



## Endodontics

**EFFECT OF DIFFERENT ANTIOXIDANTS ON SHEAR BOND STRENGTH OF COMPOSITE RESIN TO BLEACHED ENAMEL USING 5<sup>TH</sup> AND 7<sup>TH</sup> GENERATIONS OF BONDING AGENTS**

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**ABSTRACT**

**AIMS:** The effect of 10% sodium ascorbate and 5% proanthocyanidin on shear bond strength of composite resin to bleached enamel using 5<sup>th</sup> and 7<sup>th</sup> generations of bonding agents.

**METHODS AND MATERIAL:** Eighty recently extracted maxillary central incisors were randomly divided into four groups of 20 teeth each. Except for group I (control), the labial surfaces of all other specimens were bleached with 38% hydrogen peroxide gel. Groups III and IV were treated with antioxidants 10% sodium ascorbate and 5% proanthocyanidin respectively for 10 min. All four groups were further subdivided into two subgroups of 10 teeth each depending upon the adhesive system used to bond the composite resin. Subgroup A and B were treated with 5<sup>th</sup> and 7<sup>th</sup> generation of bonding agent respectively. All the specimens were stored in deionized water for 24 hours before testing the shear bond strength under universal testing machine.

**RESULTS:** The shear bond strength was maximum for the control group followed by the group which was treated with 5% proanthocyanidin solution. Subgroup A which was treated with 5<sup>th</sup> generation bonding agent resulted in higher shear bond strength than that obtained with Subgroup B.

**CONCLUSIONS:** Within the limitations of this study it was observed that the shear bond strength was greatly increased on the use of antioxidants after bleaching. The shear bond strength on using 5<sup>th</sup> generation bonding agent was higher than that obtained with 7<sup>th</sup> generation bonding agent.

**KEYWORDS :** Antioxidants, sodium ascorbate, grape seed extract, bonding agents, shear bond strength.

**INTRODUCTION:**

Increased interest in aesthetic dentistry has made vital tooth bleaching more popular as a conservative treatment modality for discoloured teeth involving both surface and intrinsic staining to teeth. The bleaching technique is commonly classified as vital and non-vital bleaching or the office and home bleaching. The commonly used bleaching agents contain high concentrations of hydrogen peroxide (30% to 38%) or carbamide peroxide (35% to 37%).<sup>1</sup>

The commonly occurring complications reported after bleaching are pulp irritation, tooth structure alterations, microleakage of restorations, and reduced bond strength of composite resin to tooth structure. One of the important complications reported is the reduction in bond strength of composite resin performed immediately after the bleaching process and it is because of the presence of residual free oxygen radicals that interferes with resin polymerization.<sup>2,3</sup>

Treating the bleached enamel with alcohol before restoration, removal of the superficial layer of enamel and use of adhesives containing organic solvents are some of the techniques that has been used to improve the bond strength to bleached enamel. A delay of at least a week has been recommended in bonding procedure for enamel to return to normal conditions. However this delay in the bonding procedure can be overcome by the use of antioxidants such as sodium ascorbate.<sup>4</sup> Grape seed extract containing oligomeric proanthocyanidin complexes are 50 times more potent than sodium ascorbate.<sup>5</sup>

The two most commonly used adhesive techniques are 5<sup>th</sup> generation and 7<sup>th</sup> generation bonding agents. 5<sup>th</sup> generation bonding system involves etch and rinse technique in which there is a separate etching step with 37% phosphoric acid followed by rinsing with water prior to application of primer/adhesive. 7<sup>th</sup> generation bonding system involves self etch technique in which there is no separate etching step and bonding is achieved by application of a single solution which contains an acid monomer which simultaneously etch, infiltrate and polymerize making it easy and time saving approach for clinician.<sup>6</sup>

However lower bond strength is a common complication with 7<sup>th</sup> generation bonding agents.

However, very few studies can be found in the literature on the effects of antioxidant proanthocyanidin and on the performance of a fifth generation bonding agent and a seventh generation bonding agent, with respect to various surface contaminants.<sup>7</sup> Hence, the aim of this in vitro study was to evaluate and compare the effects of 10% sodium ascorbate versus 5% proanthocyanidin on the shear bond strength of composite resin to bleached enamel on using 5<sup>th</sup> and 7<sup>th</sup> generation bonding agents.

**SUBJECTS AND METHODS:**

Preparation of solutions:

Two solutions were prepared to conduct this study:

- 10 g of sodium ascorbate (Kemphasol Pharmachem Ltd.) was dissolved in 100 ml of distilled water to prepare 10% sodium ascorbate solution.
- 5 g of grape seed extract in the form of powder (Vedeesh Herbs) was dissolved in 100 ml of distilled water to prepare 5% proanthocyanidin solution.

**SPECIMEN PREPARATION:**

The crowns of eighty recently extracted maxillary central incisors were separated from their roots using a diamond disk. On a glass slide the crown segments were fixed on the double sided adhesive tape with the exposed enamel facing downwards (Figure 1). A mold that was open on both ends was placed on the adhesive tape and filled with the mixed self-curing resin. On a polishing machine the 600-grit sandpaper was used to ground flat the labial surfaces of specimens. Depending upon the bleaching regimen used the specimens were randomly divided into four groups of twenty teeth each. Based on the generation of bonding agent used for the bonding procedure each group was further divided into two subgroups of ten teeth each. The distribution of specimens and the study groups are listed in Table 1.

The labial enamel surfaces of 60 specimens were bleached with 38%

hydrogen peroxide gel (Opalescence Boost) twice for 20 minutes (Figure 2). Twenty teeth served as controls (Group I) and did not receive any bleaching treatment. In Group II bleaching was done followed by bonding with composite without the use of antioxidants. In Groups III and IV bleaching was followed by treatment with antioxidants 10% sodium ascorbate solution and 5% grape seed extract solution respectively for 10mins and then rinsed.

All groups were further divided into two subgroups A and B depending upon the adhesive system used for bonding of composite. In Subgroup A, 5<sup>th</sup> generation bonding agent was used in which the labial surface of each specimen was etched with 37% phosphoric acid for 15 seconds, rinsed with water for 15 seconds and bonded with Prime & Bond NT. This was then followed by composite resin build-up of 3mm diameter and 3 mm height. In Subgroup B, 7<sup>th</sup> generation bonding agent was used in which the labial surface of each specimen was bonded with Xeno V+ as per the manufacturer's instructions. This was followed by composite build-up of 3mm diameter and 3 mm height (Figure 3). All the specimens were stored in distilled water for 24 hrs. Shear bond strength (SBS) testing was performed in a universal testing machine (Star Testing System).



Figure 1. Placement of Crown Segment And Mold

Table 1: Distribution Of Specimens And The Study Groups

Group	Bleaching (Y/N)	Antioxidant Used	Subgroup	Bonding Agent Used
Group I (n=20)	No	None	A (n=10)	5 <sup>th</sup> generation
			B (n=10)	7 <sup>th</sup> generation
Group II (n=20)	Yes	None	A (n=10)	5 <sup>th</sup> generation
			B (n=10)	7 <sup>th</sup> generation
Group III (n=20)	Yes	10% sodium ascorbate solution	A (n=10)	5 <sup>th</sup> generation
			B (n=10)	7 <sup>th</sup> generation
Group IV (n=20)	Yes	5% proanthocyanidin solution	A (n=10)	5 <sup>th</sup> generation
			B (n=10)	7 <sup>th</sup> generation



Figure 2. Application of Hydrogen Peroxide Gel



Figure 3. Composite Resin Build-Up of 3mm Diameter and 3mm Height

Table 2. Comparison Of Mean And Sd Values (unpaired 't' Test Results)

		Mean ± SD	Unpaired 't' test value	'p' value	Result / Significance
Group I (n=20)	5th generation (A)	21.05±1.84	13.59	p<0.01	Highly significant
	7th generation (B)	13.17±0.86			
Group II (n=20)	5th generation (A)	12.29±1.23	11.35	p<0.01	Highly significant
	7th generation (B)	7.21±0.70			
Group III (n=20)	5th generation (A)	15.59±1.26	11.81	p<0.01	Highly significant
	7th generation (B)	9.14±1.18			
Group IV (n=20)	5th generation (A)	21.72±1.79	16.68	p<0.01	Highly significant
	7th generation (B)	12.13±1.82			

By applying Student's Unpaired 't' test there is a significant difference between mean values of shear bond strength in 5th generation and 7th generation in Groups I, II, III and IV (i.e. p<0.01)

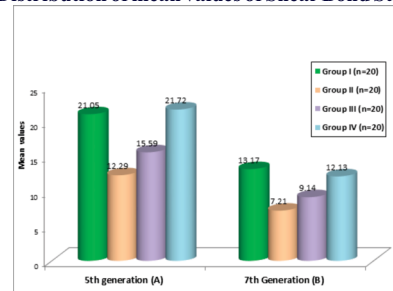
Table 3. POST HOC ANALYSIS: (Using Bonferroni multiple comparison test)

	Group	Mean difference		
		Mean ± SD	't' test value	'p' value and Significance
Group I A	Group II A	8.76±2.59	10.70	0.0014, highly significant
	Group III A	5.45±1.98	8.69	0.0022, highly significant
	Group IV A	-0.67±2.71	0.78	0.512, not significant
Group II A	Group I A	-8.76±2.59	10.70	0.0022, highly significant
	Group III A	-3.71±1.77	45.91	0.0004, highly significant
	Group IV A	-9.43±1.49	19.95	0.009, highly significant
Group III A	Group I A	-5.45±1.98	8.69	0.0011, highly significant
	Group II A	3.31±1.77	5.91	0.0028, highly significant
	Group IV A	-6.12±1.67	11.55	0.0013, highly significant
Group IV A	Group I A	0.67±2.71	0.78	0.124, not significant
	Group II A	9.43±1.49	19.95	0.0019, highly significant
	Group III A	6.12±1.67	11.55	0.0047, highly significant

Table 4. POST HOC ANALYSIS: (Using Bonferroni multiple comparison test):

	Group	Mean difference		
		Mean ± SD	't' test value	'p' value and Significance
Group I B	Group II B	5.96±1.32	47.26	0.000, highly significant
	Group III B	4.03±1.24	34.56	0.001, highly significant
	Group IV B	1.04±0.97	25.71	0.001, highly significant
Group II B	Group I B	-5.96±0.97	47.26	0.0011, highly significant
	Group III B	-1.93±1.25	4.89	0.0014, highly significant
	Group IV B	-4.93±1.79	8.71	0.0028, highly significant
Group III B	Group I B	-4.03±1.63	7.82	0.0019, highly significant
	Group II B	-1.93±1.25	4.89	0.0037, highly significant
	Group IV B	-2.99±2.38	3.97	0.0043, highly significant
Group IV B	Group I B	-1.04±1.52	2.16	0.0214, significant
	Group II B	4.93±1.79	8.71	0.0014, highly significant
	Group III B	2.99±2.38	3.97	0.0089, highly significant

Graph 1. Distribution of mean values of Shear Bond Strength



## RESULTS:

The intra group comparison using Student's Unpaired 't' test showed a statistically significant difference ( $p < 0.01$ ) between mean values of shear bond strength between subgroup A (i.e 5<sup>th</sup> Generation bonding agent) and subgroup B (i.e 7<sup>th</sup> Generation bonding agent) in Groups I, II, III and IV (Table 2). Distribution of mean values of Shear Bond Strength is shown in Graph 1. Inter group comparison test showed a significant reduction in the shear bond strength in Group II after bleaching as compared to the control group I in which no bleaching was done among both the subgroups.

Inter group comparison showed a highly significant difference between mean values of shear bond strength in Group I and Group III among both the subgroups. No statistically significant difference was seen between Group I and Group IV amongst both the subgroups. The bond strength was found to be higher after the use of sodium ascorbate and grape seed extract as antioxidants after bleaching. A significant difference was found between the mean values of shear bond strength in Group II and Group III among both the subgroups.

The values were significantly higher in Group IV in which grape seed extract was used when compared to Group II and Group III ( $p < 0.001$ ) in both the subgroups. The results of one way ANOVA showed that the variation among mean values of shear bond strength were very highly significant ( $p < 0.0001$ ) in all groups and subgroups compared together. The shear bond strength values were compared among 5<sup>th</sup> and 7<sup>th</sup> Generation bonding agent, in all the four groups using Post Hoc Analysis (Bonferroni multiple comparison test. Table 3,4).

## STATISTICAL ANALYSIS:

Statistical analysis was done by descriptive statistics as mean, and SD. Student's Unpaired 't' test of difference between two samples are used to compare all groups and control group. One-way ANOVA test (Tukey-Kramer multiple comparison test) was applied to compare all groups together. Probability  $p < 0.05$  was considered as significant. Post hoc Using Bonferroni multiple comparison test was also used. Statistical analysis software SYSTAT version 12 (By Cranes software's, Bangalore) was used to analyse the data.

## DISCUSSION:

Bleaching is known to give a more pleasing shade to the tooth when done before a composite resin restoration placement.<sup>8</sup>

To prevent cross infection the freshly extracted samples were stored in a disinfectant solution of 0.1% thymol solution until used.<sup>9</sup> To prevent the penetration of the embedding medium or the bleaching solution in the pulp chamber, the decoronated tooth surface was sealed with acrylic resin.<sup>10</sup> For artificial aging of the composite resins, the bonded specimens were stored in deionized water for 24 hours at 37°C before shear bond strength testing.<sup>11</sup>

In this study, 38% hydrogen peroxide gel (Opalescence Boost, Ultradent) was used for two applications of 20 min each. Two applications of gel is used due to the fast degradation of hydrogen peroxide and since significant amount of active ingredients are available only for the first 15-20 minutes. Also, the pH of the gel gets decreased to approximately 5 when single application of longer duration is used which may increase tooth sensitivity.<sup>12</sup>

The residual oxygen layer left behind after bleaching interferes with the resin infiltration into etched enamel and inhibits the polymerization of resin which in turn leads to the compromised bond strength.<sup>13,14</sup>

Peroxide apatite is formed during bleaching with hydrogen peroxide as a result of the substitution of hydrogen radicals by peroxide ions. These structural changes are eliminated upon storage for 2-3 weeks as the peroxide ions decompose and the substituted hydroxyl radicals re-enter the apatite lattice.<sup>15</sup> But this rendered immediate bonding of composite resin to bleached enamel impossible.

Several studies have reported reduced bond strength of composite resin to enamel following bleaching, surface alterations and decreased microhardness of enamel following bleaching.<sup>16</sup>

The research done by Lai SC et al. concluded that inclusion process of peroxide ions could be reversed by the use of antioxidants.<sup>1,17,18</sup> Sodium ascorbate, which is a neutral, non-toxic and biocompatible antioxidant, when used as 10% solution can restore the altered redox potential by

reversing the reduced bond strength of bleached enamel.<sup>19,20</sup> In the present study, the use of sodium ascorbate (Group III) had resulted in higher bond strength with both, 5<sup>th</sup> (15.59 Mpa) and 7<sup>th</sup> (9.14 Mpa) generation bonding agents, when compared to Group II (12.29 Mpa, 7.21 Mpa) in which no antioxidant was used. The previous research done by Kimayi et al<sup>19</sup>, Bulut et al<sup>2</sup> and Lai et al<sup>1</sup> showed the similar findings.

Natural plant extracts are being frequently used as an alternative to chemical and synthetic antioxidants. Hence in this study, emphasis was placed on the use of grape seed extract as antioxidant. Natural sources such as grape seed extract, pine bark extract, lemon tree bark, cranberries and hazelnut tree leaves contain high concentrations of oligomeric proanthocyanidins. As a natural metabolite, it has been proven to be safe as an antioxidant in dietary supplements and in various clinical applications.<sup>20</sup>

The antioxidant potential of vitamins E and C is twenty and fifty fold lesser than those of Grape seed extract (GSE) respectively. The level of health hazard for sodium ascorbate is higher than that of oligomeric proanthocyanidins and also sodium ascorbate is found to be mutagenic for mammalian somatic cells, while oligomeric proanthocyanidins have no mutagenic effect.<sup>21</sup> Natural antioxidant used in this study was capable of reversing the compromised bond strength of composite resins to bleached enamel. So this could be used to avoid the waiting period before bonding to bleached enamel which makes it clinically significant.

It was concluded that treatment with 5% proanthocyanidin increases the bond strength significantly which was 21.72Mpa with 5<sup>th</sup> generation bonding agent and 12.13Mpa with 7<sup>th</sup> generation bonding agent, when compared to other groups (II, III). This could be attributed to the specificity of oligomeric proanthocyanidins (OPCs) for hydroxyl free radicals and the presence of multiple donor sites on oligomeric proanthocyanidins that trap superoxide radicals. Also the esterification of epicatechin by gallic acid in oligomeric proanthocyanidins, which enhances the free radical scavenging activity, contributed in the increased bond strength with 5% proanthocyanidin solution.

5<sup>th</sup> generation 'etch-and-rinse' adhesive (Prime & Bond NT) and the 7<sup>th</sup> generation self-etching bonding agent (XenoV+) were also compared in this study. It was seen that the 5<sup>th</sup> generation bonding agent resulted in higher bond strength when compared to 7<sup>th</sup> generation bonding agent. It is in agreement with other studies that have shown seventh-generation adhesives to be the less effective.<sup>22,23</sup> Primer with inadequate pH and less pronounced etching pattern with self-etch adhesive might have resulted in lower bond strength of 7<sup>th</sup> generation bonding agent.

Stereomicroscopic analysis of the fractured specimens in shear bond testing revealed that all three types of failure were observed in this study. The number of adhesive type failure was higher than the other failure types with 7<sup>th</sup> generation bonding agent groups. The number of cohesive failure was more common with 5<sup>th</sup> generation bonding agent groups.

## CONCLUSION:

Under the limitations of this in vitro study, it can be concluded that:

- For immediate composite bonding after bleaching, the use of antioxidants is mandatory as it reverses the reduced bond strength post bleaching.
- 5% proanthocyanidin yields significantly higher bond strength than that of 10% sodium ascorbate.
- 5<sup>th</sup> generation bonding agent yields significantly higher shear bond strength when compared to shear bond strength with 7<sup>th</sup> generation bonding agent.

However, further research needs to be carried out on the use of these oligomeric proanthocyanidin (OPCs) as surface treatment agents to improve dentin bond strength.

**Clinical Significance:** Use of antioxidants is necessary post bleaching for immediate composite resin bonding.

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