Acta Scientific Dental Sciences (ISSN: 2581-4893)

Volume 6 Issue 11 November 2022

Research Article

Evaluation of the Shear Bond Strength Between Ceramic and Pretreated Enamel using Different Etching Systems - Sem Analysis

Abdul Rafe, Jaya Krishna Kumar S*, Chitra Shankar Krishnan, Hariharan Ramakrishnan, Vallabh Mahadevan and Azhagarasan NS

Ragas Dental College and Hospital, Uthandi, Chennai, Tamilnadu, India

***Corresponding Author:** Jaya Krishna Kumar S, Ragas Dental College and Hospital, Uthandi, Chennai, Tamilnadu, India.

DOI: 10.31080/ASDS.2022.06.1491

Received: April 14, 2022
Published: October 14, 2022

Abstract

Aim: To evaluate the shear bond strength of the bond between ceramic and enamel pretreated with different etching systems.

Materials and Methods: A total of thirty-three freshly extracted maxillary central incisors were embedded in acrylic blocks using a custom-made jig. The teeth were prepared using a custom-made preparation guide limiting the depth of the preparation into the enamel. The prepared teeth were divided into three different groups of ten each as Group A, Group B and Group C, based on three different surface treatments namely, acid etching, laser etching and combination of acid etching followed by laser etching. One sample from each surface treated group was randomly selected for scanning electron microscope (SEM) analysis (10x,500x,1000x) before bonding of ceramic blocks to the prepared teeth. A total of thirty ceramic blocks were fabricated and were then bonded to the teeth etched with their respective surface treatment methods. The bonded test samples of Groups A, B and C were subjected to aging for a period of seven days and were tested for shear bond strength using a universal testing machine. One debonded test sample from Groups A, B and C was randomly selected for a qualitative analysis by SEM analysis. (10x,500x,1000x) The results were tabulated and subjected to statistical analysis.

Results: Group B (laser etching) exhibited the highest mean shear bond strength value followed by Group A (acid etching) and Group C (combination of acid etching followed by laser etching). The difference in the shear bond strength values among the three groups was statistically significant, found using One-way ANOVA. (Group B > Group A > Group C). Tukey HSD post hoc comparisons between the test groups revealed a statistically insignificant difference in mean shear bond strength value for Group A in comparison to Groups B and C and a statistically significant difference in mean shear bond strength value for Group B in comparison to Group C.

Conclusions: Er; Cr: YSGG laser etching is a viable alternative to acid etching of lithium disilicate ceramic. This was further corroborated by a predominantly cohesive pattern of failure of the resin cement with few areas of adhesive failure at the enamel-cement interface as observed on the debonded sample.

Keywords: Lithium Disilicate; Shear Bond; Laser Etching; Acid Etching; Sem Analysis; Bond Failure

Introduction

Durable bond between the adhesive cement and the restoration is also critical throughout the lifetime of a restoration [1]. A strong durable resin bond provides high retention, improves marginal adaptation, prevents microleakage and increases the fracture resistance of the restored tooth and restoration [2,3].

Citation: Jaya Krishna Kumar S., *et al.* “Evaluation of the Shear Bond Strength Between Ceramic and Pretreated Enamel using Different Etching Systems - Sem Analysis". *Acta Scientific Dental Sciences* 6.11 (2022): 51-65.

The bonded all-ceramic restorations provide a successful esthetic and functional service for patients. Clinical studies show excellent long-term success of bonded ceramic restorations such as inlays, onlays, laminate veneers and crowns [4]. Contemporary restorative dentistry places a definite emphasis on adhesion. Accordingly, a long-term survival of adhesive porcelain restorations depends on the success of a reliable bond between the porcelain, the composite luting agent and the dental substrates [5,6].

The ceramic restorations require considerable support from the underlying luting agent and enamel/dentin in order to optimize the bond strength between the restorations and the natural tooth [7,8]. The durability and the clinical performance of bonded porcelain restorations are mainly due to the cementing agents and adhesive systems. The cementation procedure is one of the factors for the clinical success of ceramic restoration [9]. This includes optimum surface treatment of the ceramic as well as proper choice and manipulation of the luting agent. Therefore, adequate ceramic surface conditioning is essential in order to have a strong resin bond that relies on the micromechanical interlocking and chemical bonding to the ceramic surface. Common treatment options for ceramic surface are grinding, abrasion with diamond rotary instruments, airborne particle abrasion with aluminium oxide, acid etching and combinations of any of these methods [10,11].

Acid etching with solutions of hydrofluoric acid (HF) or ammonium bifluoride can achieve proper surface texture and roughness. Hydrofluoric acid solutions between 2.5% and 10% applied for 2 to 3 minutes seem to be most successful. Silane coupling agent application improves the bond strength of porcelain to resin luting agent.

The surface treatment of dental substrate prior to adhesive restorative procedures is an extremely important step of the bonding protocol and accounts for the clinical success of restorations. In the literature, various surface treatment methods like air abrasion, acid etching and laser irradiation have been shown to etch enamel/dentin for the ceramic bonded restorations [12,13]. Air abrasion is a technique that involves use of air pressure with aluminium oxide powders to abrade dental tissues and produce large rough, irregular surface areas [14]. This can be regarded as a form of macro etching. The air abraded surface (sand blasted) displayed obtuse angularities instead of the sharp irregularities of etched enamel surfaces which could lead to weak bond strengths [15].

The chemical treatment of enamel was first proposed by Buonocore by etching the enamel surface with orthophosphoric acid and has been commonly used to increase the bond strength of bonded ceramic restorations [16]. The technique of etching with orthophosphoric acid is used to create an irregular surface of enamel. This allows an increase in the prepared surface area available for the retention of the resin cement and an improvement in the marginal adaptation of all ceramic restorations. The retentive characteristics of acid conditioned enamel surfaces depend on the type of acid, etching time and chemical composition of the enamel. Acid etching contributes to micromechanical retention of the adhesive components between the restoration and the enamel. The disadvantage of acid etching is that demineralization of the enamel surface makes it more permeable and prone to long term acid attack and caries. Currently, the most widely used protocol for enamel etching is with 37% phosphoric acid for 15 seconds [15-17].

The Er: YAG laser, originally developed by Zharikov., *et al.* in 1975, was approved by the FDA in 1997 for removal of caries, cavity preparations and modification of dentin and enamel surfaces prior to restoring with adhesive restorations. The Er; Cr: YSGG laser system was investigated in 1995 by Eversole and Rizolu. This pulsed laser device, when used with an air-water spray, has cut enamel, dentin, cementum and bone efficiently and cleanly without creating a significant smear layer. This laser system has been designated as hydrokinetic system (HKS) and can be used for tooth preparation without causing deleterious pulpal effects [18-20].

Laser etching is painless and does not involve either vibration or heat, making it highly attractive for routine use. Furthermore, laser etching of enamel has been reported to yield an anfractuous surface (fractured and uneven) and open dentinal tubules, both ideal for adhesion [13].

The surface produced by laser irradiation is also acid resistant. Laser irradiation of the enamel modifies the calcium-phosphate ratio and leads to the formation of more stable and less acid-soluble compounds, thus reducing susceptibility to caries attack. Therefore laser etching of enamel might be advantageous over phosphoric acid etching [20]. The use of both laser and acid together has also been reported to enhance the strength of bonding to hard tooth surfaces relative to those exposed to acid alone [13]. Resin cements have been selected for their advantageous mechanical and adhesive properties when compared with the conventional luting cements. The applications of dual-polymerizing resin cements for all-ceramic restorations have considerably increased due to the ability of these cements to polymerize completely and their greater resistance to occlusal loading [1,21-24].

The international standards organization document, TR110405 Dental Materials-Guidance has recommended longer periods of storage in a solution may be necessary to determine durability of bonds [21]. The complex nature of the oral environment has a direct influence on the bond that is achieved between the interfaces of bonded ceramic restoration especially of the cementing agent and hard tissue. Water absorption may reduce the mechanical properties of the resin based luting agents and is detrimental to the silane-ceramic bond [25,26]. Therefore, testing the samples following water storage is essential to better simulate the oral conditions and achieve predictable results.

The common tests used in literature for measuring the bond strength are three-point bending, tensile, microtensile and shear bond strength tests [27]. Shear strength testing is perhaps more clinically applicable because resistance to shear stresses are thought to be important in retaining restorations that have been bonded to enamel surfaces [19]. In this study, a conventional shear bond strength was used to evaluate the bond strength.

Studies that comparatively evaluate the shear bond strength between ceramic and enamel subjected to acid etching or irradiated with different laser systems are available [13,14,16,19,20]. However, research comparing the effects of Er; Cr: YSGG irradiated enamel with acid etched enamel on the shear bond strength with ceramic is sparse [13,16,20]. Also there are fewer studies comparing the combined effects of acid etching followed by laser etching with Er; Cr: YSGG laser system.

The null hypothesis adopted was that there would be no differences in shear bond strength between pretreated enamel and ceramic using different etching methods.

Materials and Methods

Thirty-three freshly extracted maxillary central incisors were utilised for the study which were free of caries, fractures, and restorations. The selected teeth were sectioned at 2 mm below their cemento enamel junction using a separating disc (Dentorium, New York, USA). On the palatal surface of the crowns two longitudinal 2 mm deep grooves of 1 mm width were made to aid in the retention of the sectioned crowns with the acrylic. The inner surfaces of both halves of the custom made stainless split jig (Figure 1), were then coated uniformly with petroleum jelly and then screwed tightly into place. Autopolymerizing acrylic resin (Cold cure, DPI- RR, India) was then poured into the mold space till the top and the sectioned natural tooth was embedded into the acrylic resin. The natural tooth was embedded in such a way that the labial surface was exposed, for tooth preparation. Once the excess was removed, the custom-made stainless-steel preparation guide (Figure 2) was then placed over the custom-made stainless-steel split jig and then secured into place further ensuring that the crown was mounted correctly.

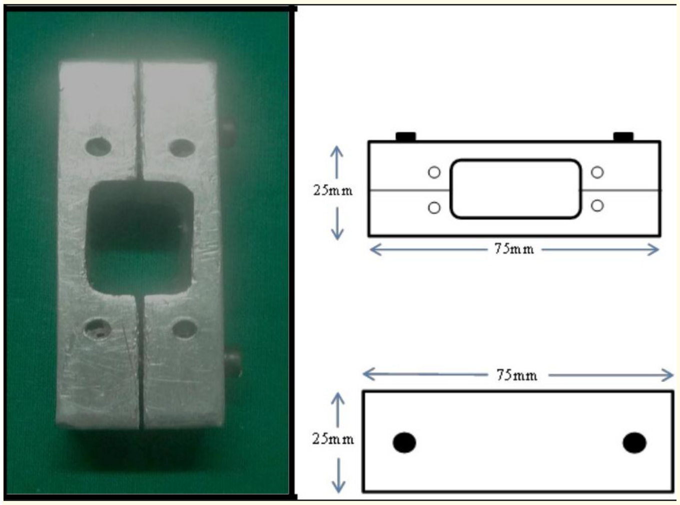

Figure 1: Custom made jig.

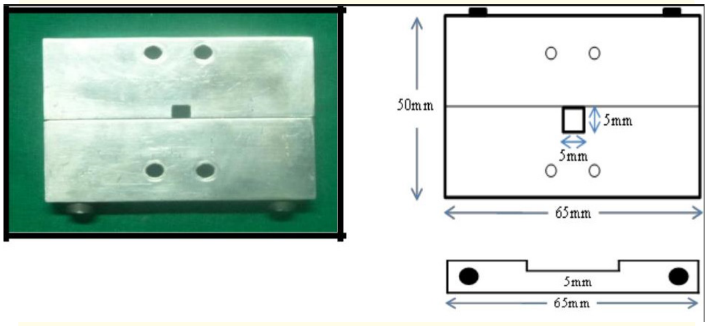

Figure 2: Custom made tooth prepartion guide.

Once the autopolymerizing resin (Cold cure, DPI-RR, India), had sufficiently cured, the custom-made stainless-steel preparation guide was unscrewed and the custom-made stainless-steel split mold was separated by removing its screws and the acrylic block was retrieved. The selected thirty-three natural teeth were embedded into the acrylic resin in an identical manner.

The middle portion of the labial surface of the teeth was selected for the preparation because of its larger width. The acrylic block with the embedded tooth was positioned in the custom made stainless steel split mounting jig and was secured tightly.

The custom made stainless steel tooth preparation guide was then placed on top and locked in place. This enabled to make the preparation in the middle one third of the tooth with the guide.

Premarked inverted cone burs (Dia Burs, Mani, Germany), were used with a 7 mm marking on their shanks measured from the tip to prepare through 2 mm into the enamel surface as the thickness of the metal preparation guide was 5 mm. Care was taken to limit the depth of the preparation in accordance with the markings on the burs, so as to not extend into the dentin surface. The preparation was done in order to simulate the clinical preparation of ceramic laminate veneer restoration. After accomplishing the general outline of the intended test sample (a 5 x 5 mm square with 2 mm depth), the area was marked and the tooth structure around this area was ground using a flat end tapered diamond abrasive (Dia Burs, Mani, Germany), to ensure no impedance during the test for shear bond strength.

The custom made stainless steel split mold was lined with die lubricant (Yeti Dental, Germany) on each side of the mold spaces to aid in the retrieval of the wax blocks. The wax custom made stainless steel split mold was then secured close and placed over a clean glass plate flush with its surface. Inlay wax (GC Corporation, Tokyo, Japan), was poured into the mold space in a molten state and was allowed to cool gradually at room temperature.

Then the mold was placed in a bowl of chilled water to further harden the wax blocks, for a minute. After this the mold was removed from the bowl and wiped dry. Before the screws on the split mold were removed the excess formed at the top was then gently carved out using a PKT no.4 instrument (Dispodent, India). The resulting wax block (measuring 5 x 5 mm) was then eased out with gentle finger pressure. In this manner a total of 33 wax blocks were obtained. The wax blocks were sprued using preformed sprue wax (Bego, Germany) of 2 mm diameter, invested with graphite free phosphate investment (Pressvest, Ivoclar Vivadent, Liechtenstein), and casted with the selected ingot (IPS e.max Press, Ivoclar Vivadent, Liechtenstein) by following all required manufacturer's instructions.

The flat ceramic surface was etched with 7% hydrofluoric acid gel (IPS Ceramic Etching gel, Ivoclar Vivadent, Liechtenstein) for 1 minute in order to condition the ceramic. The teeth were divided into three groups of eleven each, namely, Group A, Group B and Group C and subjected to three different surface treatments, namely, acid etching, laser etching and a combination of acid etching followed by laser etching respectively.

37% orthophosphoric acid (N Etch, Ivoclar Vivadent, Liechtenstein) was injected onto the prepared enamel surface of the teeth in Group A and left for 15 seconds. The tooth surface was then washed with water under pressure using a two-way syringe. Each surface was then dried using a chip blower only. The treated specimen was then kept aside carefully in a separate container to avoid contamination before bonding it to the ceramic sample.

Er; Cr: YSGG laser system (Waterlase MD, Biolase, USA) was used to ablate the prepared enamel surface of the teeth in Group B. The distance between the tip of the device and the surface of the sectioned crown was kept at 1 mm, and the laser beam was applied to the entire surface for 20 seconds. The laser was applied at a wavelength of 2,780 nm with pulse duration of 140 µs and a repetition rate of 15 Hz. The laser energy was 75 mJ.

Laser energy was delivered through a fibre-optic system via a sapphire tip terminal 600 µm in diameter and the surface was bathed with an adjustable air/water spray using a water level of 30% and an air level of 60%. The treated specimen was dried using a chip blower and then kept aside carefully in a separate container to prevent it from contamination before bonding it with the ceramic block.

Foe eleven samples in Group C, 37% orthophosphoric acid (N Etch, Ivoclar Vivadent, Liechtenstein) was injected onto the prepared surface of the teeth and left for 15 seconds. The teeth surface was then washed with water under pressure using a two way syringe. Each surface was then dried using a chip blower only. Er; Cr: YSGG laser system (Waterlase MD, Biolase, USA) was used. The laser was used procedure wise similar to the one described for Group B One representative prepared tooth sample from each group (A, B and C) was randomly selected and set aside for the qualitative analysis of the surface topography of surface treated, prepared teeth samples before bonding with ceramic blocks. The remaining thirty pretreated teeth were kept for cementation procedures.

The silane coupling agent (Monobond S, Ivoclar Vivadent, Liechtenstein) was applied onto the previously etched ceramic block's bonding surface using a microbrush and left for 60 seconds and then air dried. The bonding agent (Heliobond, Ivoclar Vivadent, Liechtenstein), was then applied onto the silanated surface of the ceramic bloc and then cured using a light cure unit (Confident, India) according to the manufacturer's instructions.

The primer (Syntac Primer, Ivoclar Vivadent, Liechtenstein) was then applied onto the prepared tooth surface using a microbrush tip and left to dry for 20 seconds. Excess was then removed by blowing air using a chip blower. An adhesive (Syntac Adhesive, Ivoclar Vivadent, Liechtenstein) was then applied onto the prepared tooth surface using a microbrush tip and left to dry for 20 seconds. The excess was then removed by blowing air using a chip blower. The bonding agent (Heliobond, Ivoclar Vivadent, Liechtenstein) was then applied onto the tooth surface and then cured using a light cure unit (Confident, India) according to the manufacturer's instructions. Equal amounts of the dual-cure resin luting cement's (Variolink N, Ivoclar Vivadent, Liechtenstein) base and catalyst paste were then dispensed onto the mixing pad and mixed using a plastic spatula.

The mixed cement was then applied onto the previously etched and silane treated surface of the ceramic block and then the ceramic block was then pressed against the tooth surface under light finger pressure. The excess was carefully removed from the sides and the cement was further polymerized using a light cure unit (Confident, India) for 40 seconds. In this manner thirty ceramic blocks were cemented to the enamel pretreated with three different etching methods.

The ceramic bonded to natural teeth test samples of Groups A, B and C were then stored in distilled water placed in an incubator (Narang Industries Ltd., India) at 37^0C for seven days before testing them for their shear bond strengths. The water was changed on a daily basis. This was done to simulate the oral conditions. The test samples were tested for shear bond strength using a universal testing machine (Lloyd Instruments, Farnham, United Kingdom). The force was applied at 90^0 to the long axis of the tooth. The acrylic mold was mounted in the lower member and the upper member had the chisel with a cross head. A shear force was applied to the ceramic test sample at a cross head speed of 0.5mm/min until fracture occurred. The maximum fracture loads were recorded in Newton. The recorded values were then divided by the surface area of the sample to obtain the shear bond strength values in MPa. A total of 30 test samples were tested in identical manner and the shear bond strengths were tabulated for statistical analysis.

SEM analysis was carried out on one representative surface treated, prepared teeth sample, randomly selected from each test group (Group A, Group B and Group C) before bonding of ceramic blocks using a scanning electron microscope (SA400N, Canada). The samples were placed on stubs, secured in place with an adhesive tape and coated with a thin layer of gold in a gold sputtering system. Coated samples were examined under SEM to examine the surface topography of the treated samples at 10x, 500x and 1000x magnification.

SEM analysis was carried to identify the mode of failure, on one representative tested sample from each test group (Group A, Group B and Group C) after debonding of ceramic blocks, using a scanning electron microscope (SA400N, Canada). The samples were placed on stubs, secured in place with an adhesive tape and coated with a thin layer of gold in a gold sputtering system. Coated samples were examined under SEM to examine the mode of failure of the samples at 10x, 500x and 1000x magnifications.

Results

All the statistical tabulations were done using Microsoft Excel (Microsoft, U.S.A.). the SPSS (SPSS for Windows 10.05, SPSS Software Corporation, Munich, Germany) software package was used for statistical analysis. One-way ANOVA was used to compare the mean values of the three groups (A, B, and C). Tukey-HSD was used as the post hoc test and a p value < 0.05 was considered statistically significant.

The mean shear bond strengths of Group A, Group B and Group C using One-way ANOVA showed that there was a statistically significant difference between the mean shear bond strength of the three groups. Group B (laser etching) had the highest mean shear bond strength followed by Group A (acid etching) and the lowest shear bond strength value was observed in Group C (combination of acid etching followed by laser etching).

Comparison between the mean shear bond strengths of Group A and Group B it was found that Group B had exhibited a higher mean shear bond strength value compared to Group A. On statistical analysis using Tukey HSD, ($p > 0.05$) there was no statistically significant difference between these two groups. comparison between the mean shear bond strengths of Group A and Group C it was found that Group A had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that (p- > 0.05), there was no statistically significant difference between these two groups.

Comparison between the mean shear bond strengths of Group B and Group C it was found that Group B had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that (p- <0.05), denoting a statistically significant difference between these two groups.

Discussion

Ceramic surface treatment is fundamental for bonding to resin [2,4,28]. The common surface treatments listed in literature are acid etching, airborne particle-abrasion, grinding and a combination of any of these methods [4,29].

Acid etching of porcelain creates microporosities on the porcelain surface, which form a micromechanical interlock with the luting agent [30]. Several porcelain etchants have been developed like hydrofluoric acid and acidulated phosphate fluoride (APF) [30]. The most commonly used etchant is a 10% solution of hydrofluoric acid [30]. Hydrofluoric acid attacks the glass phase of conventional ceramic materials producing a retentive surface for micromechanical bonding [4,28]. It has also been reported in literature that hydrofluoric acid solutions between 2.5% and 10% applied for one to four minutes are most successful in achieving a proper surface texture and roughness of ceramic surface [4]. In accordance with the literature available, the present study used a 7% hydrofluoric acid gel applied onto the ceramic blocks for one minute as the surface conditioning agent.

Recent developments in modern surface conditioning methods with silane coupling agents have resulted in improved bond strength of porcelain to the luting agent [4,27]. Silane application improves the wettability of the ceramic and contributes to covalent bond formation between the ceramic and the luting agent [4,31]. Silanes are bifunctional molecules that bond silicon dioxide with the OH groups on the ceramic surface and copolymerizes with the organic matrix of the resin cement [4]. It has also been reported that, etching and silanization significantly decreases microleakage [2,4,27].

The treatment of dental substrate prior to adhesive restorative procedures is an extremely important step of the bonding protocol and accounts for the clinical success of all-ceramic restorations [32]. During conventional tooth preparation with rotating instruments a smear layer is produced on the surface which consists mainly of pulverized enamel and dentin, carious debris, and bacteria [13,18]. The low surface energy of this layer prevents, impregnation of the enamel and dentin with the adhesive agent and thus, an adequate adhesion thereby affecting the durability of the bond between the restoration and the tooth [18]. The standard approach to solve this problem has been removal of the smear layer before sealing or bonding by surface treatment of the dental substrate [18]. The primary effect of enamel etching is to increase the surface area and thereby change the surface substrate from a low energy hydrophobic surface to a high-energy hydrophilic surface [12,33].

In the literature, various surface treatments for treating enamel/dentin have been reported using chemicals like phosphoric acid, maleic acid and mechanical methods like intra oral air abrasion and laser etching [12,34]. Buonocore (1955), postulated that acids could be used to treat the prepared tooth surface before the application of resins [5,7,15,35]. The most widely used method is the application of 37% phosphoric acid for the enamel surface [15]. Phosphoric acid acts on the enamel by selectively dissolving the hydroxyapatite of the prisms, thereby facilitating penetration of the bonding agents and tag formation [15]. A disadvantage attributed to acid etching is that demineralisation of enamel surface makes it more permeable and prone to long term acid attack and caries, especially if the demineralised substrate is not completely filled by the resin monomers [15,34,36].

The other methods tried as alternatives to acid etching with phosphoric acid were other acids such as maleic acid [16] or air abrasion using alumina 50 μm with/without acid etching [14] and laser etching [12]. Berk., *et al.* (2008) [12], in their *in vitro* study concluded that air abrasion was not a viable alternative to acid etching as it resulted in macroetching as opposed to microetching, attained with acid etching [12]. It has also been reported that etching with 37% phosphoric yields better bond strengths than etching with 10% maleic acid [16]. In accordance with the literature, the present *in vitro* study used 37% phosphoric acid to etch the enamel surfaces of the prepared tooth samples for 15 seconds.

The action of lasers depends on their wavelengths and their subsequent absorbtion by the target tissue. CO_2 laser and the erbium family of lasers, (Er; Cr: YSGG and Er: YAG) are the lasers preferred for working with hard tissues like the tooth and bone, because of their absorbtion by water [37]. Some of the laser systems have the ability to treat dental surfaces to create a rough microretentive pattern [12]. Lasers such as Nd: YAG and CO_2, have been examined, but the initial results with these lasers were not encouraging due to the thermally induced injuries to the surrounding tissues including pulpal damage [18,19,38]. Many investigators have reported the ability of the Er: YAG laser to ablate tooth structure, which has also been indicated for selective removal of carious lesion, cavity preparation and modification of dentin and enamel surfaces prior to restoring with adhesive materials [18,19,38].

The mechanism of action of erbium lasers has been reported to be the same, [37] with only a minor difference in their wavelengths with Er: YAG being 2.94 µm as opposed to the Er; Cr: YSGG wavelength of 2.89 µm [18,19]. When the laser energy is focused onto the tooth, the water contained therein, is heated and the steam causes an increase in the irradiated volume. This expansion surpasses the crystal strength of the dental structures, and results in ablation. This mechanism explains the anfractuous, microretentive pattern obtained after etching with the Erbium lasers [13,15,16,19,34,39,40,41]. The present *in vitro* study used an Er; Cr: YSGG laser to etch the enamel surface of the prepared tooth samples.

Resin based composite cements are the cements of choice for the adhesive luting of ceramic restorations [2]. Resin cements are capable of producing micromechanical attachment to the tooth structure [42]. Dual-cured cements traditionally are used when ceramic thickness does not allow light penetration for maximal conversion of the luting cement [19,43]. Disadvantages of dual-cured cements include porosity from mixing, reduced working time, decreased degree of conversion and color instability due to amine degradation [4]. In accordance with the literature, the present *in vitro* study used a dual-cured resin cement for the bonding of ceramic blocks (5 x 5 mm) onto the surface treated tooth samples.

Earlier studies have reported on the effect of water storage on the bond strength. The International Standards Organization's report on the testing of dental materials TR110405 also states that longer periods of storage in a solution are necessary to determine the durability of bonds [21]. Storage in water may result in hydrolytic degeneration of the interface components especially of the resin cement and/or collagen and is also detrimental to the silane-ceramic bond [1,9]. Storage in water and additional thermocycling create stress at the cementing agent/hard tissue interface [9]. In the present *in vitro* study the samples were stored in distilled water at 37^o C for a period of seven days to simulate the oral conditions.

The occlusal forces applied to a restoration may be complex and made up of a combination of forces such as shear, tension, compression and flexure [44]. The tests most widely used to examine bond strength of resin composite to dentin are tensile and shear tests [44]. Shear strength is clinically more applicable because resistance to shear stresses are important in retaining restorations that have been bonded to enamel surfaces [20]. In the present *in vitro* study, a conventional shear bond strength test with a crosshead speed of 0.5 mm [22] was used to evaluate the long-term durability.

The effect of laser etching, acid etching and combination of acid etching and laser etching on enamel has been evaluated in various researches [12,13,14,16,19,20,45].

The shear bond strength of composites, [12,14,19,20,45] and ceramics [13] to the treated enamel surface have been evaluated in literature. Previous studies show that the bond strength values achieved with laser etching of enamel have been comparable to the bond strength values achieved with acid etching [13,14,16,19,20,32,45]. Application of laser etching has been suggested as an alternative to acid etching considering its etching property and other advantages like the increased resistance to caries, ease of handling and faster means of etching. The results obtained with the present study are broadly in line with the results obtained in the previous study. However, researches identical to the present study parameters are sparse in the literature.

Dundar., *et al.* (2009) [13] had comparatively evaluated the shear bond strength of ceramic to enamel after different surface treatment of enamel (acid etching, laser etching and a combination of acid etching and laser etching). The mean shear bond strength value obtained with acid etching (15.44 MPa) was slightly higher than that achieved with laser etching (12.89 MPa) and the combination of acid etching followed by laser etching (13.87 MPa) but the results were significant (Table 1). The compositions of the ceramic material and the resin cement used for bonding to the enamel surface in other studies are different from the ceramic and resin cement used in the present study. But the shear bond strength values yielded by the etching methods are comparable with the shear bond strengths obtained in the present study. The results obtained in the present study are in accordance with the study by Visuri., *et al.* (1996) [45] which revealed higher shear bond strength value of composite resin when it was bonded to laser prepared tooth surface (12.9 MPA) than with acid etched tooth surface (7.3 MPa).

Lin., *et al.* (1999) [19] stated that the use of an Er; Cr: YSGG laser provided surfaces that are receptive to attachment of restorative materials. Enamel surfaces treated with the Er; Cr: YSGG laser (23.7 MPa) yielded shear bond strengths similar to those obtained with acid etched bur-cut enamel (23.3 MPa) and the author has suggested the use of laser. Usumez., *et al.* (2003) [16] reported that the microtensile bond strength of porcelain laminate veneers bonded to tooth surfaces that were laser etched (12.1 MPa) showed results similar to acid etched (13 MPa) tooth surfaces. Moslemi., *et al.* (2010) [14] stated that there was no statistically significant difference between shear bond strength values obtained with acid (37% phosphoric acid) etching (23.51 MPa) and combination laser and acid etching (21.44 MPa) and these results are in accordance with the present study.

GROUP	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P-value
A	10	11.9470	+/-0.79081	0.049*
B	10	12.7640	+/-1.91180	
C	10	11.2760	+/-0.80199	

Table 1: Comparison between mean shear bond strength values of Group A (acid etching), Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples using One-way ANOVA.

*P-value < 0.05 denotes significance.

Inference: On comparison between the mean shear bond strengths of Group A, Group B and Group C using One-way ANOVA it was found that there was a statistically significant difference between the mean shear bond strength of the three groups. Group B (laser etching) had the highest mean shear bond strength followed by Group A (acid etching) and the lowest shear bond strength value was observed in Group C (combination of acid etching followed by laser etching).

The qualitative analysis of the treated surface of Group A sample before bonding to ceramic block showed a definite, type III key-hole pattern throughout the surface. The predominantly adhesive mode of failure of Group C test sample is in correlation with the significantly lower shear bond strength values obtained with this group. They presented a uniform micro-retentive pattern over the entire etched area. No smear layer was visible over the etched surface. (Figure 3a, 3b) The surface analysis of the Group B sample before bonding to ceramic block showed no definite or uniform pattern and an absence of a smear layer. The surface had numerous voids and a definite micro-retentive topography with several raised elevations and depressions. (Figure 4a,4b) The surface analysis of the Group C sample before bonding to ceramic block revealed a heterogeneous topography, showing both acid induced porosities and laser induced surface roughness (Figure 5a,5b).

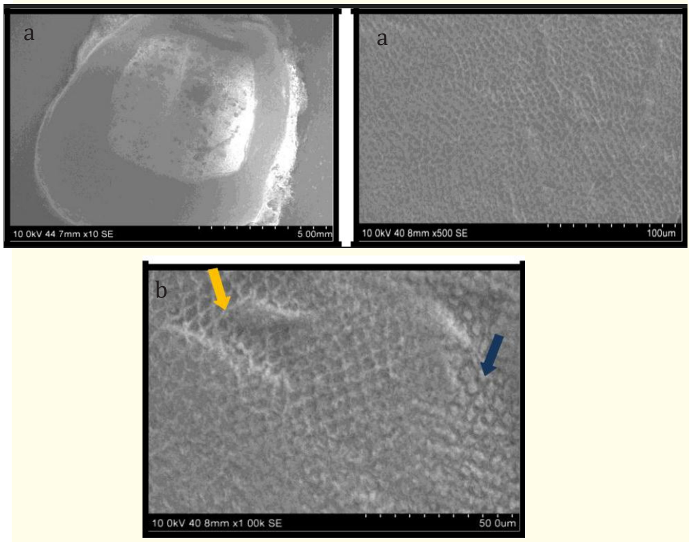

Figure 3: a)Group A sample after etching and before bonding with ceramic 10x, 500x. b)Group A sample after etching and before bonding with ceramic 1000x

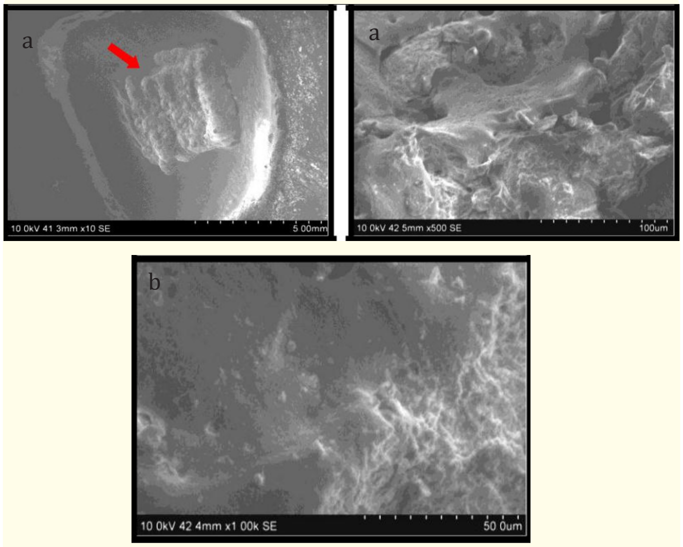

Figure 4: a)Group B sample after etching and before bonding with ceramic 10x, 500x. b) Group B sample after etching and before bonding with ceramic 1000x.

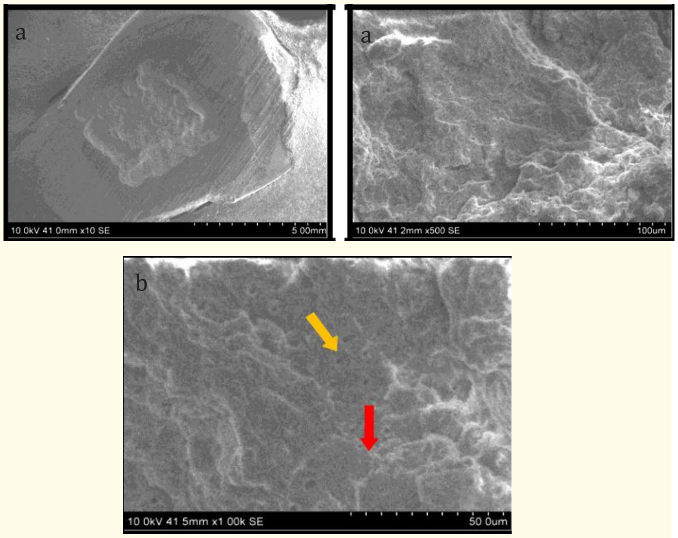

Figure 5: a)Group C sample after etching and before bonding with ceramic 10x, 500x. b) Group C sample after etching and before bonding with ceramic 1000x.

The qualitative analysis of the surface of Group A sample after debonding revealed a mixed failure pattern. There was a predominant cohesive failure occurring within the cement and an adhesive pattern of fracture between the enamel and cement (Figure 6a, 6b). The surface analysis of the Group B sample after debonding revealed a predominantly cohesive mode of failure in the cement with few areas of adhesive failure (Figure 7a,7b). The surface analysis of the Group C sample after debonding revealed a predominantly adhesive failure pattern (Figure 8a,8b).

The scanning electron microscope (SEM) analysis findings are in correlation with the results obtained from the shear bond strength test. The higher shear bond strength values obtained with Group B and Group A test samples could be attributed to the predominantly cohesive nature of failure with these groups. The lower percentage of adhesive failure areas for Group B test sample as compared to the Group A test sample could account for its higher shear bond strength value obtained in the present study. results obtained from the qualitative analysis of this study are in correlation with the results obtained from the quantitative

Citation: Jaya Krishna Kumar S., *et al.* “Evaluation of the Shear Bond Strength Between Ceramic and Pretreated Enamel using Different Etching Systems - Sem Analysis". *Acta Scientific Dental Sciences* 6.11 (2022): 51-65.

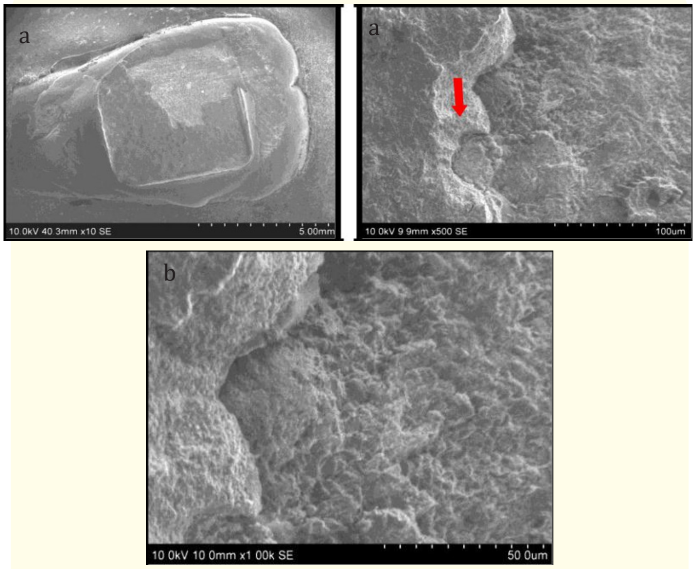

Figure 6: a)Group A sample after debonding with ceramic 10x, 500x. b) Group A sample after debonding with ceramic 1000x.

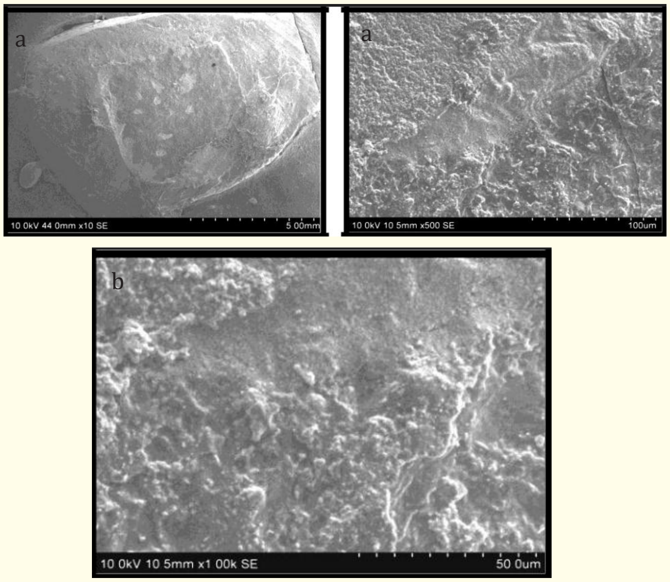

Figure 7: a)Group B sample after debonding with ceramic 10x, 500x. b) Group B sample after debonding with ceramic 1000x.

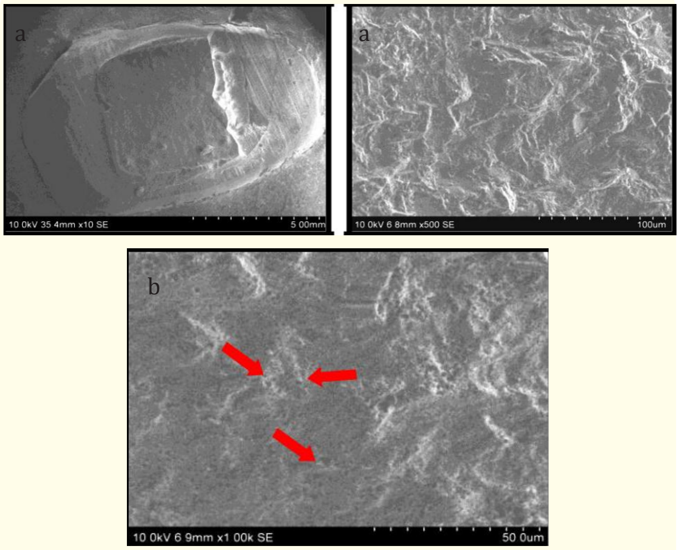

Figure 8: a)Group C sample after debonding with ceramic 10x, 500x. b) Group C sample after debonding with ceramic 1000x.

analysis of the study with Group B (laser etching) samples showing the highest mean shear bond strength followed by Group A (acid etching) and Group C (combination of acid etching followed by laser etching) samples.

This study reports a higher mean shear bond strength for laser etched enamel as opposed to acid etched enamel but there was no statistical significance. This study also reports higher mean shear bond strength for laser etched samples on comparison with samples which received a combination of acid etching followed by laser etching. The difference observed between laser etching and a combination of acid etching and laser etching was statistically significant (Table 2-4). The lower mean shear bond strengths exhibited by the samples which were treated by a combination of acid etching followed by laser etching may be due to the lower amount of surface roughness produced by it as observed under the SEM. It is well accepted that a high surface roughness is closely related to greater bonding [39]. Therefore, the null hypothesis is rejected.

GROUP	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P-value
A	10	11.9470	+/-0.79081	0.342 *
B	10	12.7640	+/-1.91180	

Table 2: Comparison of mean shear bond strength values of Group A (acid etching) and Group B (laser etching) test samples using Tukey HSD test.

P-value > 0.05; insignificant.

Inference: On comparison between the mean shear bond strengths of Group A and Group B it was found that Group B had exhibited a higher mean shear bond strength value compared to Group A. On statistical analysis using Tukey HSD, it was found that the p-value >0.05, denoting no statistically significant difference between these two groups.

Group	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P-value
A	10	11.9470	+/-0.79081	0.480*
C	10	11.2760	+/-.80119	

Table 3: Comparison of mean shear bond strength values of Group A (acid etching) and Group C (combination of acid etching followed by laser etching) test samples using Tukey HSD test.

P-value > 0.05; insignificant.

Inference: On comparison between the mean shear bond strengths of Group A and Group C it was found that Group A had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that the p-value >0.05, denoting no statistically significant difference between these two groups.

Group	Number of samples	Mean Shear Bond Strength (MPa)	Standard Deviation	P-value
B	10	12.7640	+/-1.91180	0.039*
C	10	11.2760	+/-0.80119	

Table 4: Comparison of mean shear bond strength values of Group B (laser etching) and Group C (combination of acid etching followed by laser etching) test samples using Tukey HSD test.

*P-value < 0.05; significant.

Inference: On comparison between the mean shear bond strengths of Group B and Group C it was found that Group B had exhibited a higher mean shear bond strength value compared to Group C. On statistical analysis using Tukey HSD, it was found that the p-value <0.05, denoting a statistically significant difference between these two groups.

Acid etching with 37% phosphoric acid has yielded satisfactory bond strengths for a long time, but it's liability to leave the enamel demineralised has always been a point of concern. Laser etching, in turn leaves the enamel more resistant to acid dissolution [15,21]. The area to be etched can be very precisely limited with a laser without any damage to the surrounding tooth structure. The results of the present study suggest laser etching to be a viable alternative to acid etching.

Conclusions

- Er; Cr: YSGG laser etching is a viable alternative to acid etching of lithium disilicate ceramic.
- This was further corroborated by a predominantly cohesive pattern of failure of the resin cement with few areas of adhesive failure at the enamel-cement interface as observed on the debonded sample.
- Surface treated using both laser etching and acid etching provided least bond strength between ceramic and enamel.

Bibliography

1. Pekkan G and Hekimoglu C. "Evaluation of shear and tensile bond strength between dentin and ceramics using dual-polymerizing resin cements". *Journal of Prosthetic Dentistry* 102 (2009): 242-252.
2. Blatz MB., *et al*. "Resin-ceramic bonding: a review of the literature". *Journal of Prosthetic Dentistry* 89.3 (2003): 268-274.
3. Piwowarczyk A., *et al*. "Microleakage of various cementing agents for full cast crowns". *Dental Materials* 21 (2005): 445-453.
4. Kim TH., *et al*. "Selection of luting agents: part 2". *Journal of the California Dental Association* 34 (2006): 161-166.
5. Cura C., *et al*. "Effect of different bonding agents on shear bond strengths of composite-bonded porcelain to enamel". *Journal of Prosthetic Dentistry* 89 (2003): 394-399.
6. Duarte S., *et al*. "The effect of immediate dentin sealing on the marginal adaptation and bond strengths of total-etch and self-etch adhesives". *Journal of Prosthetic Dentistry* 102 (2009): 1-9.
7. Shimada Y., *et al*. "Micro-shear bond strength of dual-cured resin cement to glass ceramics". *Dental Materials* 18 (2002): 380-388.
8. Yilmaz H., *et al*. "Flexural strength and fracture toughness of dental core ceramics". *Journal of Prosthetic Dentistry* 98 (2007): 120-128.
9. Piwowarczyk A., *et al*. "Long-term bond between dual-polymerizing cementing agents and human hard dental tissue". *Dental Materials* 23 (2007): 211-217.
10. Blixt M., *et al*. "Bonding to densely sintered alumina surfaces: effect of sandblasting and silica coating on shear bond strength of luting cements". *The International Journal of Prosthodontics* 13.3 (2000): 221-226.
11. Stewart GP., *et al*. "Shear bond strength of resin cements to both ceramic and dentin". *Journal of Prosthetic Dentistry* 88 (2002): 277-284.
12. Berk N., *et al*. "Comparison of sandblasting, laser irradiation, and conventional acid etching for orthodontic bonding of molar tubes". *European Journal of Orthodontics* 30 (2008): 183-189.
13. Dundar B and Guzel KG. "An analysis of the shear strength of the bond between enamel and porcelain laminate veneers with different etching systems: acid and Er, Cr: YSGG laser separately and combined". *Lasers in Medical Science* 26.6 (2011): 777-782.
14. Moslemi M., *et al*. "The effect of Er, Cr: YSGG laser and air abrasion on shear bond strength of a fissure sealant to enamel". *The Journal of the American Dental Association* 141 (2010): 157-161.
15. Martínez-Insua A., *et al*. "Differences in bonding to acid-etched or Er: YAG-laser-treated enamel and dentin surfaces". *Journal of Prosthetic Dentistry* 84 (2000): 280-288.
16. Usumez A and Aykent F. "Bond strengths of porcelain laminate veneers to tooth surface prepared with acid and Er, Cr: YSGG laser etching". *Journal of Prosthetic Dentistry* 90 (2003): 24-30.
17. Kim JT and Cho SA. "The effects of laser etching on shear bond strength at the titanium ceramic interface". *Journal of Prosthetic Dentistry* 101 (2009): 101-106.

18. Celik EU., *et al*. "Shear bond strength of different adhesives to Er: YAG laser-prepared dentin". *The Journal of Adhesive Dentistry* 8 (2006): 319-325.
19. Lin S., *et al*. "Topographical characteristics and shear bond strength of tooth surfaces cut with a laser-powered hydrokinetic system". *Journal of Prosthetic Dentistry* 82 (1999): 451-455.
20. Türkmen C., *et al*. "Shear bond strength of composite bonded with three adhesives to Er, Cr: YSGG laser-prepared enamel". *Quintessence International* 41 (2010): 119-124.
21. Kitasako Y., *et al*. "Shear bond strengths of three resin cements to dentine over 3 years *in vitro*". *Journal of Dentistry* 29 (2001): 139-144.
22. Piwowarczyk A., *et al*. "*In vitro* shear bond strength of cementing agents to fixed prosthodontic restorative materials". *Journal of Prosthetic Dentistry* 92 (2004): 265-273.
23. Qeblawi DM., *et al*. "The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement". *Journal of Prosthetic Dentistry* 103.4 (2010): 210-220.
24. Stangel I., *et al*. "Shear strength of the composite bond to etched porcelain". *Journal of Dental Research* 66 (1987): 1460-1465.
25. Ozcan M and Niedermeier W. "Clinical study on the reasons for and location of failures of metal-ceramic restorations and survival of repairs". *The International Journal of Prosthodontics* 15.3 (2002): 299-302.
26. Ritter AV., et al. "Dentin and enamel bond strengths of dual-cure composite luting agents used with dual- cure dental adhesives". *Journal of Dentistry* 37 (2009): 59-64.
27. Khoroushi M and Motamedi Sh. "Shear bond strength of composite resin to porcelain: effect of thermocycling". *Journal of Dentistry (Tehran)* 4 (2007): 21-25.
28. Borges GA., *et al*. "Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics". *Journal of Prosthetic Dentistry* 89.5 (2003): 479-488.
29. Brosh T., *et al*. "Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites". *Journal of Prosthetic Dentistry* 77 (1997): 122-126.
30. Al Edris A., *et al*. "SEM evaluation of etch patterns by three etchants on three porcelains". *Journal of Prosthetic Dentistry* 64 (1990): 734-739.
31. Oztoprak MO., *et al*. "Effects of different application durations of scanning laser method on debonding strength of laminate veneers". *Lasers in Medical Science* (2011).
32. Chimello-Sousa DT., *et al*. "Influence of Er:YAG laser irradiation distance on the bond strength of a restorative system to enamel". *Journal of Dentistry* 34 (2006): 245-251.
33. Iwami Y., *et al*. "Effect of enamel and dentin surface wetness on shear bond strength of composites". *Journal of Prosthetic Dentistry* 80 (1998): 20-26.
34. Bester SP., *et al*. "The effect of airborne particle abrasion on the dentin smear layer and dentin: an *in vitro* investigation". *The International Journal of Prosthodontics* 8 (1995): 46-50.
35. Yuasa T., *et al*. "Effects of long-term storage and thermocycling on bond strength of two self-etching primer adhesive systems". *European Journal of Orthodontics* 32 (2010): 285-290.
36. Chen JH., *et al*. "Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain". *Journal of Dentistry* 26 (1998): 53-58.
37. G van As. "Erbium lasers in dentistry". *Dental Clinics of North America* 48 (2004): 1017-1059.
38. Bader C and Krejci I. "Indications and limitations of Er: YAG laser applications in dentistry". *American Journal of Dentistry* 19 (2006): 178-186.
39. Khatavkar R and Hegde V. "Surface analysis of Erbium: YAG laser etching v/s acid etched surface: ESEM observations *in vitro* study". *Laser* 1 (2011): 40-44.
40. Ramos RP., *et al*. "Bonding of self-etching and total-etch systems to Er: YAG laser-irradiated dentin. Tensile bond strength and scanning electron microscopy". *Brazilian Dental Journal* 15 (2004): 9-20.
41. Souza-Gabriel AE., *et al*. "Shear bond strength of resin-modified glass ionomer cements to Er: YAG laser-treated tooth structure". *Operative Dentistry* 31 (2006): 212-218.

42. O El -Mowafy. “The use of resin cements in restorative dentistry to overcome retention problems”. *Journal of the Canadian Dental Association* 67 (2001): 97-102.

43. Rosenstiel SF., *et al*. “Dental luting agents: A review of the current literature”. *Journal of Prosthetic Dentistry* 80 (1998): 280-301.

44. Al-Salehi SK and Burke FJ. “Methods used in dentin bonding tests: an analysis of 50 investigations on bond strength”. *Quintessence International* 28.11 (1997): 717-723.

45. Visuri SR., *et al*. “Shear strength of composite bonded to Er: YAG laser-prepared dentin”. *Journal of Dental Research* 75 (1996): 599-605.