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EFFECT OF DIODE LASER AND WARM AIR DRYING ON THE SHEAR BOND STRENGTH OF LITHIUM DI-SILICATE TO DENTIN. AN IN-VITRO STUDY

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ABSTRACT

Objective: This study aimed to assess the effect of diode laser irradiation (980 nm) and warm air drying (50°C) on shear bond strength between Lithium di-silicate and human dentin using both Etch & Rinse (ER) adhesive, Adper Single Bond 2 and Self-etch (SE) adhesive, Single Bond Universal before adhesive polymerization. **Materials and methods:** One hundred and eight sound lower molars were sectioned perpendicular to the long axis to obtain flat dentinal surfaces. Specimens were divided into 2 equal groups (n=54): Group 1 (ER) and Group 2 (SE) according to bonding approach. Each group was subdivided according to dentin moisture elimination technique into 3 equal subgroups (n=18): Control (Co), Diode laser irradiation (L) and Warm air drying (W). All specimens were adhesively cemented to IPS e.max® CAD discs using RelyXTM Ultimate ClickerTM (3M ESPE) resin cement. Half of the samples were then subjected to pre-loading in a thermodynamic manner (C=9). Afterwards, all samples were tested for shear bond strength using computer-controlled material testing machine. **Results:** Data analysis was performed using three-way (ANOVA) (p< 0.05) followed by pair-wise Tukey's post-hoc tests. Three-way ANOVA showed significant difference between moisture elimination techniques in both groups, except ERNC. Also, for both groups, subgroup (W) had the highest shear bond strength values followed by (Co) subgroup and the least was (L) subgroup. **Conclusion:** Warm air drying for both bonding approaches increased shear bond strength of IPS e.max to human dentin and can be introduced to enhance bonding to dentin.

KEYWORDS: Dental adhesives, IPS e.max, Shear bond strength, Diode laser, Warm air drying.

INTRODUCTION

Adhesion of indirect restorations to dentin is considered a challenging procedure which affects the success rate of these restorations. Adequate bonding between tooth structure and the indirect restoration enhances marginal adaptation, and achieves strong reliable bond which will increase fracture resistance of both the restored tooth and the bonded indirect restoration ⁽¹⁾. Dentinal adhesion is obtained through hybridization process, in which chemical ionic bonding occurs between calcium in hydroxyapatite crystals and acid monomers in the adhesive system ⁽²⁻⁵⁾.

The aim of hybridization is to retain the restorative material or resin cement to dentin, thus preventing micro-leakage, marginal pigmentation, secondary caries, counteracting the contraction stresses due to polymerization shrinkage of cement or restorative material and finally, less invasive to dentin with less postoperative sensitivity ^(3,4,6).

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Etch & Rinse bonding approach still remains an acceptable choice for bonding indirect restorations⁽⁴⁾. Phosphoric acid etchant is applied before the adhesive, which partially removes the mineral content of the dentinal tissue and exposes the collagen mesh⁽⁷⁾. Over drying of dentin after washing out of the etchant will lead to shrinkage of collagen network, which prevents adhesive monomer diffusion in dentin and would eventually jeopardize hybridization process ⁽⁷⁻⁸⁾.

While in case dentin becomes over wet, phase separation between hydrophobic and hydrophilic components of adhesive will result in formation of globules and voids at the interface between adhesive and dentin surface⁽⁹⁾. In addition, excessive moisture will prevent complete polymerization of the adhesive monomer, which will be incorporated in the hybrid layer thus, leads to deterioration of bond strength between dentin and adhesive resin⁽¹⁰⁾. Volatile organic solvents are added to the primer and/or adhesive to partially facilitate water evaporation from the dentinal surface and to permit adhesive penetration into the moist collagen mesh in order to produce the hybrid layer ⁽¹¹⁾.

Single-step Self-Etch adhesives combine all three bonding steps into one single application to the tooth surface prior to cementation of the restoration^(2,5). The etching effect of these adhesives is due to the presence of acidic functional monomers which interact with the mineral content of dentin substrate, that creates a continuum by simultaneous demineralization and resin solvent penetration ⁽¹²⁾. Thus, Self-Etch adhesives should contain water and water-soluble hydrophilic monomers.

These monomers such as 2-hydroxyethyl methacrylate (HEMA), which dissociates water into hydrogen ions are required for effective demineralization and penetration of the hydrophilic dentin⁽¹³⁾. Methacryloyloxydecyl Dihydrogen phosphate (MDP) is a functional hydrophillic acidic monomer which have mild etching properties. MDP interact with calcium to produce stable non-soluble Calcium salts, which promote adhesion to the tooth structure⁽³⁾. The main problem of Self-Etch adhesives is their excessive hydrophilicity, therefore they attract water from dentin substrate which is intrinsically moist⁽¹⁴⁾. It has been reported that these adhesives act as semipermeable membranes after their polymerization; thus, moisture will diffuse from dentin substrate to the adhesive layer ⁽¹⁵⁾. This leads to accumulation of small water droplets between adhesive layer and (resin restoration or cement) especially if polymerization of the adhesive was delayed, which will eventually decrease bond strength ⁽¹⁶⁾.

Ideally, residual moisture and solvents should be eliminated completely from the demineralized dentin before polymerization for both adhesive approaches. However, complete evaporation is difficult to obtain and it depends on the properties of the solvent ⁽¹⁶⁾. One clinical approach was introduced in order to enhance dentin bonding, was using of a warm air stream that evaporates excessive solvent from the adhesive ^(17,18).

Several studies reported increase of shear bond strength to dentin after warm air drying. One invitro study analyzed the effect of warm air drying on shear bond strength of dentin/resin bond using different types of Self-Etch adhesives. Samples subjected to warm air drying had significantly higher shear bond strength than control samples⁽¹⁹⁾. Another in-vitro studies evaluated the effect of warm air drying on micro tensile resin/dentin bond strength. Samples bonded using ethanol and water-based, Etch & Rinse adhesives showed higher statistically significant bond strength values after warm air drying, compared to cold air-dried samples^(17,19,20).

A more recent approach was introduced to improve adhesion to dentin using laser. Gonçalves et al ⁽²¹⁾. suggested that Nd:YAG laser irradiation before the adhesive polymerization developed a substrate, in which both adhesive and dentin fuse together by the laser action. This substrate increased micro-tensile and shear bond strength values of

293

simplified dentin bonding systems (DBSs) ⁽²²⁻²⁴⁾. According to these authors, laser irradiation could possibly increase dentin bond strength by increasing penetration of DBSs into dentin ⁽²²⁾ or promoting the development of a substrate in which adhesive and dentin fuse together by the laser action. ⁽²³⁾. Diode laser also can be an alternative to Nd:YAG and it has near-infrared irradiation, due to its has more attractive usage and availability, such as lower size, weight, and cost ⁽²⁴⁻²⁵⁾.

Also, various in-vitro studies reported the increase of micro-tensile bond strength of resin dentin bond after diode laser irradiation (both 940 and 970 nm wavelengths). The bond strength values of dentin samples were significantly higher after laser application for both adhesive approaches ^(24,25). Another study showed significant effect of diode laser irradiation having (940 nm wavelength) on micro-tensile bond strength of resin/dentin bond using Etch & rinse approach ⁽²⁶⁾. Also, diode laser irradiation increased shear bond strength of resin/ dentin bond using Self-Etch adhesives ^(27,28).

Accordingly, the rationale of this study was to evaluate the capability of diode laser irradiation and warm air drying to remove dentin moisture before adhesive application using both Etch & Rinse and Self-Etch strategies as an attempt to enhance shear bond strength between IPS e.max and dentin. The null hypothesis of this study was that diode laser and warm air had no effect on shear bond strength using both adhesive approaches.

MATERIALS AND METHODS

Calculation of sample size

Sample size calculation was performed according to similar previous $study^{(24)}$. A sample size of (n=9) in each group had 80% power to detect a difference between means of 4.94 with a significance level (alpha) of 0.05 (two-tailed) and 95% confidence interval. In 80% (the power) of those experiments, the P value would be less than

0.05 (two-tailed) so the results would be deemed "statistically significant". In the remaining 20% of the experiments, the difference between means would be deemed "not statistically significant". Report created by GraphPad StatMate 2.00.

Dentin sample preparation

108 extracted human lower molars were gathered from periodontially affected patients in the outpatient clinic of Oral Surgery Department, Faculty of Dental Medicine, Al-Azhar University approved by ethical and scientific committees (602/3289). Average both buccolingual and mesiodistal of anatomical crowns were (9 \pm 1mm). Each crown was sectioned horizontally at the occlusal third, 2.5 mm depth from buccal cusp tip perpendicular to the long axis of the tooth in order to to expose the dentinal surface using diamond saw (Isomet 4000 precision cutting microsaw, Buehler, USA) ^(21,23).

A custom-made cylindrical counter split mold having dimensions (inner diameter 15 mm and height 20 mm) was machined milled. The mold was filled by self-cured polymethyl-methacrylate (PMMA) acrylic resin. Then the dentin specimen was immersed centrally by the aid of dental surveyor (Paraskop®M. Bego, Bremer, Germany) parallel to the wall of the mold. This process was repeated for each specimen. Exposed dentin surface was placed on a polishing machine (EXAKT 400 CS, EXAKT Technologies, Oklahoma City, OK) at speed 300 rpm and wet polished using 600 grit silicon carbide paper for 60 seconds to standardize the smear layer thickness ⁽²⁹⁾.

IPS e.max discs preparation

108 square-shaped IPS e.max discs having dimensions 5×5 mm and 2 mm thickness were sectioned from IPS e.max CAD blocks using same diamond saw. A digital caliber (Pachymeter, Electronic Digital Instruments, China) was used to verify the exact dimensions of each disc. Then all surfaces of the discs were also wet polished using 400, 600, 800 and 1000 grit silicon carbide papers at using same polishing machine also using speed 300 rpm ⁽²⁹⁾.

IPS e.max discs were then placed in the EP 3000 Press furnace (Ivoclar, Schaan, Liechtenstein) for 30 minutes at temperature 850°C, until crystallization took place according to manufacturer instructions. The bonding surface of each disc was etched using 10% Hydrofluoric acid (IPS® Ceramic Etching Gel, Ivoclar, Vivadent AG, Liechtenstein) for 20 seconds, then air-dried and silanized using silane coupling agent (Monobond®Plus, Ivoclar, Vivadent AG, Liechtenstein.

Adhesive cementation

Specimens were divided according to bonding approach to dentin into two equal groups (n=54) into Etch & Rinse (ER) and Self-Etch (SE). As for (ER) group, each dentin specimen was etched using 35% phosphoric acid etchant (3M Dental Products, St. Paul, MN, USA) for 15 seconds, then surface moisture was blotted using cotton pellet. Then, two layers of Adper Single Bond 2 (3M ESPE) were applied. In (SE) group, Single bond Universal (3M ESPE) self-etch adhesive was applied directly on dentin specimens for 20 seconds. Both adhesives were used according to manufacturer instructions.

Specimens were further subdivided into three equal subgroups (n=18) according to the elimination technique of excessive adhesive wetness into Control (Co), warm air drying (W) and laser irradiation (L). In (Co) subgroup dentin specimens were dried using oil free compressed air for 5 seconds after application of both adhesives according to manufacturer instructions. While in (W) subgroup, warm air drying of specimens was performed using warm air tooth dryer device (YS-AD-A, TDOU, China), instead of air drying at a distance 2 cm away from dentin surface for 10 seconds. The temperature of warm air was 50°C and the constant air speed was 30 Mph ^(18,19).

In (L) subgroup, dentin specimens were irradiated with 980 nm diode laser (Wiser, Doctor Smile, Italy) using bio-stimulation tip 5 mm diameter at a distance 1 mm away from dentin for 10 seconds and the device was set to (CW) continuous wave with average power 1 watt ⁽³⁰⁾. (ER) group specimens were light cured for 10 seconds using LED unit 650 mW/ cm2 (LED-D, Woodpecker, Guilin, China) for each coat of the adhesive, while (SE) group specimens were light cured for 20 seconds for the single coat of adhesive following manufacturer instructions.

Afterwards, IPS e.max discs were adhesively cemented to dentin specimens using resin cement (Rely X Ultimate Clicker, 3M, ESPE, USA). A specialized loading device having static load of 49 N was applied on each specimen after cementation for standardization. Each of four surfaces of the specimens was light cured for 20 seconds following manufacturer instructions and then stored in distilled water until testing was performed.

Scanning Electron Microscope (SEM)

An additional specimen from each subgroup (n=12) was selected for SEM analysis in order to analyze the intact hybrid layer and resin infiltration within the dentin. Specimens cemented to IPS e.max discs, were sectioned longitudinally in bucco-lingual direction within the long axis of the tooth into two halves using the same diamond saw mentioned earlier. Sectioned samples (n=24) were observed by scanning electron microscopy Jeol JSM-5510LV SEM (Jeol, Tokyo, Japan) using high vacuum mode and voltage (10-15) kilo volts. The SEM magnification used was 1000 x.

Thermo-mechanical aging

Each subgroup was further subdivided to according to thermo-mechanical aging into two equal classes (n=9), in which class (C) specimens were thermo-mechanically cycled and class (NC) weren't aged. Half of the specimens in each subgroup were mounted on a programmable logiccontrolled equipment multimodal chewing simulator (ROBOTA, AD-TECH TECHNOLOGY CO., LTD., GERMANY), integrated with servo-motor (ACH-09075DC-T, AD-TECH TECHNOLOGY CO., LTD., GERMANY) to generate horizontal and vertical occlusal movements simultaneously, which simulated thermodynamic conditions in oral cavity at a temperature between 5 C° and 55 C°. A weight of 5 kg, which is equivalent to 49.03 N of chewing force was exerted. The test was repeated 10000 times similar to the previous study ⁽³¹⁾.

Shear bond test

Specimens were mounted on Instron material testing machine (Industrial Products, Norwood, USA) with a loadcell 5 kN. Data were recorded using Bluehill Lite software (Instron Instruments). The load required to de-bond each IPS e.max disc and dentin specimen was recorded and calculated in Megapascals (MPa). A custom-made metallic holding device was fabricated in order to support each specimen during shear bond testing. It consists of two parts, fixed base portion on which the resin block is seated and the upper movable portion which is attached to a fixed base through two metallic rods. The two parts are surrounded by a spring wire, that is fixed by tightening screws, which control their compressibility, figure (1).

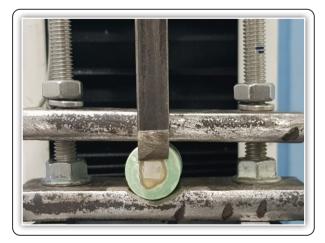


FIG (1) Dentin specimen subjected to shear bond test

Statistical analysis

Data analysis was performed in several steps. Initially, descriptive statistics for each group results. Three-way ANOVA followed by pair-wise Tukey's post-hoc tests were performed to detect significant effect of variables (moisture elimination technique, bonding approach and thermo-mechanical aging). One-way ANOVA and student t-test was done between groups and subgroups. Chi square was carried out between failure modes. Sample size (n=9) was large enough to detect large effect sizes for main effects and pair-wise comparisons, with the satisfactory level of power set at 80% and a 95% confidence level. Statistical analysis was performed using Graph-Pad InStat statistics software for Windows (www.graphpad.com). P values ≤0.05 are statistically significant in all tests.

RESULTS

Three-way ANOVA showed significant difference between moisture elimination techniques in the tested groups, except ERNC. Also, for both groups, subgroup (W) had the highest shear bond strength values followed by (Co) subgroup and the least was (L) subgroup. Descriptive statistics showing mean values and standard deviation of shear bond strength test results measured in mega-Pascal (MPa) as function of moisture elimination technique and bonding approach before and after thermo-mechanical aging were summarized in table (1).

SEM analysis

After SEM analysis of (SE) group as seen in figure 4(a), dentin/resin interface of (Co) subgroup showed intact hybrid layer with no surface alteration. While in figure 4(b), (W) subgroup revealed excellent adaptation with smaller interfacial gap and the formation of a transitional hybrid layer between the adhesive and dentin was evident. As regards to (L) subgroup, recrystallization of dentinal tubules and melting of surface dentin surface occurred as seen in figure 3(c).

Variables -		ER group		SE group		Statistics
		NC	С	NC	С	P value
Moisture elimination technique	(L) subgroup	11.19 ^B _a ±3.14	9.914 ^a _a ±1.03	12.06 ^B _a ±2.39	9.59 ^B _a ±2.42	0.1418 ns
	W) subgroup	15.81 ^A _{ab} ±3.77	11.29 ^A _c ±3.17	16.52 ^A _a ±5.51	13.11 ^A _{bc} ±2.19	0.0249 *
	(Co) subgroup	15.09 ^A _a ±3.62	11.5 ^A _b ±3.03	15.01 ^A _a ±2.18	12.94 ^A _{ab} ±0.96	0.0177*
Statistics	P value	0.0256*	0.3877 ns	0.0499*	0.001*	

TABLE (1) Shear bond strength test results (Mean±SD) as function of moisture elimination technique and bonding approach before and after thermo-mechanical aging

Different superscript large letter in the same column indicating statistically significant difference (p<0.05) *; significant (p<0.05). Different subscript small letter in the same row indicating statistically significant difference (p<0.05) ns; non-significant (p>0.05)

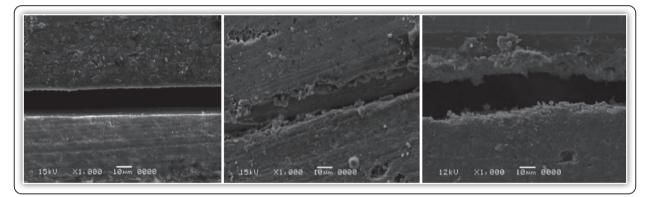


FIG (2) Representative SEM photomicrographs of the resin/dentin interface magnification ×1000.

DISCUSSION

IPS e.max (Ivoclar, Vivadent, Germany) was used in this study, which consists of lithium disilicate, that has diverse processing techniques. Lithium dislicate has both improved mechanical properties and high esthetic properties after adhesive cementation ⁽²⁾. Also, adhesive cementation was used in this study which improves fracture resistance, retention and marginal adaptation ⁽⁶⁾. Therefore, this study aimed to achieve stronger adhesion between indirect restoration and dentin, which is considered a critical issue for clinical success ⁽³⁾.

Warm air tooth dryer was used in this study to warm the adhesive before polymerization up to 50°C. Warm air was applied on to dentin surface to increase the temperature of the adhesive. This procedure was performed in same manner as several previous studies ⁽¹⁷⁻²⁰⁾. During function, restorations are exposed to thermal stresses, water sorption and micro-leakage. Half of specimens of each subgroup in this study were subjected to thermo-mechanical aging, before testing in order to simulate oral cavity conditions. The number of thermal cycles applied, was (10000) which was equivalent to one year of clinical performance ⁽³¹⁾.

It was shown that, laser wave length determines its absorption rate and its interaction with tissues⁽³²⁾. Wavelength 980nm was chosen in this study, because it has higher absorption in water than other wavelengths, causing less heat generation to the pulp during clinical application. It was also shown that, increase of pulpal temperature more than 5.5°C above room temperature would eventually cause irreversible pulpitis. Therefore, a power level 1 watt was chosen in this study to avoid excessive heat generation and occluding of the dentinal tubules and as well. 5 mm diameter bio-stimulation tip was used to ensure complete diode laser irradiation of dentin specimens ⁽³¹⁾. The tip was placed 1 mm away from dentin surface for 10 seconds to avoid carbonization due to overheating of dentin ⁽³³⁾.

According to the results of this study, the null hypothesis was partially accepted, in which warm air drying had significantly increased shear bond strength of both groups except ERNC, while dentin laser irradiation (L) subgroup, had significantly the lowest shear bond strength values except for ERNC. Shear bond strength test was chosen in this study, since it is the most common method to evaluate bonding of dental materials and can be easily compared with other previous studies ⁽³⁴⁾.

Shear bond strength values of this study were similar to a previous study which recorded results between 10 and 50 MPa⁽³⁵⁾. As for (SE) group, warm air drying had increased shear bond strength for (W) subgroup. This study agrees with several previous studies which reported that increase of adhesive temperature rises the kinetic energy of the molecules in the solvent (ethanol) which allowed further evaporation and thus improved adhesive polymerization and enhanced the bond strength^(8,20,21).

Other studies that agreed with this study stated that, rise of adhesive temperature decreases the solvent viscosity and improves wettability of adhesive on dentin surface⁽¹⁷⁾. Representive SEM images for (W) subgroup showed excellent adaptation of resin /dentin interface as well figure 3 (b).

Other studies were consistent with our study showed that warm air drying didn't increase microtensile bond strength of (ER) adhesive using (Adper Single Bond 2; 3M ESPE) ^(17,20). A study which partially agreed with this study, explained that the main problem with HEMA hydrophilic monomer is its high affinity to water which would decrease its vapor pressure and it would retain moisture forming hydrogel. Therefore, it would be difficult to eliminate water even by warm air drying for the HEMA containing adhesives ⁽¹⁹⁾.

Shear bond strength values of (L) subgroup were the lowest which agrees with previous studies ^(26,30). Subgroup (L) had less values than subgroup (Co) in general which could be due to partial blockage of dentinal tubules⁽³⁰⁾. While, recordings of this study didn't match with other studies that reported increase of bond strength after laser irradiation. However, these studies recorded immediate bond strength and didn't apply thermomechanical aging on the specimens ⁽²¹⁻²⁵⁾.

As observed in SEM images of subgroup (L) there were surface changes, which were mostly due to thermal effect of diode laser on dentin. Several studies showed that during interaction of laser with the tooth structure, photon energy is converted to heat. The photo-ablative effects will induce modifications of dentin surface including recrystallization and melting of dentin ⁽³³⁾, figure 4(c).

(SE) group values were slightly higher than (ER), which could be attributed to the presence of (MDP) and polyalkenoic acid copolymer in (SE) adhesive ⁽³⁾. MDP contains phosphate and carboxylate monomers which ionically bond to calcium in the hydroxyapatite crystals and interact with it to produce stable non-soluble Calcium salts, which improve adhesion to dentin. Mild dentin etching, preserves hydroxyapatite in the hybrid layer which act as a receptor for chemical bonding and support the collagen matrix to avoid its collapse and thus bond degradation ⁽²⁶⁾. Polyalkenoic acid decreases water sorption and thus enhance shear bond strength to dentin ⁽³⁾.

Also, another previous study reported that, Selfetch adhesion had comparable shear bond results to Etch & Rinse, using Single Bond Universal adhesive after aging for 6 months ⁽³⁶⁾. The limitation of this study is that it wasn't identical to the clinical situation, in which vital dentin has its own inherent moisture content ^(8,29). Accordingly, further clinical research is highly recommended to obtain consistent outcome.

CONCLUSION

The following can be concluded, within limitations of this study

- Despite the fact that warm air drying results were higher in range, the impact of this approach wasn't noticeable compared to traditional air drying.
- 2. Laser irradiation using the above-mentioned parameters and wavelength didn't improve bond strength to dentin.

RECOMMENDATIONS

Warm air drying can be introduced as a useful protocol to enhance the shear bond strength between IPS. emax restorations and dentin. Further research using other laser wavelengths and parameters could improve bond strength.

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