly resolve the requirement for waiting a certain period of time, if it succeeded in restoring the tensile bond strength to pre-bleach levels. Ascorbic acid and its sodium salt are potent antioxidants with the capacity to quench reactive free radicals in biological systems (Buettner, 1993; Rose and Bode, 1993; Gutteridge, 1994). Studies regarding peroxide-induced oxidation and related harm in biological structures have revealed a protective effect of ascorbic acid *in vitro* (Smit and Anderson, 1992; Hawkins and Davies, 1999; Carr *et al.*, 2000) and *in vivo* (Brennan *et al.*, 2000). The food industry makes wide use of ascorbic acid as an antioxidant, since it is non-toxic. In the present study, the salt of ascorbic acid, sodium ascorbate, was used to prevent the acidic effect. Sodium ascorbate treatment of the bleached enamel before immediate bonding with composite resin appeared to restore the reduced tensile bond strength of metal brackets. Lai *et al.* (2002) immersed the bleached specimens in 10 per cent sodium ascorbate solution for three hours. In more recent studies (Kaya and Turkun, 2003; Turkun and Kaya, 2004), 10 minutes of antioxidant treatment was found to be effective. This time period was used for antioxidant treatment since it is a beneficial time for clinical conditions. This short treatment period, dispensing and agitating continuously in refreshed sodium ascorbate solution, enhanced its effect on the bleached enamel surface.

An attempt was made to provide an explanation for the structural aberration and reduction of bond strength in the presence of peroxide. It has been shown that hydroxyl radicals in the apatite lattice are substituted by peroxide ions and produce peroxide-apatite (Zhao *et al.*, 2000). When peroxide ions decompose, substituted hydroxyl radicals re-enter the apatite lattice, resulting in the elimination of the structural changes caused by the incorporation of peroxide ions. Lai *et al.* (2002) assumed that the inclusion process of peroxide ions might also be reversed by an antioxidant. In addition, they postulated that 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 (Lai *et al.*, 2001).

Supplementary research to improve the antioxidant agent in different forms is necessary. The effects of different antioxidants on bond strength and structural patterns of bleached enamel should also be studied.

Conclusions

The results obtained in this *in vitro* study demonstrate that bleaching of enamel with 10 per cent CP immediately before bonding results in a reduction of bracket tensile bond strength. A period of 7 days in artificial saliva solution after bleaching provides re-establishment of inadequate tensile bond strength. In the samples where antioxidant was applied for 10 minutes immediately after bleaching, tensile bond strength was found to be at the same level as in those samples kept in artificial saliva solution for 7 days. Treating the bleached enamel surface with 10 per cent sodium ascorbate reversed the decreased tensile bond strength and may be an innovative option for 'instant' fixed orthodontic treatment after whitening.

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