



ORIGINAL ARTICLE

Shear-bond strength of different Self-Etching adhesive systems to dentin with or without laser irradiation before photopolymerization (A comparative Study)



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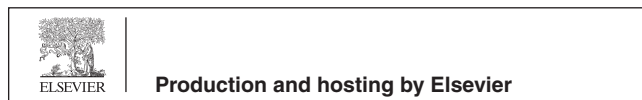
Adhesive System;
Dentin;
Diode laser;
Shear Bond Strength;
Self-etch

Abstract This study aimed at comparing shear-bond strength (SBS) of different self-etching adhesive systems (Clearfil S³ Bond Plus, G-Premio BOND and IBond) to dentin without or with diode-laser irradiation before photo-polymerization and to determine the effect of storage and thermo-cycling on SBS of adhesive systems. Methods: The buccal surface of 84 extracted maxillary premolars was grounded to create flat surface. The specimens were allocated into 3 groups (n = 28) depending upon the adhesive systems, then each group was divided into two sub-group (I, II) (n = 14). After the placement of respective adhesive systems on the flat surface, adhesive system in group I was photo-polymerized immediately, while in group II, the adhesive systems were exposed to diode-laser before photo-polymerization. Composite cylinder (4 mm in diameter and 2 mm height) was built on the flat surface of each specimen. Then group I and II were divided into two sub-groups (n = 7) according to the storage time and thermo-cycling (1 day without thermo-cycling or 72 days with thermo-cycling) then all the specimens were stored in distilled water. The SBS was measured at the end of storage period. ANOVA, Duncan's Multiple Range Test and independent *t*-test " $P \leq 0.05$ " were used for data analysis. Results: G-premio BOND showed the highest mean value of SBS followed by Clearfil S³ Bond plus without significant difference between them, while IBond revealed the least mean value. Laser irradiation had positive effect on the bond-strength of all tested adhesive systems. The results also showed that the storage with thermo-cycling had negative effect on the bond-strength in groups without laser irradiation for all tested adhesive systems, while for groups with laser irradiation, the reduction in the bond-strength of

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all tested adhesive systems was not significant. Conclusion: Diode-laser application prior to photopolymerization of self-etch adhesive systems significantly increased the bond-strength to dentin and can increase the durability of composite adhesion.

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1. Introduction

It has been approximately-seven decades since Buonocore (1955) and later Nakabayashi et al. (1982) founded the concept responsible for the most popular branch in operative dentistry, dental adhesion. Ever since, dental composite restorations have acquired extreme popularity as they were considered a promising tooth-resembling materials (Aminoroaya et al., 2021). Theoretically, dental adhesion eradicates marginal gaps, diminishes micro-leakage and avoids secondary caries around composite materials, due to its capability of creating an intimate contact between composite restoration and tooth structure (Nicholson, 1998).

The quality of dental adhesion is dependent upon the performance of adhesive systems being utilized, but unfortunately that performance is still compromised when it is related to dentin. Due to complex heterogenetic nature of dentin, hydrolysis of both resin and collagen fibrils within the adhesive substrate eventually happens (Breschi et al., 2008). Considerable literature has shown that self-etching adhesive-systems (6th, 7th and 8th generations) outperformed etch and rinse adhesive systems (Koshiro et al., 2004; Van Meerbeek et al., 2011; Masarwa et al., 2016; Zhang et al., 2016).

The elimination of additional etching step eradicates the dilemma of over-dryness or over-wetness of dentin after etchant rinsing (Hashimoto et al., 2011; Van Meerbeek et al., 2011). Furthermore, resin monomer incorporation with the etchant also buffers the etchant acidity, avoiding the complications associated with the usage of strong acid (Pashley, 1992). Hydrolytic and enzymatic degradation of collagen fibrils (Pashley et al., 2004; Zhang and kern, 2009), nanoleakage (Tay et al., 2002a) and dentin hypersensitivity (Van Meerbeek et al., 2011) have all related to adhesive systems that incorporate strong acid ($\text{pH} < 1$). In that context, self-etch adhesive systems are categorized according to the pH value into mild “pH of around 2.5” moderate “pH about 1” and strong “pH < 1” (Sofan et al., 2017).

Nevertheless, other studies revealed that water related deterioration of bond was also evident even with self-etch adhesive systems with mild pH (Tay et al., 2002b; Takahashi et al., 2002). The presence of HEMA (2-hydroxyethylmethacrylate), ethanol and water within the composition of self-etch adhesive systems have been blamed for the deterioration of hybrid layer created by simplified-adhesive systems (Van Landuyt et al., 2010).

Relatively new advance in restorative dentistry was the incorporation of dental lasers in restorative dentistry. In fact, the use of dental lasers could be traced back to 1999, when Gonçalves et al. claimed that Nd:YLF laser irradiation before photopolymerization of etch and rinse adhesive system could increase the SBS of adhesive system to bovine dentin. The researchers proposed that photo-thermal effect of laser might result in the development of new substrate in which

hydroxyapatite crystals have melted and recrystallized in the presence of adhesive monomer (Gonçalves et al., 1999). Ever since, the incorporation of laser irradiation prior to adhesive system polymerization has been investigated by several researchers and has been theorized that it might increase depth of penetration of adhesive system (Franke et al., 2006), promote solvent evaporation (Batista et al., 2015) and increase degree of conversion (Brianezzi et al., 2017). Heating of resin-based materials has been confirmed to enhance their mechanical properties and adhesion ability (Vale et al., 2014; Lopes et al., 2020), which may justify the effectiveness of laser in the same manner.

Diode laser, owing to its affordability, portability and its small size, becomes popularly used by dental professionals and investigated by researchers. Ramachandrani et al., 2019, concluded that SBS was significantly improved when self-etch adhesive system was irradiated with diode laser (pulsed mode) prior to photopolymerization. In addition, El-Hakim et al., 2019, concluded that diode laser irradiation improved the adhesion of self-etch adhesive system to cemented composite blocks. However, long-term effect of 940 nm diode laser irradiation on dental adhesion was not previously investigated.

The aims of this research were to *in-vitro* compare shear-bond strength (SBS) of different self-etching adhesive systems (Clearfil S³ Bond Plus, G-Premio BOND and IBond) to human dentin with or without 940 nm diode laser irradiation used in continuous mode before photopolymerization and to determine if the SBS of these adhesive systems would be affected by storage and thermo-cycling or not. Hence, the null hypotheses stated that there would be no statistically significant difference in SBS of tested adhesive systems, 940 nm diode-laser irradiation had no effect on SBS of all experimental groups and finally storage and thermo-cycling had no significant influence on SBS of the all experimental groups.

2. Materials and methods

In the current experimental study, freshly extracted eighty-four permanent maxillary human premolars, that were extracted for orthodontic treatment, free from caries, cracks, previous restoration and cavities were selected to be investigated. Once they were extracted, the teeth were disinfected by immersing them in thymol solution “0.1 %” at room temperature for one week (Kasraei et al., 2019). Any soft tissues remnant was removed by utilizing dental hand scaler (John-Quayle/England) and the buccal surfaces were polished by non-fluoridated pumice (Bilkim Ltd/ Turkey). They were examined under stereo microscope (20X) (HAMILTON, ALTAY/ Italy) to ensure that they met the inclusion criteria of the research and kept in distilled water till the experiment. All the teeth were mounted vertically in cold cure acrylic resin (Hiflex-RR/ India) and PVC mold to the level of cement-enamel junction and with the long axis of the tooth perpendicular to the

floor. The buccal surface was grounded by diamond disc and high speed hand-piece to expose the dentin, which was verified by visualizing the prepared surface under a stereo microscope (20X). The exposed dentin surface was wet sanded by (600 grit) water proof silicon-carbide paper (King spor/ Germany) to obtain standard smear layer (Kasraei et al., 2019). The buccal surface was covered with nail polish except for 4 mm diameter window at the center of the buccal surface to standardize the area of treatment.

2.1. Samples distribution

The specimens were randomly allocated into 3 equal groups (n = 28) according to self-etch adhesive system that was utilized as follows:

Group A: G- Premio BOND was utilized.

Group B: Clearfil S³ Bond plus was utilized.

Group C: I BOND was utilized.

The characteristics and the manufacturing information of the utilized adhesive systems are registered in Table 1.

Every group was randomly subdivided into 2 subgroups (n = 14) based on pre-photopolymerization diode-laser application as follows:

Subgroup I: The adhesive system was photopolymerized by using LED curing device (woodpecker, China.) with 1200 mW/cm² power density, standardized distance of 1 mm between the sample and the tip of curing unit (Kasraei et al., 2019) and according to manufacturing instructions of each adhesive system.

Subgroup II: After the application of the adhesive system and prior to photopolymerization, the adhesive system was subjected to 940 nm diode-laser radiation (Epic 10; Biolase; Irvine, California, USA) with continuous wave mode, 1 W power, 400 μm tip size (EZ tips, Biolase, Irvine,

California, USA) (Kasraei et al., 2019). The diode laser spot size was 0.36 mm² and the distance between the laser tip and adhesive system was 1 mm, which was standardized with the aid of milling machine (Bio art 1000 Max, Brazil.) and condensation silicon impression material (DUROSIL L, Germany) (Fig. 1) that held the laser hand-piece during irradiation (Ali and Sulaiman, 2022). The tested area was irradiated with free scanning motion for 3 times during the 15 seconds of irradiation period. The surface area of the irradiated zone was 12.56 mm², total energy was 15 J, power density was 275 W/ cm², while energy density of diode-laser irradiated area was 39.8 J/ cm². After that, the adhesive system was photopolymerized in same manner of subgroup I.

After adhesive polymerization, the composite material (clearfil majesty, Kuraray Noritake Dental Inc, Tokyo, Japan) was applied with aid of rubber mold that had central hole (4 mm diameter and 2 mm height) and condensed against the adhesive system to form composite cylinder (4 mm diameter and 2 mm height). The polymerization was accomplished by LED curing unit with 1200 mW/cm², 1 mm distance between the composite and LED tip and for 40 s during which the mold was covered with celluloid strip to prevent oxygen inhibition surface layer (Khalid et al., 2018). The power density was calibrated at regular intervals (every 5 exposures) by using LED light tester.

Both subgroup I and subgroup II were further subdivided according to storage time and thermo-cycling into two groups (n = 7):

Group 1: The specimens were stored in distilled water at 37 °C for 24 h before shear -bond strength evaluation.

Group 2: The specimens were stored in distilled water at 37 °C for 72 days (Al-Chalabi et al., 2022) and then subjected to thermo-cycling of 1000 cycles between (5–55) °C with a transfer time of 5 sec and dwell time of 60 sec in dis-

Table 1 The characteristics and manufacturing information of adhesive systems.

Materials	Manufacturer	Type	Composition	Mode of application
Clearfil S ³ Bond plus	Kuraray Noritake; Dental Inc.; Tokyo; Japan	7th generation, self-etch adhesive	HEMA, 10-MDP, Bis-GMA, silanated colloidal silica, water, ethanol, camphoroquinone pH = 2.7	Dry the dentin surface Apply the adhesive for 10 s Air blow for 5 s Cure for 10 s
I BOND	Kulzer, Hanau, Germany	7th generation, Universal self-etch adhesive	UDMA, 4-META, gluteraldehyde, water, acetone, photo initiator, stabilizer pH = 2.2	Dry the dentin surface Apply the adhesive and agitate for 20 s Evaporate the solvent by air flow for 5 s to 10 s Apply second layer if the dentin surface does not appear glossy Cure for 10 s
G- premio BOND	GC, Tokyo, Japan	8th generation, Universal self-etch adhesive	10-MDP; 10-MDTP; 4MET, methacrylate acid ester; acetone; distilled water; silica; photo-initiator pH = 1.5	Apply the adhesive, Air dry instantly Photopolymerized for 10 s

HEMA: 2-hydroxyethylmethacrylate, 10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: Bisphenol-A-diglycidyl methacrylate, UDMA: Urethane dimethacrylate, 4-META: 4-methacryloxy ethyl trimellitate anhydride; 4-MET: 4-methacryloxyethyl trimellitic acid; 10-MDTP: 10-Methacryloyloxydecyl dihydrogen thiophosphate.

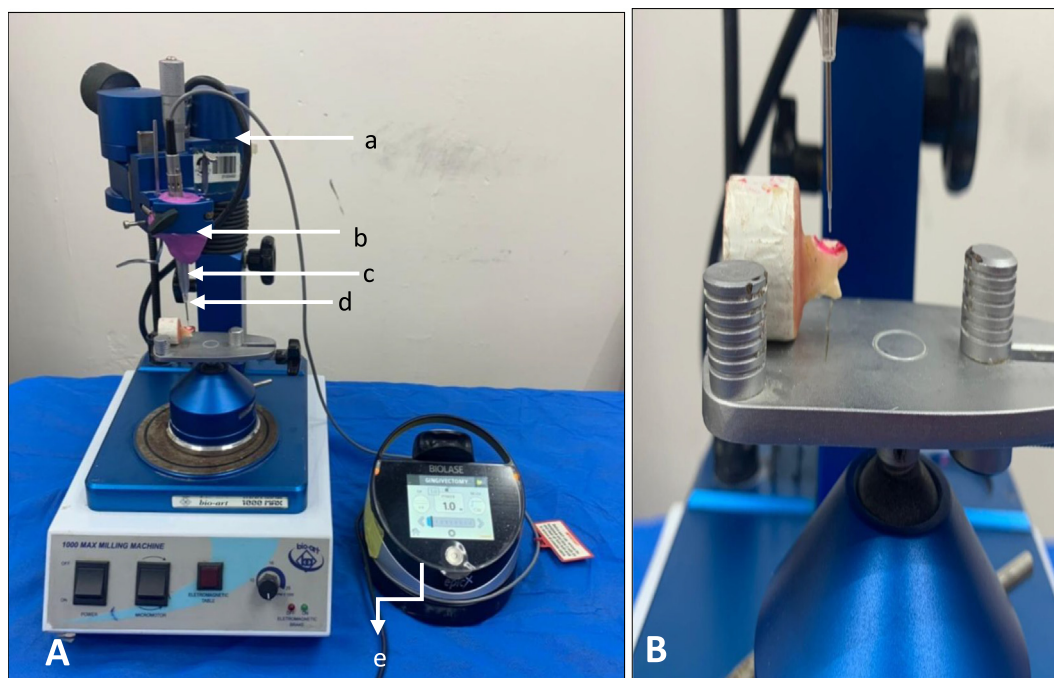


Fig. 1 (A) Diode laser hand-piece stabilized by milling machine and condensation silicon impression material. a: Milling Machine, b: Condensation silicon impression material, c: Laser hand-piece, d: Laser tip, e: Diode laser device. (B) Zoomed in photo of the specimen.

tilled water by utilizing thermocyclare machine (USA) (Kasraei et al., 2019).

2.2. Shear-bond strength testing

After storage time, each sample was investigated individually for the shear-bond strength of the composite by utilizing universal testing machine (Gester, Fujian, China) with cross-head speed of “1mm/min” (Kim et al., 2022). Each specimen was fixed on the lower part of the machine so that the chisel rod was applied vertically to dentin-composite interface and the test continued till the failure in bonding. The values of shear-bond strength were expressed in MPa (Newton/mm²) by dividing the failure load by the bonded surface area (Hussein and Al-Shamma 2019).

2.3. Failures mode

A stereo microscope was used to examine the debonded surface of each specimen at magnification “40X” to determine the mode of failure. The failure mode was categorized into one of the following: 1- cohesive within the tested material. 2- adhesive at dentine-tested material interface. 3- mixed both adhesive and cohesive failures (Raji et al., 2022).

2.4. Statistical analysis

Shapiro-Wilk and Levene tests were utilized in order to assess the normal distribution and the homogeneity of variances of the data. According to the results, One Way Analysis of Variance and Duncan’s Multiple Range Tests at “ $P \leq 0.05$ ” were performed in order to determine which adhesive-systems gave

the best result. For the evaluation of the effect of diode-laser application and for determination of the effect of storage and thermo-cycling with and without laser application on the bond strength of these adhesive-systems, independent samples *t*-test was performed. The data were analyzed by utilizing (IBM SPSS Statistical program 24.0).

3. Results

The results showed that there was a statistically significant difference in the bond strength at “ $p \leq 0.05$ ” among the tested adhesive-systems. G-premio BOND showed the highest mean value of bond strength followed by Clearfil S³Bond plus with no statistically significant difference between them. However, both of them showed significant difference from I Bond, which revealed the least mean value of bond strength as shown in Fig. 2. The findings also revealed that diode-laser application had a significant positive effect on the bond strength of G- premio BOND, Clearfil S³ Bond plus and I Bond as shown in Fig. 3. The analysis of the results also showed that the storage accompanied by thermo-cycling had significant negative effect on the bond strength of groups without diode-laser application for all tested adhesive-systems, while for groups with laser-application, the reduction in the bond strength of the all tested adhesive-systems did not reach the level of significance as shown in Table 2.

The percentages of failure mode are presented in Table 2. Adhesive failure had the highest percentage in tested groups without laser application, without storage and without thermo-cycling. While after diode laser application the percentage of mixed failure increased even after storage and thermo-cycling. Representative of failure modes are illustrated as seen under stereo microscope in Fig. 4.

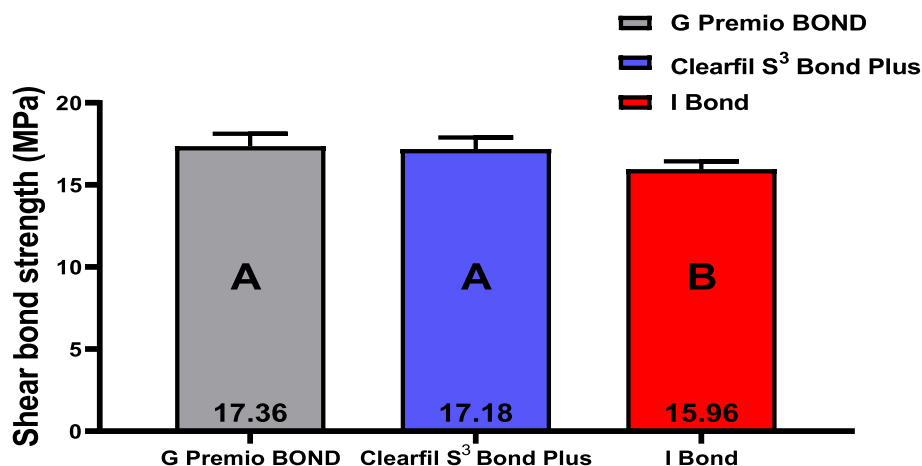


Fig. 2 Bars chart comparing between the mean value of shear bond strength of the tested adhesive-systems with no laser-application, no storage and no thermo-cycling. Different letters indicate a significant difference at $p \leq 0.05$.

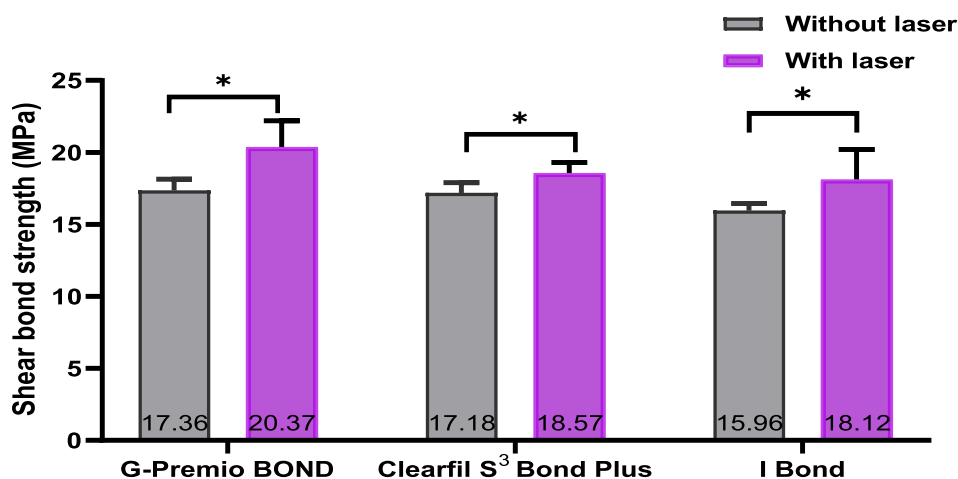


Fig. 3 Bar chart illustrates the effect of laser-application on shear bond strength of the tested adhesive-systems. * indicates significant difference at $p \leq 0.05$.

4. Discussion

This study was designed to compare SBS of three types of self-etching adhesive systems (Clearfil S³ Bond Plus, G-Premio BOND and, I Bond) to human dentin and to determine diode laser radiation effect, which was applied on these self-etching adhesive system before photopolymerization, on the SBS. In addition to determine the effect of storage and thermo-cycling on the bond strength of these adhesive systems. Based on the outcomes of the study, the null hypotheses were rejected.

In the current study, G-Premio BOND showed the greatest value of SBS followed by Clearfil S³ Bond Plus, while I Bond registered the least reading. A possible explanation may be belonged to the chemical components of the bonding systems which directly affect the bonding capability. Both G-Premio BOND and Clearfil S³ Bond Plus contain functional acid monomer, 10 - MDP “10 -thachryloyloxydecyl dihydrogen phosphate”, while I Bond contain 4 - META as a functional group that showed lower bonding ability than 10 -MDP-containing adhesives (Khosravi et al., 2009; Sánchez-Ayala et al., 2013). The 10 - MDP monomer provides acidity that

leads to etch the dentin surface allowing others components in the adhesive system to penetrate inside the demineralized dentin (Oshida et al., 2004). In addition to that, 10 -MDP owns the capacity for bonding chemically with the hydroxyl-apatite(HA) in the enamel and dentin forming 10 -MDP - Ca salts. In self-etch mode, the remaining HA which left over around the collagen fibers acts like receptors for chemical reaction with 10- MDP and participates later to bonding quality (Muñoz et al., 2015).

This result coincides with the finding of Yoshihara et al., 2018 who compared etching efficiency of self-etching monomers (phenyl -P, 4 -META, GPDM, 6 -MHP, MTEGP, and 10 -MDP) and found that 10 -MDP had higher etching potential than other tested monomer and only 10-MDP created Ca salts, indicating that 10 -MDP released more Ca from HA than was measured by 4 -META. The outcomes of this study is also in line with the finding of El Sayed et al., 2015, as they found that I Bond gave lower result when compared to Clearfil S³ Bond Plus. Furthermore, Asgartooran et al., 2020 concluded that the SBS of G- Premio BOND was higher than Clearfil S³Bond Plus.

Table 2 Independent samples *t*-test for the effect of the storage and thermo-cycling with and without laser treatment, values are expressed in MPa with failure mode percentage.

Self-adhesive system	Treatment	N*	Mean ± Std. Deviation	<i>t</i> -value	Sig**	Failure mode percentage A/M/C
G-Premio BOND	Without laser	7	17.36 ± 0.77	2.719	0.025	(85,7/14,3/0) %
	Without laser with storage and thermo-cycling.	7	16.48 ± .36			
Clearfil S ³ Bond Plus	Without laser	7	17.18 ± 0.72	2.269	0.043	(71,4/28,6/ 0) %
	Without laser with storage and thermo-cycling.	7	16.18 ± 0.90			
I Bond	Without laser	7	15.96 ± 0.49	5.520	0.000	(71,4/14,3/ 14,3) %
	Without laser with storage and thermo-cycling.	7	14.45 ± 0.53			
G-Premio BOND	With laser	7	20.37 ± 1.83	0.869	0.402	(42,9/57,1/0) %
	With laser with storage and thermo-cycling.	7	19.62 ± 1.37			
Clearfil S ³ Bond Plus	With laser	7	18.57 ± 0.95	2.031	0.073	(28,6/71,4/ 0) %
	With laser with storage and thermo-cycling.	7	16.96 ± 1.86			
I Bond	With laser	7	18.12 ± 2.08	2.154	0.062	(57,1/42,9/0) %
	With laser with storage and thermo-cycling.	7	16.25 ± 0.96			

N*: Number of Samples, ** $p \leq 0.05$. A: Adhesive, M: Mixed, C: Cohesive within the adhesive.

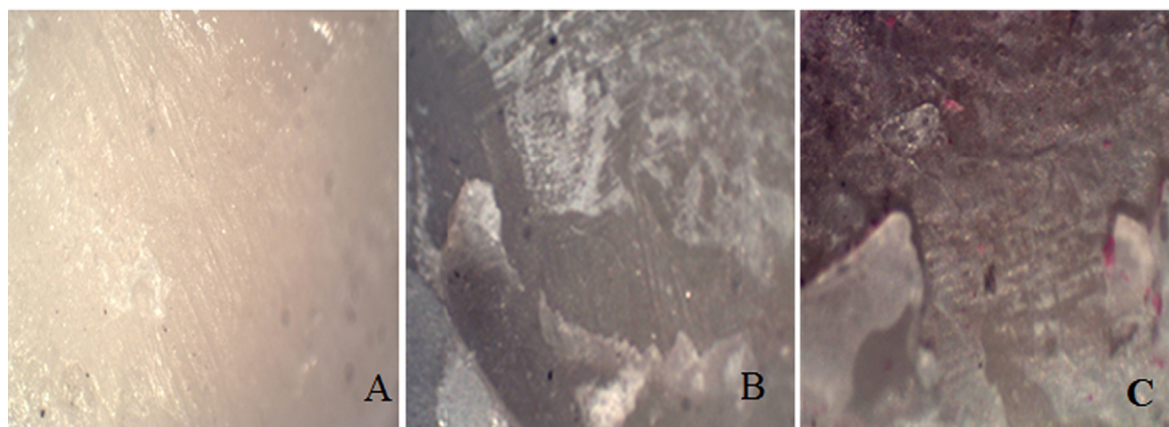


Fig. 4 Images of mode of failure under stereo microscope at 40X magnification, **A**: Adhesive failure, **B**: Mixed failure **C**: Cohesive failure.

Other possible cause of high bond strength of G- Premio BOND and Clearfil S³Bond Plus is that both of them have silicate fillers, these fillers are able to increase the mechanical properties of dental adhesive system and reduce shrinkage that may occur by polymerization (Lührs and Geurtsen, 2009). Silica particles have formerly been demonstrated the ability to enhance the formation of calcium- phosphate precursors “which is, an important step needed for mineralization”, so forming like a nucleating mineral (Watson et al., 2014). The existence of silica may cause attraction to calcium’s particles to create a bioactive compound “calcium-silicate” which may bind to phosphorus (Profeta, 2014).

Although the variation in the SBS wasn’t significant between G- Premio BOND and Clearfil S³Bond Plus, the mean value of the bond strength was higher in G- Premio BOND group. This may be related to that G-premio Bond has unusual

collection of three functional monomers (4 -MET, 10 -MDP and MDTP). In addition to that, the lower pH value of G-Premio BOND (1.5) as compared to pH of Clearfil S³ Bond Plus (2.7), which allows better dissolution of smear-layer and dissolving the HA crystals, so G-primo BOND makes a deep retentive form by creating a deep resin-tag in contrast with other adhesive system. Furthermore, it creates a denser adhesive-layer with higher flexible-interface (Somani et al., 2016.). Those superior characteristics of G- primo Bond aid to resist stress.

In the present study, long storage period and thermo-cycling for the groups without diode-laser treatment, caused a statistically significant decreasing in the SBS of all adhesive systems that included in the study. This is related to the fact that the hydrolytic dissolution process is an actuality in the adhesive systems. A clarification for the dissolution is that sin-

gle bottle bonding has hydrophilic monomers and water in their component (Tay and Pashley, 2001; Cadenaro et al., 2005). The bonded interfaces of self-adhesive system act like semipermeable membrane, which permit water to move through them and lead to Hydrolysis (Tay et al., 2002b. This finding is in agreement with finding of El-Araby et al., 2007, Costa et al., 2017 and Asgartooran et al., 2020.

Other outcome of this study was that the SBS of groups subjected to laser irradiation before photopolymerization was significantly increased. The explanation of this rising in bond strength belongs to the heat supplied directly by laser irradiation causing an increase in solvent evaporation and lead to deep penetration of bonding. Several studies found that the residual solvent in the bonding cause monomers dilution, this can affect polymerization and may lead to voids formation and rise the permeability of cured adhesives' layer which adversely affect bond strength to the dentin (Ikeda et al., 2005; Ikeda et al., 2008; Klein-Júnior et al., 2008). This outcome is coinciding with finding of Maenosono et al., 2015 and Ramachandruni et al., 2019, but disagrees with the finding of Zabeu et al., 2018 as they used non-simplified adhesive systems in their study.

One another interesting finding was that the bonding of all lased groups was not compromised by thermo-cycling and long storage period. Self-etch adhesives demineralize and infiltrate collagen fibrils of dentin simultaneously in which any incomplete resin infiltration may result in exposed collagen fibrils with subsequent degradation by MMPs "matrix metalloproteinases". Additionally, any residual water in bonding area may prevent complete resin polymerization, increasing permeability and nanoleakage of hybrid layer" (Li et al., 2001). Deeper resin infiltration and water evaporation by heat generated from laser irradiation could contribute to collagen fibrils protection and justify the preservation of bonding after 72 days' storage and thermo-cycling (Ikeda et al., 2005; Ikeda et al., 2008; Klein-Júnior et al., 2008). In addition to that, it is worthy to be mentioned that heat elevation might rise adhesive degree of conversion (Maenosono et al., 2015). Yet, FTIR analysis of polymerized adhesive system that previously subjected to laser irradiation is suggested to verify any changes in chemical composition of adhesive systems that could increase the dental adhesion.

The limitations of the current *in-vitro* study consisted of the absence of SEM evaluation of representative samples from the experimental groups to better visualize the depth of penetration of adhesive system, quality of hybrid layer and correlate the finding with the SBS values. Intra-pulpal temperature assessment of lased samples was also necessary to disclose its biological safety in order to upgrade this experiment to an *in-vivo* level.

It can therefore be assumed that irradiation with diode-laser following the placement of self-etch adhesive system and before photopolymerization could be considered a promising clinical additional step in an attempt to improve the bonding and preserve the durability of composite adhesion.

5. Conclusions

Under the conditions of this study, the followings could be found:

- 1- Highest SBS values were obtained by G-Premio BOND group followed by Clearfil S³ Bond Plus without significant difference between them.

- 2- Diode- laser significantly increased the bond- strength of self-adhesive systems to the dentin.
- 3- The storage and thermo-cycling had a significant negative effect on the bond strength of self- adhesive system that was not undergone diode-laser treatment.
- 4- Diode-laser treatment can minimize or prevent the negative effect of the storage and thermo-cycling and can increase the durability of the self-adhesive systems.

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Regulatory Statement

"This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Faculty of Dentistry, Mosul University, Iraq. The approval code for this study is": UoM.Dent/H. L.74/21 on 27/9/2021.

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