



BOND STRENGTH OF SELF ADHERING COMPOSITE AS POST ENDODONTIC RESTORATION AFTER NON VITAL BLEACHING- AN EX-VIVO STUDY

Dental Science

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ABSTRACT

Objectives: To evaluate the bond strength of self adhering composite as a post endodontic restoration after non vital bleaching.

Material and Methods: 80 freshly extracted intact human permanent mandibular first molars were embedded in acrylic resin with their occlusal surfaces facing upwards, which were then ground axially till 2mm below the DEJ. The mounted samples were then randomly divided into two groups: Group 1 (unbleached), further subdivided into SUB (Self adhering composite), SSUB (Self etch+ Self adhering composite), NUB (Self etch+ Nanohybrid composite), and Group 2 (bleached), in which samples were first subjected to non vital bleaching and subdivided into two subgroups; Subgroup A consisting of: SAB1 (Bleaching+Self adhering composite), SAB2 (Bleaching+Self etch+Self adhering composite), SAB3 (Bleaching+Antioxidant+Self adhering composite), SAB4 (Bleaching+Antioxidant+Self etch+Self adhering composite); and Subgroup B, which included NHB (Bleaching+Antioxidant+Self etch+Nanohybrid composite). Thereafter, composite cylinders were fabricated on the surface of all the samples using teflon molds according to the type of composite for each group. The samples were then incubated for 24 hours and then subjected to shear bond strength testing. The results obtained were subjected to statistical analysis by One Way ANOVA F-Test and Unpaired "t" test were used.

Result: The mean bond strength values obtained were:

Unbleached: NUB> SSUB> SUB

Bleached: NHB> SAB4> SAB2> SAB3> SAB1

Conclusions: Self adhering composites exhibited significantly higher bond strength when used with self etch approach. If immediate bonding after non vital bleaching is necessary, antioxidant treatment should be done.

KEYWORDS

Self Adhering Composites, Non Vital Bleaching, Shear Bond Strength

INTRODUCTION:

Discolouration of traumatised teeth with pulpal necrosis is a major concern affecting their esthetics. Due to the raging awareness regarding esthetic improvement among the patients of all age groups, many therapies have been proposed. Of these, non vital intracoronal bleaching has been successfully carried out for managing discolouration with effective outcome. Different techniques and agents have been evaluated and recommended for the treatment of tooth discolouration, which utilise hydrogen peroxide, sodium perborate or carbamide peroxide in varying concentrations depending on the technique adopted for the clinical condition. In-office intracoronal bleaching and walking bleach are two commonly used techniques along with their modifications, followed by adhesive post endodontic restorations.¹

Success of endodontic treatment is highly dependent not only on apical sealing but also on coronal seal obtained through post endodontic restoration. Poor post endodontic restorations can result in coronal microleakage, tooth discolouration, dislodgement of restorations and tooth fractures as well. Recently, self adhering composites have been introduced which claim to be less technique sensitive as they do not involve the traditional steps of etching and bonding, while offering sufficient bond strength to dentin.²

However, irrespective of the technique, a significant reduction in the bond strength of enamel and dentin after bleaching has been frequently documented by many authors,^{3,4} which has been correlated with the formation of a layer of oxygen free radicals in the form of oxygen, hydroxyl and perhydroxyl ions liberated from the dissociation of hydrogen peroxide present in the bleaching agent, inhibiting the polymerisation of the composite resin materials.⁵ Various strategies have been documented in literature to allow reversal of the bond strength for successful placement and outcome of adhesive resin restorations. A delay of one to two weeks is usually recommended before final restoration, during which the layer of oxygen free radicals

gets removed by itself.⁶ Immediate adhesive restorations after bleaching can be carried out by treating the bleached surface with antioxidants such as sodium ascorbate which has been documented to remove the oxygen free radicals from the bleached surface, thus restoring normal bond strength values.^{1,6,7}

There have previously been studies evaluating the effect of bleaching and further surface treatment with antioxidants on the bond strengths of conventionally used composites, which have reported to give satisfactory results. However, the newer self adhering composites have not yet been tested under this protocol. Thus, the present study was undertaken to evaluate the effect of different bonding protocols on dentin on the bond strength of self adhering composites.

MATERIAL AND METHODS:

Sample selection and storage:

Eighty freshly extracted intact human permanent mandibular first molars, were obtained and debrided of soft and hard tissue debris using ultrasonic scaler (DTE D5, Gullin, Woodpecker Medical Instrument Co. Ltd.). Teeth that were free of any surface defects (caries, calculus, cracks, hypoplasia and other developmental defects) and previous restorations were selected after observing under magnification (X20) (MINTRON, dental chair mount microscope, Model- MTV-32K9HP, Taiwan). The selected teeth were stored in Chloramine T solution (0.5%) (Hi-Media Labs, India) until further use.

Sample fabrication:

The selected teeth were then individually mounted in self cure acrylic resin using teflon moulds by embedding the roots into the resin close to CEJ, with their occlusal surfaces facing upwards. The occlusal crown surfaces were then reduced axially using a diamond disc in a straight handpiece (NSK, Eschborn, Germany) under water coolant till a level 2mm below DEJ, keeping the discs parallel to CEJ. The exposed dentin surfaces were then smoothed with polishing discs (Super Snap Mini by Shofu, GmbH Ratingen, Germany). Care was taken to prevent

contamination as well as dehydration of the exposed dentin surface by storing the samples in Chloramine T solution until further use.

The dentin surfaces of mounted samples were rinsed thoroughly with running water for 20 seconds and blot dried so as to obtain a clean and reasonably dry surface, and were then randomly divided into two groups based on the bonding protocol to be followed. The materials used in the study have been enumerated in the following table (Table 1).

Table 1: Materials used in the study

1. Self adhering composite	Dyad Flow ((Kerr Corporation, 1717 West Collin Avenue Orange, CA 92867 USA)
2. Nanohybrid composite	Filtek Z250 XT (3M ESPE, Conway Avenue, St. Paul, USA)
3. Self etch bonding agent	Clearfil SE Bond (Kuraray Noritake Dental Inc., 1621 Sakazu, Kurashiki, Okayama, Japan)
4. Bleaching agent	Opalescence Boost PF (Ultradent Products, 505 West Ultradent Drive, South Jordan, USA)
5. Antioxidant	10% Sodium ascorbate (Now Foods, 385 S, Glen Elyn Road, Bloomingdale, IL 601108, USA)

Samples in Group 1 were further distributed randomly into **three subgroups** based on the type of composite and the bonding strategy used for cylinder fabrication and bond strength testing.

- **Sub Group SUB: Self-adhering composite cylinder** on unbleached dentin surface (n=10)
- **Sub Group SSUB: Self etch + Self-adhering composite cylinder** on unbleached dentin surface (n=10)
- **Sub Group NUB: Self etch + Nanohybrid composite cylinder** on unbleached dentin surface (n=10)

Samples assigned to Group 2 were first subjected to in-office intracoronal bleaching procedure using **Opalescence Boost PF** (Ultradent Products, 505 West Ultradent Drive, South Jordan, USA). The activator and the bleaching gel were mixed according to the manufacturer's instructions. The bleaching agent was applied on the dentin surface of the samples for 20 minutes and then removed from the surfaces of all the samples by thorough rinsing for one min by running water.

Thereafter the samples in Group 2 were again randomly divided into **two subgroups** based on the type of composite and the bonding strategy used for cylinder fabrication and bond strength testing.

A. Subgroup A: Self-adhering composite Bleached group (SAB) (n=50) which further included:

- 1) **Bleaching + Self-adhering composite cylinder (SAB1) (n=10)**
- 2) **Bleaching + Self etch + Self adhering composite cylinder (SAB2) (n=10)**
- 3) **Bleaching + Antioxidant treatment + Self adhering composite cylinder (SAB3) (n=10)**
- 4) **Bleaching + Antioxidant treatment + Self etch + Self adhering composite cylinder (SAB4) (n=10)**

B. Subgroup B: Nanohybrid composite Bleached group (NHB) which included: Bleaching + Antioxidant + Self etch + Nanohybrid composite cylinder (n=10)

Protocol for antioxidant treatment:

A 10% sodium ascorbate solution was prepared by mixing 10 mg of sodium ascorbate powder (Now Foods, 385 S, Glen Elyn Road, Bloomingdale, IL 601108, USA) into 100ml of distilled water. The bleached dentin surfaces of the samples in subgroups SAB3, SAB4 and NHB were then immersed in 10% sodium ascorbate solution for one hour. A fresh solution of 10% sodium ascorbate was prepared for the different groups. The surfaces were then rinsed with distilled water and blot dried for the procedure of bonding of the composite cylinders.

Preparation of bonding surface:

In subgroups (SSUB, NUB, SAB2, SAB4, NHB) where the samples were subjected to self etch approach, Clearfil SE Bond was applied in the following manner. First, Clearfil SE Bond Primer was applied on the exposed surfaces with the help of an applicator tip and left for 20

seconds, followed by thorough drying with mild air flow, after which Clearfil SE Bond bonding agent was applied on the exposed surfaces with the help of an applicator tip, air flowed gently and light cured (Woodpecker ILED, China) for 10 seconds.

Protocol for fabrication of composite cylinders:

The bonding surfaces of all the samples were delimited by placing a piece of adhesive paper tape with a hole of 4 mm diameter punched in it over the dentin surface. This left a circular area of dentin available for bonding of resin cylinders in the centre of the dentin surface for shear bond strength testing.

Teflon ring moulds (4mm x 5mm) were used for making composite resin cylinders. The ring moulds were stabilized in the centre of the dentin surface over the hole with the help of sticky wax placed outside the mould (junction of adhesive tape and mould). The teflon ring moulds were filled incrementally with composite resin (Filtek Z 250 XT) in increments of 2mm with the help of plastic filling instrument. In case of self adhering composite (Dyad Flow), it was dispensed onto the dentin surface into the teflon ring with provided dispensing tip. The first increment was brushed on the sample surface and light cured followed by subsequent increments. Level of 2 mm increment was pre-marked on the ring. Curing of each increment was done from all the sides exposing the entire surface of cylinder to a visible light source for 20 seconds at a power output of 800 mW. The incremental addition was done until the ring mould was completely filled. Then the teflon ring moulds were safely removed from the tooth by cutting it with a scalpel without causing any harm to the bonded composite cylinder.

All the prepared samples were then incubated at 37 °C and 100% humidity for 24hrs and then subjected to testing for shear bond strength. The samples were mounted on the Universal Testing Machine (Saumya Technologies, UTM-D2) to calculate the shear bond strength (at a cross head speed of 0.5 mm/minute with 250 KgF load) parallel to the machine line of travel.

The values thus obtained were tabulated and subjected to statistical analysis using One Way ANOVA F-Test and Unpaired "t" test.

RESULTS:

The mean shear bond strength values of all the subgroups are shown in Fig 1 & Fig 2. In the unbleached group, the highest mean shear bond strength was observed for subgroup NUB and the lowest for subgroup SUB, whereas, in the bleached group the highest mean shear bond strength was observed for subgroup NHB and the lowest for subgroup SAB1. On pair wise comparison between subgroups in unbleached and bleached groups, statistically significant difference was found between the shear bond strengths of subgroups of both the groups, except for subgroups SAB3 and SAB2 ($p < 0.05$) (Table 2). The bond strength of unbleached groups was significantly higher than that of the bleached groups. Subgroups involving the use of self adhering composite exhibited significantly lower bond strength than that of nanohybrid composite subgroups. However, the use of self etch resulted in a significant increase in bond strength of self adhering composites, with or without bleaching. (Figures and Tables are as below)

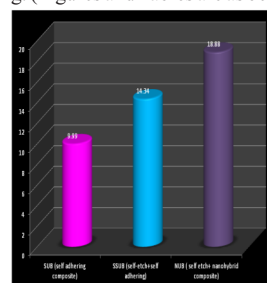


Fig 1: Mean shear bond strength values (MPa) in unbleached group

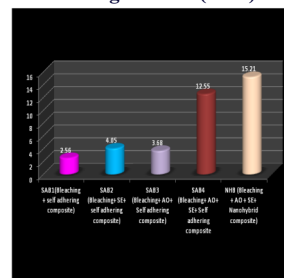


Fig 2: Mean shear bond strength values (MPa) in bleached group

Table 2: Pair wise comparison of shear bond strength (MPa) between different pairs of subgroups in unbleached and bleached groups.

S.NO.	Pair of groups	p-values (unpaired "t" test)
	Unbleached group	
1	SUB & SSUB	0.0001* (p<0.05)
2	SSUB & NUB	0.0000* (p<0.05)
3	SUB & NUB	0.0000* (p<0.05)
	Bleached group	
1	SAB1 & SAB2	0.0013* (p<0.05)
2	SAB1 & SAB3	0.0101* (p<0.05)
3	SAB1 & SAB4	0.0002* (p<0.05)
4	SAB1 & NHB	0.0000* (p<0.05)
5	SAB2 & SAB3	0.1785** (p>0.05)
6	SAB2 & SAB4	0.0001* (p<0.05)
7	SAB2 & NHB	0.0000* (p<0.05)
8	SAB3 & SAB4	0.0001* (p<0.05)
9	SAB3 & NHB	0.0000* (p<0.05)
10	SAB4 & NHB	0.0002* (p<0.05)

*shows a significant difference between groups at 0.05 level of significance, i.e. p<0.05

**shows no significant difference between groups at 0.05 level of significance, i.e. p>0.05

DISCUSSION:

Dentin dynamicity and heterogenicity are the major factors influencing the shear bond strength of resin composites to dentin. In the present study, the bond strength values obtained in general for both, **Dyad Flow** and **Filtek Z250** were comparatively lower than in previously conducted studies.^{2,3} This can be attributed to the fact that in the present study, dentin near the pulp was chosen, which has been reported to have lower bond strength as compared with superficial dentin.^{8,9,10}

In the present study, subgroup **NUB** resulted in significantly higher bond strength as compared to the subgroups **SUB** and **SSUB**, which is in accordance with previous studies that have reported lower bond strengths of self adhering composites. Yazici *et al.*¹¹ reported in their study that the acidity of GPDM (pH-1.9) present in Dyad Flow is not high enough to modify the smear layer and allow proper penetration of resin into the substrate, resulting in low micromechanical retention, and subsequently lower bond strength. In the present study, although removal of smear layer was done from the surface of samples by polishing, poor bond strengths were obtained with Dyad Flow, which suggests that the etching effect of GPDM is insufficient to facilitate penetration of resin even in the absence of smear layer. Furthermore, Dyad Flow features incorporation of a bonding agent into the resin material, causing incomplete infiltration of filled adhesive into the demineralised dentin, ineffective sealing of dentinal tubules, degradation of exposed collagen and the resin material.¹² Miyazaki *et al.*¹³ and Brueckner *et al.*¹⁴ suggested that the increased viscosity of resin due to incorporation of fillers might decrease the dentinal wetting, decreasing the penetration of monomers resulting in decreased bond strength. They also reported that Dyad Flow exhibited formation of thin and sparse tags with limited depth and density.

However, an increase in the bond strength was observed when Dyad flow was used with a self etching bonding agent in both unbleached and bleached groups. The probable explanation for this can be the increased penetration of the resin composite facilitated by the additional decalcification caused by the acidic primer of the self etch bonding agent used in this study, that is, Clearfil SE Bond (pH=2). Various studies have reported improved bond strength of self adhering composites when used with a self etch bonding system.^{15,16}

In the present study, the bleached subgroups **SAB1**, **SAB2** and **SAB3** exhibited a significantly reduced mean bond strength than the unbleached group, for Dyad Flow. Other resin composites have also reported reduced bond strengths after bleaching. **Toko and Hisamitsu**¹⁷ attributed the loss in bond strength to the adverse effect of hydrogen peroxide which removes the non fibrous organic content within the tooth substrate. It has been reported that matrix metalloproteinases (MMP's) are associated with degradation of the collagen component of dentin. The oxidative products of the bleaching agents

have also been shown to inactivate tissue inhibitors of MMP's, which leads to further breakdown of collagen. Presence of degraded collagen further reduces bond strength of resin composites to dentin.

Moreover, decomposition of hydrogen peroxide, the bleaching procedure results in a residual layer of free oxygen radicals on the tooth substrate, since hydrogen peroxide degradation releases oxygen free radicals or the perhydroxyl anion. **Torneck *et al*¹⁸** and **Rueggeberg and Margeson**¹⁹ reported that oxygen free radicals could either interrupt the penetration of resin components into the enamel and dentin or disrupt the polymerization of resin. Several studies have confirmed that composite resin restoration performed immediately after bleaching results in decreased bond strengths with dentin.

The exposure of dentin to bleaching agents has been shown to cause reduction in microhardness values which in turn may also result in changes in the mechanical properties of dentin. **Perinka *et al*²⁰** found that dentinal characteristics such as dentin hardness, dentin thickness and concentration of calcium might influence bond strength to dentin. **Rotstein *et al*²¹**, **Pecora *et al*²²** and **Chng *et al*²³** also reported that bleaching with hydrogen peroxide based bleaching agents results in lower concentrations of calcium and phosphate compared with unbleached dentin, and that a significant reduction in human dentine surface microhardness following treatment with 30% HP.

In the present study, the use of antioxidant resulted in significantly increased bond strengths after bleaching. The antioxidantizing ability of sodium ascorbate aids to neutralize and reverse the oxidizing effects of the bleaching agent. Therefore, there is restoration of the redox potential of the oxidized bonding substrate and continuation of polymerization of adhesive without permanent termination.⁶ As mentioned earlier, the reactive oxygen species generated by the bleaching gels are capable of activating the MMP's present in the dentin. Considering that sodium ascorbate reduces the amount of reactive oxygen species present on the surface of dentin, thus, it may inhibit or even significantly reduce the action of MMP's on bleached dentin, subsequently stopping the collagen degradation and establishing better bond strength of resin composites to dentin.^{6,7} **Whang *et al*²⁴**, **Kaya and Turkun²⁴** and **Trinidad *et al*²⁵** found that 10% sodium ascorbate solution was able to reverse compromised bond strengths of composites to dentin equivalent to delayed bonding to dentin after 4 weeks. However, in this study it was observed that use of antioxidant alone after bleaching (**SAB3**) resulted in significantly lower bond strength of self adhering composite as compared to when self etch bonding agent was used with antioxidant treatment (**SAB4**).

Nevertheless, even after the application of self etch and antioxidant, the bond strength of self adhering composites remained significantly lower than the unbleached group (**SSUB**). The bonding of self adhering composites occurs majorly by micromechanical retention and partly by chemical bonding to the calcium of hydroxyapatite. Due to increased decalcification of dentin after bleaching, the available calcium is not sufficient for proper chemical bonding. Moreover, the bond formed by micromechanical retention is not sustainable all by itself to maintain the bond strength of self adhering composite to deep dentin comparable to that of a nanohybrid composite, even after surface treatment with antioxidant and application of a self etch bonding agent, as seen in this study.

CONCLUSION:

The following conclusions can be drawn from the present study:

- Nanohybrid composite provided significantly higher bond strengths to dentin (with or without bleaching) as compared to self adhering composite, when compared for all the adhesive strategies tested in the study.
- The application of a self etch bonding system before bonding of self adhering composite to dentin resulted in significant increase in the bond strength, with or without bleaching.
- Application of 10% sodium ascorbate after bleaching resulted in significantly improved bond strength values of both nanohybrid and self adhering composites, when used with self etch.

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