ABSTRACT

Studies have shown that bleaching treatment of enamel tooth surface interferes with bonding and adhesion of composite resin placed immediately after bleaching. The aim of this study was to evaluate the influence of bleaching agent on micro shear bond strength of composite resin to bleached enamel after different intervals of time (control, immediate, one week) using two different based adhesive system of bonding (single bonding and self etch adhesive). Sixty human molar teeth were assigned into six groups (n=10) according to time elapsed between bleaching and bonding and type of bonding used. Group 1: Control group, no bleaching treatment – composite resin bonded using single bond. Group 2: Bleached 38% Hydrogen peroxide – composite resin bonded immediately after bleaching using single bond. Group 3: Bleached 38% Hydrogen peroxide – composite resin bonded one week using single bond. Group 4: No bleaching – control group composite bonded using self etch adhesive system. Group 5: Bleached 38% Hydrogen peroxide – composite resin bond immediately after bleaching using self etch adhesive system. Group 6: Bleached 38% Hydrogen peroxide – composite bonded using self etch one week later.

The micro shear bond strength test was performed using Nexygen MT Lloyd instrument at across head of 0.5 mm/min. The data in MPA were subjected to analysis of variance ANOVA and Tukey’s test as significant difference at P<0.05 – data showed that control group without bleaching using both single bond and self etch adhesive system in bonding composite resin to enamel attained statistically significant highest micro shear bond strength followed by one week group while group bonded immediately after bleaching using self etchant adhesive showed the lowest bond strength. The micro shear bond strength value of composite resin to bleached enamel is time and adhesive system dependant, seven days delay in bonding procedure for composite resin post bleaching to enamel is recommended.
INTRODUCTION

Bleaching and bonding have become a new reality in dentistry, not only because of the availability of new technologies and materials but also as a result of an increase in knowledge and understanding of the mechanism of action of bleaching agents and adhesive systems. Discoloration of anterior teeth is a serious esthetic problem in restorative dentistry and it requires effective treatment.

Bleaching is the most conservative and economical alternative for improving the appearance of discolored vital tooth (Elehaldeb, 2000) \(^1\). Since its introduction by Haywod and Heymann in 1989 \(^2\) night guard vital bleaching has been a procedure that has grown dramatically in popularity in the dental offices because of its efficiency and simplicity to remove intrinsic stains from the teeth (Leonard et al, 1998) \(^3\).

Modifications, improvements and variations of the technique of bleaching process have been introduced. The bleaching process is based on the characteristics permeability of hard tissues that form part of the tooth structure. The bleaching agent is applied to the surface of the tooth and permeates to tooth tissue acting at a distance from the location where it was firstly applied. The bleaching agent will be dissociated when come in contact with saliva and oral fluids, the hydrogen peroxide owing to its instability will decompose into water and oxygen, which penetrates through the pores of the enamel dentine to provide lightening of the teeth. Through oxidation reactions, the large pigmented molecules in enamel and dentine are opened and converted into smaller pigmented molecules which are lighter in color (Goldstein 1995 \(^4\)).

The action mechanism of bleaching agent is based on a complex oxidation reaction releasing oxygen free radicals that penetrate through porosities of enamel prism to dentine and chemically break down organic molecules that pigmented the dentin into carbon dioxide and water released together with nascent oxygen, in some cases restorations will need to be changed because of discoloration and color changes which is not clinically acceptable. When esthetic restorations are needed they should be placed using bond technique (Swift et al, 1988) \(^5\), (Peridago, 1998) \(^6\) however it is very critical that immediate bonding of resin after bleaching will alter and compromise bond strength of composite to tooth structure.

The bleaching agents used in tooth whitening causes oxygen to be released and can cause morphologic alterations in the mineralized structure (Zalkid, 1996) \(^7\).

Alterations on the enamel surface are attributed to the modification of its inorganic and organic composition after treatment with peroxide-based bleaching agent.

These changes in the chemical composition of the enamel considerably diminished the amount of calcium and phosphorus in addition to modifying the morphology of the majority of crystals of the surface layer when compared with enamel not submitted to bleaching (Peridago, 1998) \(^8\). Some studies have shown significantly lower average values of bond strength of composite to bleached enamel (Torneek 90\(^9\), Torneek 91\(^10\), Sung 1999\(^11\)) compared to unbleached enamel.

Others have reported no differences in bond strength values after exposing the enamel specimens to artificial saliva during or after the bleaching treatment (Josey 1996) \(^12\), Murchison 1992) \(^13\). Many methods have been indicated to counteract adverse effects related to the lower bond strength values that following bleaching. The reduction in the bond strength is important since the presence of oxygen released by bleaching processes as it inhibits polymerization of the adhesive system and is responsible for compromising the bond strength between restorative material and dental substrate (Nikado et. al, 2002) \(^14\).

However this reduction in bond strength after bleaching is time dependant. The delaying of bonding procedure after bleaching is one of the recommendations (Disshman 1994) \(^15\), (Miles 1994) \(^16\). The use of different adhesive based systems has been also proposed to result in less compromised composite enamel bond (Spyrides 2000) \(^17\).

Therefore the aim of this study was to verify the proper timing of bonding composite to bleached enamel.
using two different adhesive based system by studying
the micro shear bond strength at different intervals
of time elapsed between bleaching and bonding of
composite resin to enamel using single bond system and
self etchant adhesive system.

MATERIALS AND METHODS

1) Experimental Design

The focus of this study was to examine the influence
of bleaching with 38% hydrogen peroxide on micro
shear bond strength of composite resin to enamel using
two different based adhesive systems at different time
intervals elapsed between bleaching and bonding. The
sixty specimens of molar teeth were divided into six
groups (n=10) as shown in table (1)

<table>
<thead>
<tr>
<th>Group</th>
<th>Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>No bleaching (control), bonded using SB system with composite.</td>
</tr>
<tr>
<td>G2</td>
<td>Bleached with 38% hydrogen peroxide bonded immediately after bleaching using SB system with composite.</td>
</tr>
<tr>
<td>G3</td>
<td>Bleached with 38% hydrogen peroxide bonded after seven days of bleaching using SB system with composite.</td>
</tr>
<tr>
<td>G4</td>
<td>No bleaching (control 2), bonded using SE system with composite.</td>
</tr>
<tr>
<td>G5</td>
<td>Bleached with 38% hydrogen peroxide bonded immediately after bleaching using SE system with composite.</td>
</tr>
<tr>
<td>G6</td>
<td>Bleached with 38% hydrogen peroxide bonded after seven days of bleaching using SE system with composite.</td>
</tr>
</tbody>
</table>

2) Preparation of teeth specimens

A total of sixty molar teeth stored in distilled water,
the teeth were debrided and polished with a rubber cup
along with pumice stone and water at low speed then
washed well with distilled deionized water. The external
enamel faces were ready for bonding to composite
resin by flattening the outer enamel surface to obtain
smooth surface ready for micro shear test by using an
Arotec 250 rotary electrical polisher first then by using
aluminum oxide abrasive paper under a constant water
coolant then polished with diamond pasts. The teeth
then were randomly grouped into six different groups as
experimental design shown in Table (1). The teeth were
immersed in distilled deionized water and remained in
individual jars until bleaching treatment began.

3) Treatment by bleaching agent

A commercial brand of whitening gel containing
38% hydrogen peroxide (Opalescence Quick power
whitening system) was used in the study. A prophylaxis
was performed with a rubber cup and pumice on the
flattened surface of enamel specimen. Next 38% hydrogen
peroxide was mixed with the thicker of the same syringe
as recommended by the manufacturer and applied to
enamel surface of specimens of group G2, G3, G5, G6, for
10 minutes followed by 20 minutes. The application was
repeated for three times after rinsing the surface between
each application. After the third application Group 2
and Group 5 were immediately bonded with composite
with SB and SE adhesive system and then prepared for
micro shear test. Group 3 and Group 6 were immersed
in artificial saliva solution in individual jars for seven
days before bonding with composite resin then test for micro shear were done. Group 1 and Group 4 remained without bleaching treatment immersed in artificial saliva then bonded with composite resin using SB system and SE system. Artificial saliva used in this study was the one described by feather Stone et. al.(17) and modified by Serra and Cury 1996(18).

4) Bonding procedure of composite resin

Test sixty microcylinders of composite resin (Spectrum TPH3 Dentsply) to enamel surface of teeth specimens were attached using two different based adhesive system in Group 1, G2, G3 composite cylinders were bonded using Ana single bond (nordiska dental) in accordance with the manufacturers recommendation while G4, G5 and G6 composite cylinders were bonded using self etch based adhesive system Optibond all in one Kerr.

The chemical composition of bond SB and SE adhesive system illustrated in table (2). The composite resin cylinders were prepared by placing adhesive tape with a 2.5 mm diameter perforation on the tooth surface to delimit the location for the application of bonding system and subsequent attachment of the test cylinders.

A teflon mold measuring (5 cm x 3 cm) were placed over the flattened tooth surface for the application of two layers of resin composite that were light cured for twenty second each increment. The mold was then removed and the specimens were ready for testing of micro shear bond strength with different timing which was as follows: G2 and G 5 micro shear test were performed immediately

G3 and G6 the specimens were stored in artificial saliva in individual jars for seven days then micro shear test was performed. G1 and G4 these specimens remained in artificial saliva without bleaching submitted to micro shear strength test.

5) Micro shear bond strength test procedure:

Each tooth specimen with its own bonded composite microcylinders was secured with tightening screws to the lower fixed compartment of a materials testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with a loadcell of 5 kN and data were recorded using computer software (Nexygen-MT Lloyd Instruments). A loop prepared from an orthodontic wire (0.014” in diameter) was wrapped around the bonded microcylinder assembly as close as possible to the base of the microcylinder and aligned with the loading axis of the upper movable compartment of the testing machine.

A shearing load with tensile mode of force was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The relatively slow crosshead speed was selected in order to produce a shearing force that resulted in debonding of the microcylinder along the substrate-adhesive interface. The load required to debonding was recorded in Newton.

As shown in Fig(1).

TABLE (2) Adhesive systems used in this study.

<table>
<thead>
<tr>
<th></th>
<th>Single bond System</th>
<th>Self etch ant System</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Type</strong></td>
<td>Ana single bond</td>
<td>Opti bond all in one Kerr</td>
</tr>
<tr>
<td></td>
<td>Total bonding Nordiska Dental</td>
<td></td>
</tr>
<tr>
<td><strong>Chemical</strong></td>
<td>-Methacrylated phosphoric acid.</td>
<td>-Glycerol phosphate diamethacrylate (6PDM).</td>
</tr>
<tr>
<td><strong>composition</strong></td>
<td>-HEMA.</td>
<td>-Mono and difunctional methacrylate monomers.</td>
</tr>
<tr>
<td></td>
<td>-Elastomeric Resin.</td>
<td>-Fillers.</td>
</tr>
<tr>
<td></td>
<td>-TEG DMA.</td>
<td>-Photo initiator CQ</td>
</tr>
<tr>
<td><strong>Solvent type</strong></td>
<td>Acetone-based</td>
<td>Water, acetone and ethanol (tertiary solvent)</td>
</tr>
</tbody>
</table>
Micro-Shear bond strength calculation;

The load at failure was divided by bonding area to express the bond strength in MPa:

$$\delta = \frac{P}{\pi r^2}$$

Where; \( \delta \) = bond strength (in MPa)  
\( P \) = load at failure (in N)  
\( \pi \) = 3.14  
\( r \) = radius of microcylinder (in mm)

The stress-deflection curves were recorded using computer software (Nexygen-MT Lloyd Instruments).

6) Statistical Analysis

Data of micro shear bond strength was recorded as Mean ± Standard Deviation in MPa. Data analysis was performed on several steps. Initially, separate analysis was performed with one-way analysis of variance ANOVA followed by Tukey’s multiple comparison tests to evaluate the significance between groups. A two-way analysis of variance ANOVA was used to examine effects of adhesive bonding system used (SB vs. SE) also time factor elapsed after bleaching treatment (no bleaching control immediately versus seven days delay) and also interactions between those factors were analyzed. Statistical analysis was performed using graphed prism-4 statistics software for windows. P-values less than 0.05 are considered to be statistically significant in all tests.

RESULTS

The results of micro shear bond strength tests for all specimens of all six groups using two different adhesive bonding system at different time intervals of delay following bleaching treatment were presented in Table (3) and represented in Fig(2). The data in Table (3) showed that the mean of micro shear bond strength of group 1 (Control Group) using SB system was the highest as it was 29.06 MPa.

While group 2 and group 3 after bleaching with immediate bonding or delay of seven days the mean of bond strength declined to value of 16.1, 16.3 MPa. While with self etch adhesive system in group 4 as no bleaching treatment was done mean of bond strength was 25.45 MPa. While with bleaching in group 5 and group 6 the mean value decreased sharply to 10.66 and 11.2 as shown in Fig (2).

FIG. (1) a- Nexygen-MT Lloyd Instruments Micro shear bond test used in this study and b- The tooth specimen with composite microcylinder.

FIG. (2) A column chart of \( \mu \)-shear bond strength mean values(MPa) for all groups.
Same super/sub-script letter indicating non statistically significant difference (Tukey’s $p > 0.05$).

Different super/sub-script letter indicating statistically significant difference (Tukey’s $p < 0.05$).

In all groups 1, 2, 3 using single bond adhesive system and groups 4, 5, 6 of self etch adhesive system the control groups G1 and G4 without bleaching treatment attained statistically significant highest bond strength values. After one week delay between bleaching treatment and bonding procedure as in group 3 and 6 intermediate values of bond strength were obtained. Immediate bonding after bleaching as in group 2 and 4 showed the statistically significant lowest bond strength as shown in Table (3) and represented in Fig (2). As shown in Table (4) and represented in Fig(3)

One-way ANOVA analysis showed significant differences among self etch groups ($F=114.45$ as $p<0.05$) pair wise Tukey’s multiple comparison tests showed that the difference between control group 1 and immediate bonding of G5 were statistically significant, also G4 and delayed bonding in group 6 were also statistically significant while G5 immediate versus group 6 one week was non significant. As shown in Table (5) and represented in Fig. (4) One way analysis ANOVA analysis showed significant difference among single bond groups ($F=65.49$ as $p<0.05$) pair wise Tukey’s multiple comparison tests showed the difference between control group 1 and immediate bonding group 2 were statistically significantly . Although self etch groups (G4, G5 and G6) recorded lower micro shear bond strength values than single bond groups (G1, G2 and G3) two-way ANOVA revealed influence of adhesive system tested ($F=0.1569$, $p>0.05$) a influence regarding elapsed time ($F=0.6833$, $p>0.05$) on micro shear bond strength values. The interactions of these two factors was statistically ($F=0.001608$, $p>0.05$) as shown in Table (6), Fig. (5),fig(6).

![FIG. (3) A column chart of $\mu$-shear bond strength mean values(MPa) for SE sub-groups.](image-url)
TABLE (4) One way analysis of variance ANOVA comparison \( \mu \)-shear bond strength between SE sub-groups.

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>SS</th>
<th>df</th>
<th>MS</th>
<th>F calc.</th>
<th>P-value</th>
<th>F tab.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>984.9219</td>
<td>2</td>
<td>492.461</td>
<td>114.4509</td>
<td>&lt;0.0001***</td>
<td>3.554561</td>
</tr>
<tr>
<td>Within Groups</td>
<td>77.45065</td>
<td>18</td>
<td>4.302814</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>1062.373</td>
<td>20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\( SS; \) Sum-of-squares \( df; \) degree of freedom \( MS; \) Mean square \( \text{calc.}; \) calculated \( \text{tab.}; \) tabulated **; high significant.

TABLE (5) One way analysis of variance ANOVA comparison \( \mu \)-shear bond strength between SB sub-groups.

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>SS</th>
<th>df</th>
<th>MS</th>
<th>F calc.</th>
<th>P-value</th>
<th>F tab.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Between Groups</td>
<td>771.4425</td>
<td>2</td>
<td>385.7212</td>
<td>65.49615</td>
<td>&lt;0.0001***</td>
<td>3.554561</td>
</tr>
<tr>
<td>Within Groups</td>
<td>106.006</td>
<td>18</td>
<td>5.88922</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>877.4484</td>
<td>20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\( SS; \) Sum-of-squares \( df; \) degree of freedom \( MS; \) Mean square \( \text{calc.}; \) calculated \( \text{tab.}; \) tabulated **; high significant.

![FIG. (4) A column chart of \( \mu \)-shear bond strength mean values (MPa) for SB sub-groups.](image1)

![FIG. (5) Comparisons of change in \( \mu \)-shear bond strength mean values for both groups as a function of different intervals.](image2)

TABLE (6) Two way analysis of variance ANOVA test of significance comparing variables affecting \( \mu \)-shear bond strength.

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Df</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>P value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesives</td>
<td>1</td>
<td>12.87</td>
<td>12.87</td>
<td>0.1569</td>
<td>0.6990</td>
</tr>
<tr>
<td>Intervals</td>
<td>2</td>
<td>112.1</td>
<td>56.04</td>
<td>0.6833</td>
<td>0.5235</td>
</tr>
<tr>
<td>Interaction</td>
<td>2</td>
<td>0.2638</td>
<td>0.1319</td>
<td>0.001608</td>
<td>0.9984</td>
</tr>
<tr>
<td>Residual</td>
<td>12</td>
<td>984.2</td>
<td>82.02</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\( Df; \) degree of freedom \( SS; \) Sum-of-squares \( MS; \) Mean square
DISCUSSION

The possibility that dental bleaching treatment interferes with the bonding of composite resin restoration remains a big question and confusing point some in vitro studies reported alterations in the chemical and morphological structures of enamel (Basting et al 2003(19), Josey et al 1996 (11), Rodrigues et al 2001 (20))

Such structural alterations presented in diminished micro shear strength values after dental bleaching, which may be explained and related to residues from hydrogen peroxide degradation on enamel tooth surface affecting the polymerization of adhesive systems and resin composite (Bastine et al 2001 (21), Perdiago et al 1998 (6)). Use of hydrogen peroxide at high concentration 38% as used in this investigation is the one of the most common bleaching agents used in dental office. The action mechanism occurs through a complex oxidation reaction as hydrogen peroxide decomposed into water and nascent oxygen when come in contact with water or saliva. This liberated oxygen penetrates rapidly through the enamel porosities and organic matrix of the enamel tissue. Then oxygen promptly with pigments possibly causing weaking of links between the chromatogenic molecules and the organic matrix to rupture. The chromophoric molecules are oxidized by the nascent oxygen ions and are broken down into smaller, less complex and light molecules (Mc evoysa et al, 1989) (22). However (Perdigao et al 1998) found bleaching done with carbomide peroxide didn’t cause alterations in the relative concentration of oxygen present on enamel surface and explained that the oxygen remaining in dentine could be responsible for reduction in bond strength after bleaching with hydrogen peroxide, for those reasons they recommended that immediate bonding to bleached enamel should be avoided to allow delay in time for the residual oxygen on the tooth surface to be eliminated (Titley et al 1993) (23).

Jacobson and Soderholm 1995 (24) suggested that the type of adhesive based system depending on type of solvent used in adhesive system appeared to have a great influence on the shear bond strength of composite resin to bleached enamel. Acetone and ethanol based adhesive system as being the most indicated types of solvents used that had the ability to carry the hydrophilic monomers more effectively into the tooth structure as acetone considered as strong water chasing agent. This explained the data of our study as Ana single bond used in this investigation in G1, G2 and G3 showed the highest micro shear bond strength than Opti bond self etch group used in G4, G5 and G6 as single is acetone based adhesive system which is a strong chasing agent where as Opti bond self etchant is tertiary water, acetone and ethanol based solvents. Self-etching adhesive system acting with a higher presence of humidity, which may inhibit polymerization of composite resin compromising bond strength values. This is in contradictions of some other studies relating its capacity to a reduced surface humidity and an increase shear bond strength values (Sung et al 1999) (10). Which would appear to have been to revert the reduction in the micro shear bond strength values, thus coinciding with the results obtained by Spyrides et al 2000 (16).

These findings of this investigation along with those of previous studies provide the dental professional with a fundamental knowledge of a common in-office dental bleaching technique as well as an understanding of proper timing of bonding composite to bleached enamel.

The findings of this study suggests that the acetone based adhesive system of composite resin restorations to enamel is more recommended than water based adhesive system as it showed higher values of shear bond strength.
The bonding procedure of composite resin to enamel as closing diastemas and dental reanimation should be performed after at least seven days following in office bleaching with 38% Hydrogen peroxide.

Rotstein et al (25) indicated that bleaching agents cause changes in the levels of calcium and phosphorous that was presented in the hydroxyapatite crystal, main building blocks of dental hard tissue. Perdiago et al 1998(6) stated that the changes in proteins and minerals content of superficial layers of enamel may be responsible for reducing bond strength.

Others explain compromising shear bond strength of composite to bleached enamel as resin tags in bleached enamel are less numerous, shorter and less defined than those in unbleached enamel in addition to presence and evidence of bubbling (26) (27).

CONCLUSION

In view of the results obtained from this study, it was clear that (1) after in office bleaching treatment for enamel tooth surface with a whitening agent of 38% hydrogen peroxide, composite resin restorations are recommended to be bonded with a delay of at least seven days, (2) immediate bonding should be avoided. (3) The type of adhesive system used in bonding either acetone based or water based majority affects the values of micro shear bond strength. (4) The acetone based adhesive system showed better bonding strength than water based.

REFERENCES


