



## Adhesive bond integrity of dentin conditioned by photobiomodulation and bonded to bioactive restorative material

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### ABSTRACT

**Aim:** The aim of this in-vitro study is to investigate the shear bond strength (SBS) of Bioactive bulk fill restorative material (BARM) (Activa) by surface pre-treatment using Er,Cr:YSGG (ECL) on dentin in comparison to conventional dentin conditioning techniques.

**Material and Methods:** Sixty extracted non-carious, non-fractured, restoration free human molars were collected and mounted vertically in acrylic resin. The buccal surface of all molars were ground and polished with abrasive paper. The specimens were randomly allocated into four groups (n = 15) according to the type of surface treatment. Group 1 ECL + BARM, Group 2 ECL + Ketac + BARM, Group 3 Conventional Etch and Rinse + BARM, Group 4 Self-etch + BARM. The specimens from each group were positioned in a universal testing machine for SBS testing. Ten samples from each group were assessed for modes of failure. Means and standard deviations were compared using analysis of variance (ANOVA) and Tukey's post hoc test at a significance level of  $p < 0.05$ .

**Results:** BARM bonded to dentin surface etched with conventional etch and rinse technique (group 3) exhibited SBS value of  $18.45 \pm 1.34$ . Similarly, the lowest bond strength was observed by group 4 (self-etch regime) bonded to BARM ( $16.09 \pm 0.81$ ). The bond strength among groups 1 (ECL + BARM), 2 (ECL + Ketac + BARM) and 3 (etch and rinse + BARM) were found to be comparable ( $p > 0.05$ ).

**Conclusion:** Conditioning of dentin with photobiomodulation for the adhesive bonding of BARM showed comparable bond strength outcomes to conventional conditioning techniques.

### 1. Introduction

Significant improvements in filler technology of composites has permitted them to be used in posterior restorations, where previously amalgam was considered as a material of choice [1]. Better distribution of filler particles in composites has reduced resin content, which indirectly have decreased polymerization shrinkage [2]. To overcome the shortcomings of direct posterior aesthetic restorative filling materials (i.e. poor biocompatibility, operator dependence and requirement of a bonding system) a new bio-dentin replacement material with bioactive resin and bioactive glass (BAG) filler (Activa) has been recently introduced [3,4].

Bioactive restorative material (BARM) (Activa) releases more fluoride than glass ionomers and is the first permanent restorative material that responds to variations in oral environment [5]. BARM

forms chemical bond with the tooth structure through an intermediary layer from where calcium and phosphate ions flow from dentin into BARM whereas an influx of fluoride, aluminium and strontium takes place from BARM to dentin confirming a strong adhesion. Moreover, BAG and polyacid in BARM improves bond integrity by an acid and base neutralization bonding to the tooth mechanically [6,7].

One critical factor in the clinical success of adhesive restorative material is its bond integrity [8]. To establish an adhesive bond strength, the smear layer formed by tooth preparation is either altered or removed by surface treatments including, acid etch, abrasion and photobiomodulation [9]. An array of bonding systems are employed to critically enhance and augment the bond integrity of restorative materials mainly based on the concept of total-etch and self-etch [10]. Universal conventional method of etch and rinse which comprises more steps, is technique sensitive and unfriendly to the clinician but removes

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smear layer completely [11]. In contrast, there is ‘all in one’ adhesive which is less technique sensitive, simpler and impregnates the smear layer [12]. Apart, from both dentin conditioning methods the use of Er,Cr:YSGG laser (ECL) as dentin treatment has gained interest for both clinicians and researchers.

ECL surface pre-treatment is convenient, simple and easy to use in dental practice. It works on the principle of ablation of enamel and dentin surface comparable to acid etch technique by working at a wavelength of 2790 nm [13]. It has a bactericidal property and an added benefit of removing the smear layer [14]. The use of ECL as surface conditioner for dentin and enamel surfaces have revealed convincing and positive outcomes [15–18]. Regarding the application of ECL, a study by Sakr., showed BARM to exhibit clinically acceptable bond strength [19]. Furthermore, Efflandt et al., indicated that BAG encourages bond strength due to better affinity with dentin collagen [20].

Therefore, it is hypothesized that the bond strength of BARM (Activa) to dentinal surface conditioned with ECL will exhibit comparable outcomes to conventional (etch and rinse) dentin conditioning techniques. The aim of this in-vitro study is to investigate the SBS of BARM (Activa) with dentin surface conditioning by ECL irradiation in comparison to conventional dentin conditioning techniques.

## 2. Materials and method

The present in-vitro study was permitted by the ethical committee of King Saud University under (ethical approval number E-18-3345). The study followed the Check List for reporting In-vitro Study (CRIS) guidelines.

Sixty extracted non-carious, non-fractured, restoration free human molars were collected and stored in 0.01% of thymol solution for one week. Calculus, plaque, tissue remnants and other organic debris were removed prior to tooth preparation with the help of periodontal scaler (Sonic flex 2000, Biberach, Germany). The specimens were transferred to distilled water at 4°C until preparation.

All the sample were mounted vertically in acrylic resin (Meliodent, Kulzer, Hanau, Germany) within the segments of polyvinyl pipe (4 mm radius) equal to Cemento-Enamel Junction (CEJ) revealing only the clinical crown. To homogenize dentinal depth and to expose fresh dentinal tubules the buccal surface of all molars were ground to a depth of 2 mm with an area of 3 mm using Isomet saw (Buehler, Illinois, USA). The surfaces were polished with a 300–500 grit silicon carbide paper (Buehler, Illinois) on a rotary polishing machine (Aropol 2 V, Arotec) (250 Rpm) under water irrigation for 20 s. Specimen were randomly allocated into four groups (n = 15) according to the type of surface treatment.

Group 1: Dentinal surface of the samples were conditioned by ECL (Biolase-Waterlase I-Plus) power 4.5 W and frequency 30 Hz in a non-contact mode from a distance of 2 mm using tip MZ = 8, for a duration of 60 s. After the procedure, the specimens were bathed in artificial saliva (NeutraSal, Orapharma, North America LLC) for 60 s. The samples were rinsed dried using compressed air removing all moisture. A universal bonding agent (Tetric N-Bond Universal, Ivoclar-Vivadent) was applied and light cured (Bluephase G2, Ivoclar, Vivadent) for 10 s. BARM (Activa, Pulpdent Cooperation, Watertown, Massachusetts USA) was dispensed in a polyether rubber mould which was already placed on the dentin samples using a cement plunger and was light cured for 20 s. The moulds were removed carefully.

Group 2: Dentinal surface of the samples were conditioned by ECL (4.5 W and 30 Hz) in a non-contact mode from a distance of 2 mm for a duration of 60 s. The specimens were bathed in artificial saliva (NeutraSal) for 60 s. The samples were rinsed dried using compressed air removing moisture. Ketac conditioner (3M ESPE, Dental products, Seefeld-Germany) was used on the dentinal surface for 10 s and then washed with copious water for 10 s and air dried. The use of bonding agent and bioactive restorative material build-up was performed similarly as presented in group 1.

Group 3 (control group): Dentin was surface treated through conventional method i.e. etch and rinse. 37% phosphoric acid was applied for 15 s (Harward, Etch) and washed for 30 s. The samples were dried using compressed air removing all moisture. A universal bonding agent (Tetric N-Bond Universal, Ivoclar-Vivadent) was applied and light cured (Bluephase G2, Ivoclar, Vivadent) for 10 s. Bioactive restorative material (Activa, Pulpdent Cooperation, Watertown, Massachusetts USA) was dispensed and build-up was performed as explained earlier.

Group 4: All specimens received two applications of All-In-One (OptiBond, KaVo Kerr, West Collins, Orange, CA) with a brushing motion for 20 s each (As per manufacturer’s instructions). The adhesive was dried for 5 s and light cured (Bluephase G2, Ivoclar, Vivadent) for 10 s. A universal bonding agent (Tetric N-Bond Universal, Ivoclar-Vivadent) was applied and light cured (Bluephase G2, Ivoclar, Vivadent) for 10 s. Bioactive restorative material (Activa, Pulpdent Cooperation, Watertown, Massachusetts USA) build-up was performed.

All the specimens were kept in an incubator (Memmert Universal Oven, Germany) at 37°C in a humid environment for 2 days. Further, the samples were thermocycle between 5°C to 60°C for 8000 cycles (Applied Biosystems, Automated Thermal Cycler (ATC), CA, USA) for 45 s each, before assessing the shear bond strength (SBS) testing.

All specimens from each group were positioned in a universal testing machine (Instron 8500 Plus, Canton) for SBS testing. The samples were mounted in a metallic mould and were exposed to increasing load at a crosshead speed of 1 ml/min at the dentin resin interface. The shear strength that separated the assessed material was calculated. Similarly, 10 samples from each group were assessed for modes of failure (40X magnification) using a stereomicroscope (SZX7, Olympus, Hamburg, Germany) and classified into cohesive, adhesive and admixed failures.

Data obtained through bond strength testing was tabulated using statistical program for social science (SPSS version 21, Inc., Chicago, US). Normality of data obtained was assessed using Kolmogorov-Smirnov test. Means and standard deviations were compared using analysis of variance (ANOVA) and Tukey’s post hoc test at a significance level of (p < 0.05)

## 3. Result

The mean shear bond strength values and standard deviation of each group is presented in Table 1.

BARM bonded to dentin surface etched using conventional etch and rinse technique (group 3) exhibited SBS value of  $18.45 \pm 1.34$ . Similarly, the lowest bond strength was observed by self-etch regime bonded to BARM ((group 4– $16.09 \pm 0.81$ ). The bond strength among groups 1 (ECL + BARM), 2 (ECL + Ketac + BARM) and 3 (etch and rinse + BARM) were found to be comparable (p > 0.05). SBS of specimens in group 4 was significantly lower than specimens in all experimental groups p < 0.05). For bond strength values, analysis of variance (ANOVA) showed significant difference among the study groups (p < 0.01).

**Table 1**

Means and SD for bond strength values among study groups using ANOVA and Tukey multiple comparisons test.

Experimental groups	Mean (MPa)	SD (MPa)	Variance	P value <sup>†</sup>
Group 1 ECL + BARM	18.31 <sup>†</sup>	1.171	1.063	< 0.001
Group 2 ECL + Ketac + BARM	18.05 <sup>†</sup>	0.972	1.131	
Group 3 Conventional Etch and Rinse + BARM	18.45 <sup>†</sup>	1.34	1.214	
Group 4 Self-etch + BARM	16.09	0.81	1.217	

ECL: Er,Cr:YSGG laser, Bioactive restorative material: BARM.

<sup>†</sup> Significantly different from group 4 (Tukey multiple comparison test).

<sup>‡</sup> Showing significant difference among study group (ANOVA).

**Table 2**  
Modes of failure among study groups.

Study Groups	Adhesive (%)	Cohesive (%)	Admixed (%)
Group 1 ECL + BARM	30	–	70
Group 2 ECL + Ketac Conditioner + BARM	20	–	80
Group 3 Conventional Etch and Rinse + BARM	80	20	–
Group 4 Self-etch + BARM	60	40	–

ECL: Er,Cr:YSGG laser, Bioactive restorative material: BARM.

Frequency of mode of failure among study groups is presented in Table 2. Majority of specimen's surface treated with ECL exhibited admixed failures. Whereas, specimens in group 3 and group 4, adhesive type of failures were commonly observed. (Table 2)

#### 4. Discussion

The present study was based on the hypothesis that bond strength of BARM to dentinal surface conditioned with ECL will exhibit comparable outcomes to conventional (etch and rinse) dentin conditioning techniques. Remarkably, SBS of BARM when bonded to dentin treated with ECL and conventional technique (etch and rinse) was comparable, therefore the hypothesis was not rejected. SBS values were assessed using universal testing machine. In this test, interface between dentin and restorative material are subjected to force resembling stress. The method distributes the force homogeneously between dentin and tooth interface and follows strict consistency and standardization. Unlike tensile bond strength testing SBS demonstrates lower failure rates and accounts for providing relatable results [21,22].

Bonding to dentin is a technique sensitive procedure due to its composition, complex histological structure and moisture characteristic [23]. It is of utmost importance that a strong bond exists between different biomaterials and tooth structure for a predictive treatment outcome [24]. In the present study, the highest SBS value [18.45(1.45)] was exhibited by control specimens in which dentin was conditioned with traditional etch and rinse also termed as total etch technique [25,26]. Phosphoric acid at concentration of 35% or 37% is used for etching dentin followed by rinsing of the etched surface. This acid demineralizes the hydroxy appetite crystals, eliminates the smear layer, revealing collagen fibrils and funnelling the opening of dentinal tubules [25]. However, traditional etch and rinse has a drawback of being technique sensitive (dry or wet dentin), displays nano-leakage, increases number of clinical steps, is more prone to salivary contamination and increase the incidence of tooth sensitivity [24,26].

Dentin surface conditioned with ECL displayed statistically comparable bond strength to conventional etch and rinse conditioning. ECL at wavelength of 2790 nm is well absorbed by mineralized tissues and exhibits strong affinity to water and hydroxyapatite [27,28]. ECL harvests free demineralized surface more resistant to acid attack and causes minimum damage to the pulp and better gingival contouring [29,30]. Observing comparable bond strength in ECL irradiated samples, several authors have proposed numerous reasons in this regard. Better affinity of ECL with hydroxyapatite of dentin creating scaly, rugged appearance free from smear layer, micro-retentive tags on dentinal surface and prominent tubular and peritubular dentin pattern might have influenced better bond integrity [27,31]. Moreover, the bond of dentin with BARM is over exchange of ions through intermediary hybrid layer. It is believed dentin conditioned with ECL increases ionic exchange improving adhesion [32]. A study by Garbui et al., proclaimed that ECL as dentin conditioner does not alter dentinal collagen matrix generating a strong electrostatic force between the collagen and BARM [33].

It should be noted in the present study, polyacrylic acid conditioner (Ketac conditioner) was applied to dentin already conditioned with

ECL. Polyacrylic acid conditioner is recommended to remove smear layer and facilitate formation of a hybrid layer to augment the adhesive bond integrity [31]. However, application of polyacrylic acid conditioner failed to show significant improvement of SBS in the presence of surface pre-treatment using ECL. A possible explanation to this observation, is the influence of ECL on dental tubular patency and removal of smear layer resulting in a susceptible dentin surface for adhesive bonding (free of smear layer) [34]. Therefore, the authors do not recommend the clinical use of Ketac conditioner (polyacrylic acid) in the surface conditioning of ECL treated dentin for bonding of BARM.

A major concern in the authenticity of SBS tests are failure modes. Typically, ECL irradiation on dentin displayed admixed failures, suggesting SBS of BARM to dentin reaching highest level among these groups. Micro ablation and thermo mechanical impact of laser irradiation might be a hypothetical reason that have resulted in physico-chemical bond of BARM to the dentin surfaces.

It is worthy-of-note that specimens conditioned with self-etch conditioners ('all in one' adhesive) presented significantly lower bond strength to BARM in comparison to control and photobiomodulated samples. A possible explanation for these finding is the presence of weak acids in self etch primers, and their ability to compromise (thinner) hybrid layer and resin tag formation [35]. In addition, it is also reported that calcium and phosphate ions buffer the acid of self-etch adhesive limiting the depth of demineralization and compromising bond integrity and bond strength [35,36]. Therefore, it is an opinion of authors that photobiomodulation of dentin for BARM bonding is more effective than the self-etch bonding technique.

The bond strength values were relatively low, however a comparison of bond strength of BARM with conventional resin material is not available in the existing study. Therefore, further studies are recommended to compare resin materials with bioactive resins in the presence of ECL as surface conditioner. Moreover, this is an in-vitro investigation, therefore, based on these outcomes and parameters further clinical trials are recommended to validate the clinical significance of these conclusion.

#### 5. Conclusion

Within the limitations of the present study, conditioning of dentin with ECL irradiation for the adhesive bonding of BARM showed statistically comparable bond strength outcomes to conventional conditioning techniques. ECL irradiation is less technique sensitive and has the potential to be an effective conditioning alternative for dentin bonding to BARM.

#### Declaration of Competing Interest

The authors declare no conflict of interest

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