Evaluation of the shear bond strength of orthodontic brackets bonded to different ceramic surfaces

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and my mother, she will always live in my heart.

SUMMARY

Due to the increase in the number of adults seeking orthodontic treatment, new challenges have risen for orthodontists, such as the need to bond orthodontic brackets to ceramic restorations, whose glazed surfaces are not amenable to resin penetration and have a higher degree of failure compared with bonding to enamel. The aim of this study was to conduct an inclusive and substantial analysis of the factors affecting shear bond strength (SBS) of metallic and ceramic orthodontic brackets bonded to different ceramic surfaces used for prosthetic restorations. The research was conducted with 144 ceramic specimens of three different types (feldspar-porcelain fused to metal, zirconia and lithium disilicate), on which orthodontic brackets consisting of different materials (metallic and polycrystalline ceramic) were bonded. The bonding surface of the specimens was conditioned with two different etching materials (hydrofluoric acid or phosphoric acid), and then silane was applied. SBS was tested in Universal Testing Machine and values were calculated in MPa. The samples were also analyzed using a digital microscope and Scanning Electron Microscope (SEM) in order to determine the Adhesive Remnant Index (ARI) and Porcelain Fracture Index (PFI). The results showed that the use of HFA for surface etching of feldspar, all-contour zirconia and/or lithium disilicate, does not cause a significant increase in the SBS values as compared to etching with PHA and silane application, which is a safer method to use in clinical conditions. However, ceramic brackets show significantly higher SBS values than metallic brackets in feldspar and lithium disilicate. Also, ARI significantly depends on the type of bracket ($p = 0.005$). Furthermore, since PFI depends on the type of etchant ($p = 0.029$), HFA can weaken the surface structure of the ceramic, and considering its noxious effect, might not be the best suitable conditioner prior to orthodontic bonding to feldspar, lithium disilicate, and in particular to zirconia, also taking into account its crystalline structure.

Keywords: Feldspar, Zirconia, Lithium disilicate, Orthodontic brackets, Phosphoric acid, HFA, SBS, ARI, PFI.

PROCJENA SNAGE VEZIVANJA ORTODONTSKIH BRAVICA LJEPLJENIH NA RAZLIČITE KERAMIČKE POVRŠINE

PROŠIRENI SAŽETAK

Uvod: Dentalna keramika ima široku primjenu, najviše kao restaurativni materijal, prvenstveno zbog svoje izvrsne estetike, mehaničkih svojstava i biokompatibilnosti. Zbog sve većeg broja odraslih osoba koje traže ortodontsku terapiju, pojavili su se novi izazovi za ortodonte, među kojima je i potreba za vezivanjem ortodontskih bravica na keramičke restauracije, čija ostakljena površina nije pogodna za prodiranje smole i ima veći stupanj neuspjeha u odnosu na vezivanje za caklinu. To najviše ovisi o vrsti keramike, pripremi površine, materijalu od kojeg su izrađene bravice i drugim čimbenicima. Stoga je nastala potreba pronalaska pouzdane metode vezivanja ortodontskih bravica na keramičke krunice kako bi se dobila veća čvrstoća veze. S druge strane, potrebna je odgovarajuća čvrstoća veze zbog lakog i sigurnog uklanjanja bravica kako bi se smanjila vjerojatnost oštećenja restaurirane površine.

Cilj ovog istraživanja jest provođenje sveobuhvatne i temeljite analize čimbenika koji utječu na posmičnu čvrstoću veze (*shear bond strength* - SBS) metalnih i keramičkih ortodontskih bravica vezanih na različitim keramičkim površinama koje se koriste za protetsku restauraciju zuba. Kako bi se moglo utvrditi koje metode i koji materijali imaju najveći postotak uspješnosti, analizirat će se: utjecaj vrste keramike koja se koristi za protetsku restauraciju na posmičnu čvrstoću veze ortodontskih bravica; učinkovitost vezivnih podloga ortodontskih bravica ovisno o vrsti materijala od kojih se sastoje; utjecaj različitih materijala za jetkanje i silana na pripremu keramičkih površina. Daljnji cilj istraživanja jest da se izbjegne jetkanje fluorovodičnom kiselinom, koja je vrlo štetna, te uvode jetkanje 37% fosfornom kiselinom kao mogući protokol prilikom obrade keramičke površine.

Materijali i postupci: Istraživanje je provedeno na 144 keramičkih pripremljenih uzoraka (semi-krunice), ugrađenih na dvokomponentnom epoksi materijalu. Uzorci su izrađeni u obliku krunica gornjih pretkutnjaka koji na obje strane imaju bukalnu površinu, kako bi se na svaku stranu mogla zalijepiti bravica. Uzorak je jednako podijeljen na slijedeće: 48 metalkeramičkih (PFM) na

bazi feldspara, 48 cirkonskih i 48 litij-disilikatnih uzoraka na kojima je ljepljeno 144 ortodontskih bravica: 72 metalne i 72 keramičke polikristaliničke bravice. Priprema površine uzoraka obavljena je korištenjem dviju različitih vrsta materijala za jetkanje uz primjenu 5% fluorovodične kiseline (HFA) ili 37% fosforne kiseline tijekom 120 sekundi, a onda je primijenjen silan. Vezivanje bravica je provedeno pomoću adheziva na bazi kompozita. Nakon polimerizacije LED lampom, uzorci su preneseni na termocikliranje. Istraživanje je uključivalo 12 različitih skupina, podijeljenih ovisno o materijalu od kojeg se sastoje ortodontske bravice i keramičke krunice, kao i pripreme podloge. SBS je ispitan pomoću univerzalnog stroja za ispitivanje materijala - kidalice, uz opterećenje usmjereno paralelno s bukalnom površinom restauracije u gingivookluzalnom smjeru, koristeći oštricu kidalice na 1 mm/min, do granice pucanja. Sila potrebna za odljepljivanje bravica zabilježena je u Newton mjernoj jedinici, a SBS se izračunavao u jedinicama MPa. Nakon ispitivanja posmične čvrstoće, uzorci su analizirani pod digitalnim mikroskopom i SEM-om, kako bi se procijenila vrsta kvara obveznice na dodirnoj površini bravice i ljepila u svakoj testiranoj skupini, i da bi se mogao vidjeti preostali adhezivni materijal i stanje keramike nakon uklanjanja bravica. Za to je određen Adhesive Remnant Index (ARI; po metodi Bishara et al. 1999) (12), i Porculan Fraktura Index (PFI, Bourke i Rock, 1999) (8). Statistička obrada podataka testirana je hi-kvadrat testom. Razina značajnosti postavljena je na $\alpha = 0.05$.

Rezultati: Prema rezultatima analize, glavni efekti (T-CER, T-BRA i T-ETC) ne utječu značajno na formiranje prosječnih vrijednosti SBS-a. Također, trostruka interakcija faktora ne utječe statistički značajno na formiranje vrijednosti SBS-a. Međutim, *post hoc* analiza LSD metodom otkriva šest parova poduzoraka, od ukupno 66 mogućih, koji se statistički značajno razlikuju po SBS-u. Dobivena je: statistički značajna razlika uzorka feldspar keramike s metalnom bravicom i jetkanjem PHA (mean SBS = 9.89 MPa) i iste keramike s keramičkom bravicom i jetkanja HFA (mean SBS = 14.75 MPa) što znači bolji učinak navedene kombinacije; statistički značajno veći SBS feldspara (mean SBS = 14.10 MPa) u odnosu na cirkon (mean SBS = 8.52 MPa) pod istim uvjetima, tj. u kombinaciji s keramičkom bravicom uz PHA pripremu podloge; statistički značajna razlika kombinacije lithium disilikat keramike s metalnom bravicom uz PHA pripremu podloge (mean SBS = 10.20 MPa) od kombinacije feldspara s keramičkom bravicom uz HFA pripremu podloge (mean SBS = 14.75 MPa); statistički značajno zaostajanje cirkonija u kombinaciju s keramičkom bravicom uz PHA pripremu podloge (mean SBS = 8.52 MPa) od feldspar keramike u kombinaciji s keramičkom bravicom uz HFA pripremu podloge (mean SBS = 14.75 MPa); statistički značajno različiti uzorci po SBS-u i to cirkonij u kombinaciji s keramičkom bravicom na HFA podlozi (mean SBS = 8.99 MPa) te feldspar u kombinaciji s keramičkom bravicom i PHA pripremom podloge (mean SBS = 14.10 MPa); i statistički značajna razlika SBS-a za kombinaciju cirkonija s keramičkom bravicom na HFA pripremi podloge (mean SBS = 8.99 MPa) i kombinacije feldspara također s keramičkom bravicom na HFA podlozi (mean SBS = 14.75 MPa). Prema LSD testu razlika između mogućih parova uzoraka tipova keramike statistički je značajna između feldspara i cirkonija (p=0.042). SBS za feldspar u prosjeku je veći od ostala dva materijala, a od cirkonija je i značajno veći (p=0.042). SBS se po tipu bravice ne razlikuje statistički značajno jer su oba prosjeka gotovo jednaka i procjenjuju se unutar prekrivajućih intervala pouzdanosti 95%. Isti je slučaj i s jetkanjem površine što je vidljivo iz rezultata LSD testa. Od dvostrukih interakcija samo interaktivni utjecaj T-CER i T-BRA statistički značajno utječe na formiranje vrijednosti SBS-a (p = 0.016). Detalji te značajne interakcije također su testirane LSD testom za svih šest parova uzoraka tipa keramike i tipa bravice. U slučaju tri para uzoraka razlika SBS-a je statistički značajna. SBS se statistički značajno razlikuje za feldspar u zavisnosti od tipa bravice (p = 0.013). Naime, prosjek SBS-a primjenom feldspara s metalnom bravicom iznosi 10.36 MPa, a s keramičkom bravicom statistički značajno više, 14.43 MPa. Cirkonij u kombinaciji s keramičkom bravicom postiže SBS u prosjeku 8.75 MPa što je statistički značajno manje (p = 0.001) od 14.43 MPa koju postiže feldspar također s keramičkom bravicom. SBS vrijednosti feldspara u kombinaciji s keramičkom bravicom je statistički značajno manji i učinak litium disilicata od 11.08 MPa u kombinaciji s metalnom bravicom (p = 0.040). Interaktivno djelovanje tipa keramike na formiranje vrijednosti SBS-a u kombinaciji s pripremom podloge nije statistički značajno ni u jednom od šest mogućih parova. Međutim, uočljiv je odmak feldspara od ostala dva keramička materijala i to podjednako za oba načina jetkanja. ARI ne zavisi statistički značajno o tipu keramike niti o pripremi podloge, no tipovi bravica statistički značajno utječu na pojavljivanje kategorija ARI-ja jer se prve dvije kategorije (1 i 2) ARI-ja značajno više pojavljuju uz metalne bravice, a 3. i 4. kategorija ARI-ja

učestalija je kod keramičkih bravica. Od mogućih zavisnosti PFI-ja od triju faktora pokusa, hi-kvadrat testom nađena je samo statistički značajna zavisnost o tipu jetkanja ($p = 0.048$).

Zaključak: Upotreba HFA za površinsku obradu feldspata, cirkonija i/ili litij disilikata ne uzrokuje značajno povećanje vrijednosti SBS-a u usporedbi s jetkanjem primjenom PHA i silana, što je sigurnija metoda za upotrebu u kliničkim uvjetima. Nadalje, HFA može oslabiti površinsku strukturu keramike, a uzevši u obzir njezin štetni učinak, ne mora biti najbolje sredstvo prije ortodontskog vezanja na feldspat, litij disilikat, a naročito na cirkonijev oksid, posebice uzimajući u obzir njegovu kristalnu strukturu.

Ključne riječi: Feldspat, Cirkonij, Litij disilikat, Ortodontske bravice, PHA, HFA, SBS, ARI, PFI.

List of abbreviations

Abbreviation	Term
3Y-TZP	Yttria-tetragonal zirconia polycrystal
Al_2O_3	Aluminium oxide - alumina
APF	Acidulated phosphate fluoride
ARI	Adhesive remnant index
CAD/CAM	Computer aided design / Computer aided manufacture
CaO	Calcium oxide - calcia
GIC	Glass ionomer cement
HFA	Hydrofluoric acid
K_2O	Potassium oxide
LED	Light-emitting diode
$Li_2Si_2O_5$	Lithium disilicate
MPa	Megapascal
N	Newton
Na_2O	Sodium oxide
OH	Hydroxyl
PFI	Porcelain fracture index
PFM	Porcelain fused to metal
PHA	Phosphoric acid
SBS	Shear bond strength
SEM	Scanning electron microscope
Si_3N_4	Silicon nitride
SiO_2	Silicon dioxide - silica
T-BRA	Type of bracket
T-CER	Type of ceramic
T-ETC	Type of etchant
ZrO_2	Zirconium dioxide

Contents

1.INTRODUCTION

1.1 Orthodontic bonding to various materials

In the very beginning of the history of orthodontic fixed-appliances, brackets were welded to bands made of gold or stainless steel. The bands were placed circumferentially to the teeth, which required the creation of approximal spaces between each tooth in order to accommodate the bands. The separation process was done by placing wires or elastics between the teeth which was time-consuming for the orthodontist and very uncomfortable for the patient. At the end of the treatment, these spaces had to be closed again. Bands often were the cause of soft tissue irritation and plaque formation, causing enamel decalcification under the band as a consequence of difficulties in keeping a good oral hygiene (1).

With the introduction of the acid-etching bonding technique by Buonocore in 1955, and with the later improvements made by Newman, direct bonding of orthodontic brackets became an accepted clinical technique, and since then it has been highly advanced (2–4). This approach, which mainly eliminated the use of bands on every tooth, provided better conditions for oral hygiene and enabled other numerous advantages like decreased plaque formation, gingival inflammation, more aesthetic appearance, and made the placement of orthodontic appliances more comfortable for patients and orthodontists (2,5,6).

The sufficient bracket bond strength is essential for orthodontic treatment, which means that bonded brackets have to withstand the forces of occlusion during the treatment, mastication, and arch wire stress while allowing for biomechanical control (5,7). The bracket bond must resist these multiple forces in the complex oral environment, within the moisture and the rapid temperature and pH changes. The poor bond strength and repeated bond failures result in increased treatment time and cost for the orthodontist and for the patient (7).

The adult dentition is often restored with composites, alloys and ceramics in the form of fillings, veneers and crowns resulting in different bonding requirements. Due to the increase in the number of adults seeking orthodontic treatment, new challenges have appeared for orthodontists, such as the need to bond orthodontic brackets to various restorations (8–10). Furthermore, aesthetics is an important factor for adult patients, therefore using bands in restored teeth is not an option, so bonding orthodontic brackets accurately to restorations should be achieved (5,10).

It was reported that the bond strength of brackets to various restorations and the failure model depend on many factors, such as: restoration material and its surface conditioning, bracket material and its retention mode, properties of the bonding adhesive, and the light-curing source (11). Additionally, there are numerous other factors that can potentially contribute to the bond strength, including the type of conditioner, acid concentration, length of etching time, composition of the adhesive, light-curing device, the oral environment, as well as the skill of the clinician (2).

Hereupon, the combination of different materials and products may remain decisive for clinically successful orthodontic treatment (11).

The most widely used materials to restore damaged or missing teeth are ceramics. Dental ceramics are deployed as veneers, crowns or bridges because of their excellent aesthetics, mechanical properties and biocompatibility (12).

However, bonding to ceramics, whose glazed surface is not amenable to resin penetration, has a higher degree of failure compared to bonding to enamel (11).

Consequently, it became necessary to find a reliable method for bonding orthodontic brackets onto ceramic crowns, in order to obtain sufficient bond strength. On the other hand, because generally the restoration is requested to remain in the mouth after debonding, the bond strength should be adequate for easy and safe bracket removal in order to avoid the possibilities of damaging the restoration surface (8,9,11–13).

A mechanical or chemical method other than the etching protocol on the enamel must be applied on different types of ceramics, in order to reduce difficulties in treating adults with fixed orthodontic appliances. Several techniques have been presented for bonding the brackets to ceramic surfaces which differ in surface preparation and bonding agent applied. Some of these use phosphoric acid (PHA), some hydrofluoric acid (HFA) or acidulated phosphate fluoride (APF), while others use mechanical roughening procedures like sandblasting, diamond stone burs etc. (8,12–16).

With the introduction of the laser, the idea of using it as a means of reinforcing bond strength has become popular. Although it has been evaluated by extensive research, the efficacy of different types of lasers on porcelain surface conditioning remains controversial (12,17).

To date manufacturers have presented a wide range of orthodontic bonding systems, such as the adhesives with different molecules of resin and many products for surface treatment before bonding (2,11). Overall, orthodontic adhesives are either composite resin or glass ionomer cement (GIC) based materials. Some of them are more successful in achieving sufficient bond strength in ceramic restorations (18,19).

1.2 **Dental Ceramics**

Various types of dental ceramics have been developed as the restorative materials. They differ in chemical composition, physical properties, and manufacturing method (20–22). Consequently, when bonding orthodontic brackets, the type of ceramic used in the restorations also has to be considered (6,12,20,23).

The American Ceramic Society defines ceramics as inorganic, non-metallic materials, which are typically crystalline in nature, and which are compounds formed between metallic and non-metallic elements such as aluminium and oxygen (aluminium oxide / alumina - Al_2O_3), calcium and oxygen (Calcium oxide / calcia - CaO), silicon and nitrogen (silicon nitride - Si_3N_4).

The term porcelain refers to a specific compositional range of white, translucent ceramic materials made by mixing kaolin, quartz and feldspar and fired at a high temperature to a glazed state (24).

Dental ceramics are chemically inert in the oral cavity and they also exhibit very good biocompatibility with the oral soft tissues. They possess excellent aesthetics, and the structure of porcelain restoration is probably their most important mechanical property (24).

Ceramics can appear as either crystalline or amorphous solids - glasses. The mechanical and optical properties of dental ceramics mainly depend on the nature and the percentage of those two phases. More the glassy phase more the translucency of ceramics, however, it weakens the structure by decreasing the resistance to crack propagation, and more the crystalline phase, better will be the mechanical properties, which in turn may alter the aesthetics (24).

Dental ceramics are classified due to their crystalline phase and fabrication technique (25). They vary in chemical composition, method of manufacture, and physical properties and they are divided in silicate (non-crystalline) ceramics and oxide (crystalline) ceramics (24–26).

Silicate (non-crystalline) ceramics have high contents of glass matrix in which crystalline particles are distributed. They are divided in feldspathic ceramic and glass ceramic (24).

Feldspathic porcelain is made of the mineral feldspar with additions for colour and translucency, and it contains silica (silicone dioxide - SiO_2) and alumina (aluminium oxide - Al_2O_3) with little potassium oxide (K_2O) and sodium oxide (Na_2O) for expansion control (27). Feldspar alone exhibits a low coefficient of thermal expansion, and with the addition of leucite the production of veneering ceramics was made possible with a coefficient of thermal expansion compatible with that of the metal substructure (25).

The improvement of flexural strength of the glass ceramic is achieved by adding materials that strengthen the glass ceramic, such as lithium-disilicate (28).

Oxide (crystalline) ceramics are more crystalline, and they are more frequently used as a core material for silicate ceramic. There are two types of them: aluminium and zirconium oxide ceramics. Alumina-reinforced porcelain is produced by dispersing high-strength alumina crystals in feldspathic matrix, which results in five times stronger ceramic than porcelain (25).

Zirconium does not exist in nature in its pure state, but only as a free oxide (zirconium dioxide - ZrO_2) or in conjunction with silicate (ZrO_2+SiO_2) also known as zirconia (29). Partially stabilized zirconia, because of its unexcelled mechanical properties, expanded the range of applications of ceramics in dentistry, a field where they are constantly in demand because of their chemical inertness and good optical properties (25,30).

Although dental ceramics are produced in numerous compositions, nowadays the most widely used are feldspar based porcelain fused to metal (PFM) and all-ceramic materials like zirconia (Yttria-Tetragonal Zirconia Polycrystal / 3Y-TZP) and lithium disilicate glass-ceramic ($Li_2Si_2O_5$) (25).

Porcelain fused to metal (PFM) systems for dental restorations have been available since the 1960s, and they rely on the application and firing of a veneering ceramic onto a metal substructure to produce an aesthetically acceptable restoration. Veneering ceramics for metal-ceramic restorations are commonly named feldspathic porcelains, and are usually leucite-based (25).

Metal-free materials used as dental restorations have been in the spotlight of recent research, following the development of ceramic systems and the introduction of innovative all-ceramic materials (26,31). During the recent years, the technological evolution of all-ceramic restorations for dental applications has been remarkable, as new materials and processing techniques are steadily being introduced, so their popularity has increased due to their superior aesthetic appearance and metal-free structure (31,32).

After the huge development of the CAD/CAM (Computer aided design / Computer aided manufacture) technology, zirconia became one of the most interesting materials to be examined and used in the entire dental field. It can be used as a core for all-ceramic crowns which are layered with porcelain powder, or it can be used for monolithic zirconia crowns (33–35). Zirconia is a widely used core for all-ceramic crowns due to its high strength and aesthetic appearance. This is accomplished when a veneer is layered with ceramic powder onto the zirconium core. Since fracturing of the veneer is frequently reported in the posterior teeth, because of the strong masticator forces (36,37), an increased use of monolithic zirconium crowns without veneers is encouraged (30,38). In orthodontic patients with monolithic zirconia crowns, the orthodontic bracket should be bonded directly onto the zirconium surface (10).

Another all-ceramic material that combines very good mechanical properties and excellent aesthetic results is lithium disilicate glass ceramic, known as e-max, which can be produced as CAD or Press (25,38,39).

1.3 **Surface conditioning of the ceramic**

Ceramic is an inert material, and it does not adhere chemically to any of the currently available bonding resins (20). Consequently, orthodontic brackets bond poorly to ceramic surfaces, unless the surface characteristics of the ceramic are altered through certain approaches before bonding (40). A mechanical or chemical method other than the etching protocol on the enamel must be followed in ceramics, in order to avoid the difficulties in treating adults with fixed orthodontic appliances. Numerous approaches have been reported in the literature, which can be classified into three major groups: mechanical, chemical, or a combination (20).

Generally, the ceramic restoration remains in the mouth after debonding of the bracket. Therefore, damaging of the ceramic due to extreme roughening of the surface during pre-treatment or debonding must be avoided (41).

Type of ceramic used as restoration can be a decisive factor for bond strength of orthodontic brackets and for the method of choice for surface altering of the restoration (23).

In the past, bonding to traditional feldspathic ceramics has been well-studied and various surface treatment methods of the ceramic surface have been used, including diamond burs, sandblasting, hydrofluoric acid (HFA), phosphoric acid (PHA), acidulated phosphate fluoride (APF) laser etching, etc., exposing their advantages and disadvantages (5,13,27,42–45). There is no clear consensus on the ideal bonding system, but a recent systematic review has shown that there are several bonding methods that can be acceptable (9). The most commonly employed method is etching with HFA (46,47). However, HFA is a strong acidic solution that should be applied with extreme caution avoiding the contact with the soft tissues (13,44,48).

Due to the potential toxicity of HFA, Nelson and Barghi (1989) (49) suggested that the application of 1.23 % APF for 10 minutes results in an effective bond strength similar to HFA applied for 1 minute. On the other hand, etching ceramic surfaces with 37 % PHA was reported to produce a clinically acceptable bond strength comparable with that produced by the application of HFA (12,13,20).

Mechanical removal of the glazed surface of the ceramic with diamond burs, as well as with sandblasting aluminium-oxide particles in high pressure, can enhance bond strength, but can also reduce ceramic integrity, which could lead to cracks and larger damages during debonding (20,41,50).

Obtaining a sufficient bond strength seems to be difficult when using only a mechanical etching procedure with diamond stone burs, sandblasting, or sandpaper discs, and all these procedures damage the glazed surface of restoration (12,44,51,52). For bonding to glazed ceramics, a combination with a chemical preparation of the ceramic surface with acids and a coupling agent silane is recommended (13,14,51).

Conditioning with some lasers has also been investigated as a promising technique for the surface treatment of various types of ceramics by a number of studies, but with not so satisfactory results (53–55).

Surface preparation before bonding has two goals: to remove surface contaminants and to increase the surface area of the substrate. Removal of surface contaminants increases the free surface energy of the substrate to be bonded relative to the surface liquid interface. Additionally, the liquid results in a decreased contact angle and increased surface wetting by the adhesive. Increases in the surface area can be accomplished through different means including HFA etching, sandblasting, and lasers, with the goal of creating a larger bonding surface and micromechanical retention for the adhesive (56,57).

Previous studies have shown that chemical conditioning using silane increases the adhesion of the composite resin bond to the ceramic surface (8,13,27,41,51). Silane is a bi-functional molecule capable of forming a chemical link between the hydroxyl (OH) of the silica on the ceramic surface with the resinous matrix of the composite (20,51,58). However, with the increase of the crystalline phase in the content of the ceramic, this chemical reaction becomes less efficient because of lower levels of silica (58).

Consequently, the question arises which procedure should be applied for bonding orthodontic brackets to various ceramic restorations (12). It has been recommended that methods providing a sufficient bond with less roughening should be used in order to avoid microcracks of the ceramic surfaces (20,23).

1.3.1 Hydrofluoric acid conditioning

Hydrofluoric acid (HFA), best known for its ability to dissolve glass, is mostly applied at concentrations of 5-9.6% for a period of 120 seconds to ensure optimal bond strength. HFA etching creates a porous surface by removing the glassy matrix (58), and has been shown to result in acceptable bond-strength values in porcelain (5,27,44), but it is less successful in more crystalline rich ceramics (3,10).

However, gingival barriers should be used to eliminate the negative effects of HFA in gingival tissues before application (13,14). The risk from handling with hydrofluoric acid in high concentrations is extreme, as it will also quickly destroy the corneas of the eyes (12). Also, the danger of acid burns is very high, and it can result in deep tissue necrosis (48).

The hazards related to the intraoral use of HFA have been known for some time and were presented for the first time by Moore (52). They include soft-tissue burns and both soft- and hard-tissue necrosis (12).

In addition to the harmful biological effects, etching with HFA is a destructive process for the ceramic surface through its chemical reaction with silica and it necessitates revision of the restoration (6). Also, given the high bond strengths obtained through HFA etching, bond failure is often cohesive within the ceramics, resulting in a greater risk for permanent damage to the ceramic surface (8).

1.3.2 Phosphoric acid conditioning

Surface conditioning of the ceramic with 37% phosphoric acid (PHA) has been suggested as an alternative to HFA by several authors in attempts to promote adhesive failure due to expected lower bond strengths and therefore decrease the risk to the ceramic surface (12,13).

Application of PHA to the ceramic surface is non-destructive and has been shown to simplify the process of residual bond clean-up (59). It cannot erode superficial layers of silicate ceramic (13,27,42). On the other hand, it has the ability to neutralize the alkalinity of the absorbed water layer, which is present on ceramic restorations in the mouth and thereby improve the chemical activity of the silane primer that is subsequently applied (12).

Also, PHA is not toxic or corrosive, and in combination with silane achieves satisfactory bond strength (12,13). Despite that and the tendency towards adhesive failure that was obtained in the in-vitro studies, it is unknown if ceramic surface conditioning with PHA will be sufficient for the long-term stresses encountered in the oral environment. Therefore, it has been recommended to employ longer term storage times or thermocycling in order to accurately assess the usefulness of PHA as a surface conditioner (18).

1.3.3 Application of silane

Previous studies have shown that chemical conditioning methods, such as silanation, increase the adhesion of the composite resin bond to the ceramic (8,13,41,44,51).

Silane coupling agent is often recommended following etching materials as part of the conditioning of the ceramic surface prior to procedures for adhesive application.

Silane is a bi-functional molecule capable of altering the chemical structure of silica-based ceramic surface by making a chemical link between the hydroxyl (OH) of the silica with the resinous matrix of the composite (20,51,58). Thus alkene groups are bonded covalently to the inorganic surface resulting in a surface that is compatible with the organic adhesive (60). This results in a chemical link that provides sustainable bracket bond strength, where the silica of the dental ceramic is chemically united with the acrylic group of the composite resin through silanation (41).

However, the results are contradictory, showing that using silane with HFA does not increase the bond strength (41). Also, with the increase of the crystalline phase in content of the ceramic, this chemical reaction becomes less efficient, because of lower levels of silica (58).

1.3.4 Application of adhesives

Nowadays, a wide range of orthodontic bonding systems, such as the adhesives with different chemical structure and physical properties are presented. Overall, orthodontic adhesives are either composite resin or glass ionomer cement (GIC) based materials (11,14,61).

Composite resins are used to attach orthodontic brackets to enamel or ceramic surfaces. The protocol comprises a series of technique-sensitive steps and failures with composite resins have been mostly attributed to moisture contamination (14).

Other materials used to attach brackets to enamel or ceramic surfaces are resin-modified glass ionomer cements. They have cariostatic properties due to a slow release of fluoride at low levels over an extended period (2)

However, it has been extensively demonstrated that glass ionomer cements are associated with a significantly lower bond strength than composite resins (2,14).

On the basis of a current literature review, bonding systems were categorized as clinically acceptable if they had a shear bond strength of 6 to 8 megapascals (MPa) (1,9).

1.4 Orthodontic brackets, consisting material and retention mode

The material of the bracket and its base surface design or the retention mode (Figure 1), should also be considered when bonding brackets to ceramic surfaces (42,62,63).

Some investigations have found that the shear bond strength (SBS) of ceramic brackets is higher than that of metallic brackets because of the stronger adhesion obtained with ceramic brackets. This higher bond strength of ceramic brackets is due to the increased light availability for photo-polymerization because of greater light transmission, resulting in a higher degree of polymerization and reduced stresses at the adhesive/bracket interface (11,20,42,51,61,64). This is also due to a different failure mode because of the flexibility of the metal base (62,64).

Metallic brackets base design has been in the focus of the manufacturers considerably in recent years in the attempt to improve bond strength and to reduce base sizes (62,64).

There are various bracket base designs, all in an attempt to optimize the mechanical bond between the bracket and the adhesive. The design of the bracket base adhesive pad has been found to be a significant factor in mean shear bond strength (65). Three quarters of brackets with a foil mesh base undergo bond failure at the bracket adhesive interface (66). Currently most stainless steel orthodontic bracket base designs have a fine mesh adhesive surface (62).

It has been reported that mesh based brackets with larger mesh spaces provide a greater shear bond strength than do bases with smaller mesh spaces (62).

Another concern is the allergic and cytotoxic effects induced by constituents and the corrosion products of the stainless-steel brackets. Nickel and chromium are the most common causes of metal induced allergic contact dermatitis. Nickel has recently been reported to be moderately cytotoxic (67).

The ceramic brackets have become popular since their introduction to orthodontics in 1986. Since then, their product design and clinical performance has greatly improved. The newer designs of ceramic brackets offer excellent optical properties and the promise of additional aesthetic appeal without significant functional compromises. Their acceptance by adult patients has been unprecedented in the orthodontic practice and it contributed significantly to the expansion and development of contemporary orthodontic therapeutic modalities (61).

Apart from offering aesthetics, ceramic brackets exhibit excellent biocompatibility. Ceramic brackets are mostly polycrystalline alumina and monocrystalline alumina. Another category that is being developed is the zirconium bracket (67).

Optical properties and strength are incompatible for polycrystalline ceramics. The larger the ceramic grains, the greater the clarity or translucency. However, the material tends to become weaker.(67,68).

a.  b. 

Figure 1. Two different brackets in retention mode and consisting material: a. metal; b. polycrystalline ceramic

1.5 **Other factors that influence bond strength**

Other attributes might also be relevant, such as the duration of acid etching of the surface, as well as the concentration of the acid applied, although there is a contradiction in different findings (8,69). Additionally, a light-curing source, thermocycling, as well as other factors influence the bond strength of orthodontic brackets (20).

The etching time required for optimal bonding to ceramic is controversial in the literature. Several authors showed decreased bond strengths with increasing etching times (22,70). On the other hand, other authors showed that extending etching times may increase the bond strength to ceramic (14,71,72).

Numerous types of acid etching solution with variable concentrations have been developed. These include hydrofluoric acid (HFA) gel (27,51), acidulated phosphate fluoride (APF), and phosphoric acid (PHA) gel and solutions (73). The most commonly used ceramic acid etchant is a 9.6 % HFA gel, and its 2–4 minute application on ceramic surface has been advocated (27,44).

Orthodontics has benefitted from the introduction of light-curing devices and light-curing restorative materials in dentistry, and manufacturers have introduced numerous light-cured adhesive systems to bond orthodontic brackets. The greatest advantage of a light-cured adhesive system is that it provides the clinician with ample time to accurately position the bracket on the surface before using the light to accelerate the polymerization of the adhesive (Figure 2) (2).

In-vitro studies do not always resemble to clinical situations. Thermocycling is used to simulate clinical conditions in order to assess the durability of the bond. The difference in storage conditions is one of the critical aspect in such studies (27,41,44). Differences in thermal expansion coefficients among the adhesive, metal bracket and the substrate, as well as microleakage within the bond might affect the bond strength of the bracket to the ceramics, and that is why experimental specimens must be subjected to thermocycling (41).

Another theory involves the absorption or solubility of the composite after thermocycling. This procedure causes hygroscopic expansion, as well as chemical degradation of the materials. It has been shown that thermocycled composites absorb more water than those that were not thermocycled (74).

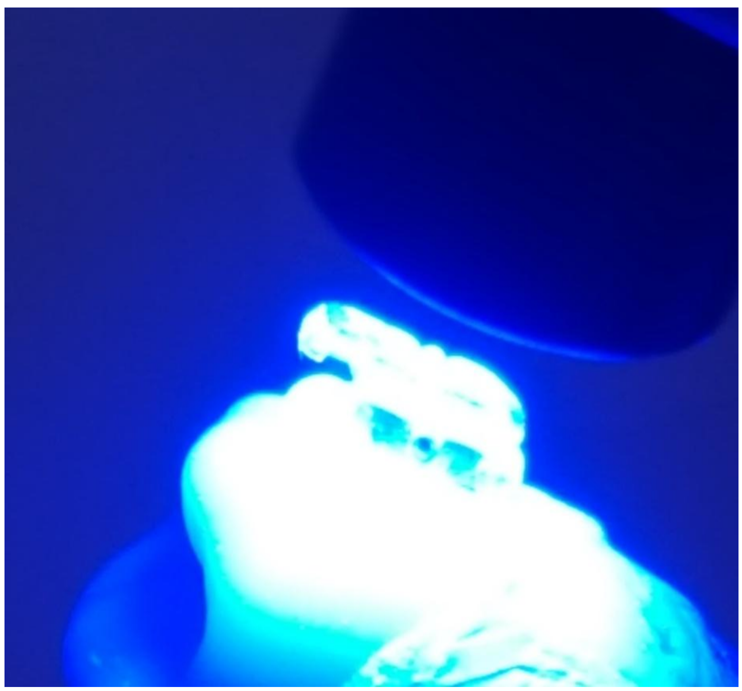


Figure 2. Light curing of the adhesive after positioning of the bracket

1.6 Bracket bond strength and SBS testing

1.6.1 Adequate bond strength

The value of 6 to 8 MPa, proposed by Reynolds is the most commonly cited in the literature as a clinically adequate bond strength (75). However, this recommended bond strength is hypothetical and has been poorly tested. It has no consideration for the complexity of the multiple forces applied by orthodontic mechanics and by mastication acting in the oral environment, or for bond aging (76). Also, bond failure cannot be always prevented by high bond strength. For example, sandblasting of the feldspathic porcelain followed by the application of HFA and silane, has been shown to result in bracket bond strengths above 14 MPa (77), well above the recommended values suggested by Reynolds. However, it has been reported that the clinical incidence of debonding using this method was 8.9% (44), which is comparable to the overall rate of bracket bond failure to enamel 11% (78).

Although it is controversial at what stage ceramic damage can occur when debonding, it is indicated to ensure that bond strength is not so high to avoid cracks and to balance the clinical benefit of increased bond strength with the risk for ceramic damage. The risk of ceramic damage is largely reduced if the bond undergoes an adhesive type of failure, which occurs largely if the bond strength is less than 13 MPa (60).

1.6.2 SBS testing

The strength measurements of various materials are undertaken in laboratories to determine their relationship to the micro-structural features of those materials, the comprehension of which will allow the production of better and stronger materials (79).

Certain in-vitro bond strength testing methods exist using different loading modes, including shear, tension and torsion (80). Although in-vitro studies are alluring due to their simplicity, the results can be controversial and might not be representative of true clinical mechanical stress challenges (81).

Testing methods generally attempt to apply a load along a single axis, but orthodontic bracket bonded as an intraoral setting is subject to six loading components, consisting of three forces and three moments (82).

The shear bond strength (SBS) test is the method in which two materials are connected via an adhesive agent and a shear load is applied until separation occurs (Figure 3). SBS is calculated by dividing the maximum applied force by the bonded cross-sectional area (80).

During the SBS testing, the forces at the bond interface are not homogeneous, exhibiting a more complex pattern of force vectors, including shear forces at the middle of the bracket and compression and tension in the gingival and coronal parts (83).

Also, similar force vector distribution patterns exist for tension and torsion testing. However, tension and shear bond strength tests utilize forces that are orthogonal to each other, while the torsion test utilizes a moment, which cannot be directly compared and can generate very different results for the same bonding protocol. Shear bond strength (SBS) tests alone are highly dependent on several parameters, including crosshead speed and design (60).

Despite limitations, SBS testing remains a relevant methodology to compare bonding protocols by providing important information regarding bracket debonding in clinical situations (82).

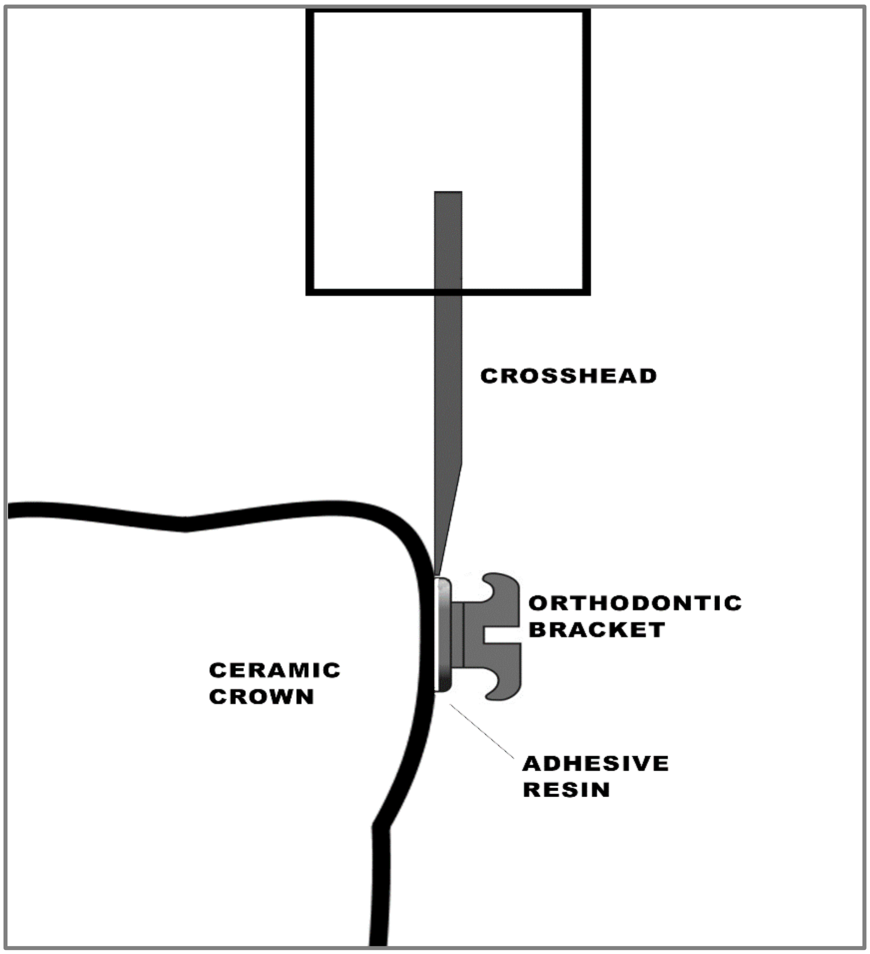


Figure 3. Schematic illustration of SBS testing

1.7 Adhesive Remnant Index and type of bond failure

Bond failure can occur as an adhesive failure on the ceramic surface, as cohesive failure within the adhesive cement, or mixed, involving both adhesive and cohesive failure (11).

Artun developed the Adhesive Remnant Index (ARI) as a four-point scale (84), which was later modified by Bishara to a five-point scale (61) as a way to qualify the type of bracket bond failure and, more importantly, the amount of material that remained on the surface of the tooth upon bracket debonding as follows:

1 - All adhesive remaining on the ceramic crown surface with the impression of the bracket base;

2 - More than 90% of the adhesive remaining on the ceramic crown surface;

3 - Less than 90%, but more than 10% of the adhesive remaining on the surface;

4 - Less than 10% of the adhesive remaining on the ceramic crown surface; and

5 - No adhesive remaining on the ceramic crown surface.

This has been regarded as a useful tool when combined with the actual bond strength data to better interpret bracket bond findings regarding the benefits of high versus low bond strength and the need for post-debonding procedures to clean the bonded surface. While this index was developed for bonding to enamel, it can and has been used to qualify bracket debonds to other substrates (46,85,86).

1.8 Porcelain Fracture Index

It is very important not to damage the integrity of ceramic crowns after debonding of orthodontic brackets, which could lead to cracks and larger damages. These damages to the ceramic surface which may have occurred during shear bond testing are recorded by using Porcelain Fracture Index (PFI) (13). The index is divided into four scores as follows:

0 - ceramic surface intact or in the same condition as before the bonding procedure;

1 - surface damage limited to glaze layer or very superficial ceramic;

2 - surface damage which features a significant loss of ceramic requiring restoration of the defect by composite resin or replacement of the restoration;

3 - surface damage where the core material has been exposed due to the depth of the cohesive failure.

2. AIM AND HYPOTHESES

2.1 Research objective

Currently, there is no consensus regarding the most efficient ceramic surface conditioning method for producing optimal bond strength of orthodontic brackets to different ceramic materials.

The aim of this study is to conduct an inclusive and substantial analysis of the factors affecting shear bond strength (SBS) of metallic and ceramic orthodontic brackets bonded to different ceramic surfaces used for prosthetic restorations. In order to determine which materials and techniques present the highest success rate, the following will be analysed:

1) the influence of the type of the ceramic used for prosthetic restoration, on the shear bond strength of the orthodontic brackets,

2) the influence of various etching materials and silane on the conditioning of the ceramic surfaces,

3) the effectiveness of bonding surfaces of orthodontic brackets depending on the type of material from which they consist,

4) the mode of adhesive failure after debonding, by assessing the Adhesive Remnant Index (ARI), and

5) the condition of the ceramic surface after debonding, by measuring the Porcelain Fracture Index (PFI).

Additionally, a further objective of this research is to overcome the etching with hydrofluoric acid, which is very noxious, with silane coupling application and etching with 37% phosphoric acid as pre-treatment procedures of the ceramic surface before bonding.

2.2 Research hypotheses

Null hypothesis 1: The type of ceramic does not affect the shear bond strength of orthodontic brackets bonded to ceramic surfaces.

Alternative hypothesis 1: The SBS is affected by the type of ceramic to which orthodontic brackets are bonded.

Null hypothesis 2: SBS of orthodontic brackets is not affected by the type of etchant, and phosphoric acid in combination with silane is a reliable conditioning alternative for all types of ceramic surfaces before bonding.

Alternative hypothesis 2: Shear bond strength of orthodontic brackets is affected by the type of etchant applied, and HFA is more efficient and should be obtained as protocol for conditioning ceramic surfaces before bonding.

Null hypothesis 3: The type of bracket does not affect their SBS when they are bonded to ceramic surfaces.

Alternative hypothesis 3: The SBS is affected by the material the bracket is made of.

3. MATERIALS AND METHODS

3.1 Materials and sampling

This research was conducted in order to investigate the shear bond strength (SBS), depending on the different ceramic materials, the different bracket materials and the ceramic surface preparation, as well as investigating the remnant after debonding and the condition of the ceramic surface.

The research was conducted on 144 ceramic glazed samples (semi-crowns). The samples were prepared from three different ceramics in equal numbers: 48 of them from feldspar-based ceramic (VITA Zahnfabrik, Bad Säckingen, Germany) in the form of porcelain fused to metal (PFM), 48 from full-contour zirconia (Copran Zr-i Monolith, White Peaks Dental Solutions GmbH&Co.KG, Wesel, Essen, Germany), and other 48 from lithium disilicate (IPS EMAX CAD, Ivoclar Vivadent AG Schaan, Lichtenstein).

The specimens were produced by the same technician, in the shape of maxillary premolars with two sides buccal surfaces, 72 in number. Subsequently, they were embedded in a two-component epoxy filling (Epoxy Repair, Bison International, Goes, The Netherlands) in a metallic rod. After this procedure, the specimens were washed with alcohol (95%) and distilled water.

On the one half of the sample metallic orthodontic brackets (Mini 2000 Ormco Corp., Glendora, California, USA) were bonded, and on the other half polycrystalline ceramic orthodontic brackets (Glam Forestadent, Bernhard Forster GmbH, Pforzheim, Germany) were bonded (Figure 4).

Figure 4. The prepared specimen.

Two different etching materials were used for conditioning the ceramic surface: hydrofluoric acid 5% (IPS Ceramic Etching Gel, Ivoclar Vivadent AG, Schaan, Lichtenstein) or phosphoric acid 37% (Etching solution, Ormco Corp., Glendora, CA, USA) for 120 seconds, and subsequently silane (Prosil, Dentscare, Joinville, Brasil) was applied.

For bracket bonding, two-component (primer and adhesive) composite resin-based bonding system (Tranbond XT, 3M/Unitek, Monrovia, CA, USA) was used (Figure 5).

All brackets were bonded by the same operator and positioned in the middle of the prepared surfaces of the ceramic sample. They were pressed firmly, and the excess adhesive was removed from around the bracket base using a dental probe.

The adhesive was light cured for 40 seconds, using a light-emitting diode (LED; Ledition, Ivoclar Vivadent AG, Schaan, Lichtenstein) (Figure 6).

Figure 5. The bonding system used for attaching the brackets to ceramic surfaces - Tranbond XT, 3M/Unitek.

Figure 6. LED used for light curing the adhesive - Ledition, Ivoclar Vivadent.

Additionally, the specimens were thermocycled for 5800 cycles, 5ºC to 55ºC in distilled water, with 10 s dwelling time, in order to simulate the moisture in the oral environment (Figure 7).

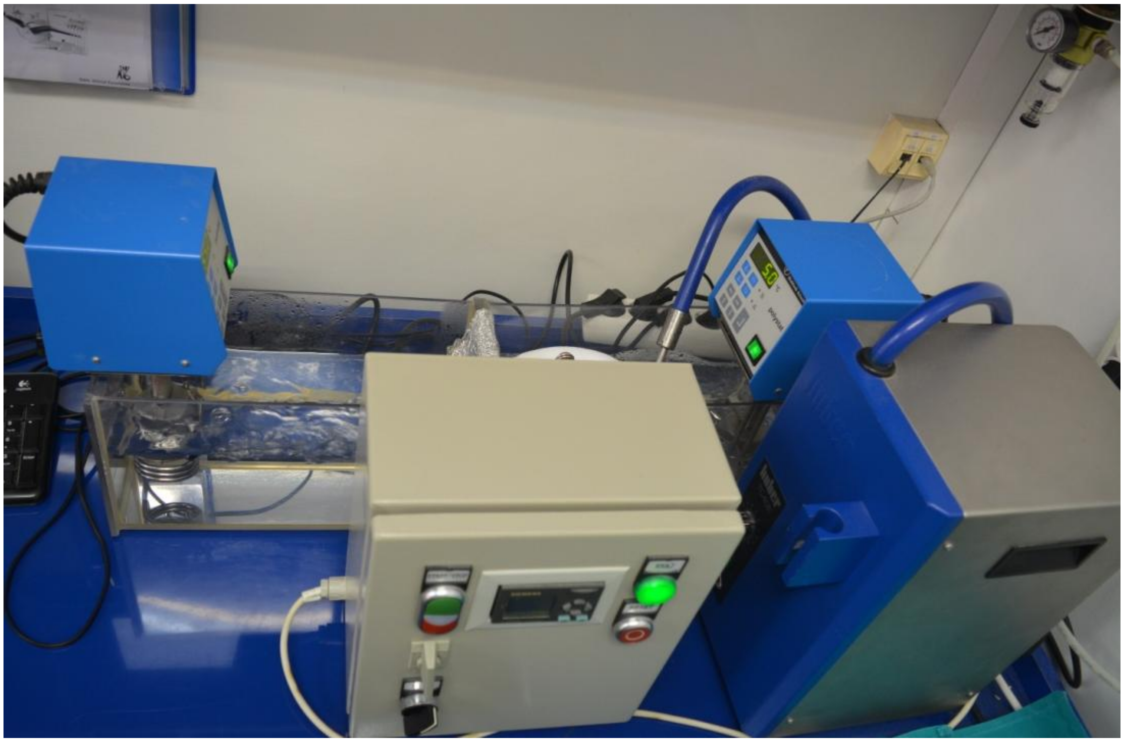


Figure 7. Thermocycling device used to simulate the oral environment.

The research sample was equally divided in 12 groups (Figure 8):

1. Feldspar surface etched with PHA, and metallic bracket bonded;
2. Feldspar surface etched with HFA, and metallic bracket bonded;
3. Feldspar surface etched with PHA, and ceramic bracket bonded;
4. Feldspar surface etched with HFA, and ceramic bracket bonded;
5. Zirconia surface etched with PHA, and metallic bracket bonded;
6. Zirconia surface etched with HFA, and metallic bracket bonded;

7. Zirconia surface etched with PHA, and ceramic bracket bonded;
8. Zirconia surface etched with HFA, and ceramic bracket bonded;
9. Lithium disilicate surface etched with PHA, and metallic bracket bonded;
10. Lithium disilicate surface etched with HFA, and metallic bracket bonded;
11. Lithium disilicate surface etched with PHA, and ceramic bracket bonded;
12. Lithium disilicate surface etched with HFA, and ceramic bracket bonded.

Figure 8. Part of the research sample.

3.2 **Methodology - SBS testing**

The shear bond strength was tested with Universal Testing Machine (Erichsen 0-2000 N, ISO 7500-1:1, AM Erichsen GmbH&Co.KG, Hemer-Sundwig, Germany) (Figure 9), with a load applied parallel to the buccal surface of the restoration in a gingivo-occlusal direction, using a knife-edged rod moving at a fixed rate of 1 mm/minute, until failure occurred (Figure 10). The force required to debond the brackets was recorded in Newton (N) (Figure 11), and the SBS values were calculated in MPa.

Figure 9. Universal Testing Machine - Erichsen 0-2000 N.

Each test was conducted 12 times, in order to examine the impact of the three types of ceramics, two different etching methods and two types of brackets on the SBS, ARI and PFI

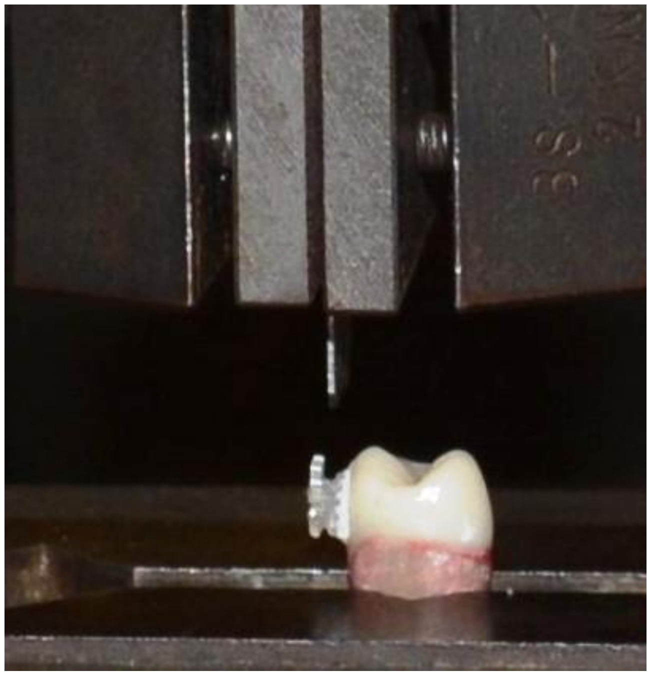


Figure 10. SBS testing

Figure 11. The force required to debond the brackets was recorded in Newton.

3.3 Methodology - Evaluation under digital microscope and SEM

In addition, the samples were analysed using a Digital Microscope (Dino-Lite, ANMO Electronics Corp., Taiwan) and Scanning Electron Microscope (SEM; Tescan Vega TS5136MM, Chez Rep) (Figure 12), in order to evaluate the type of bond failure at the bracket-adhesive interface in each test group and to visualize the adhesive remnant and ceramic condition after the removal of the brackets.

Figure 12. Scanning Electron Microscope - Tescan Vega.

Before SEM, the samples were dehydrated over a period of 5 hours in increasing concentrations of alcohol (70% and 95%). Subsequently, ceramic surfaces were coated with palladium and gold (Figure 14) with a sputter coater (SC7620 Mini Sputter Coater, Quorum Technologies Ltd, UK), because they are nonconductive materials and in order not to lose the electrons (Figure 13), and then they were examined under a field emission of SEM. The SEM photomicrographs were taken for visual inspection (in 400 and 1000 magnification; HV: 20.0 kV).

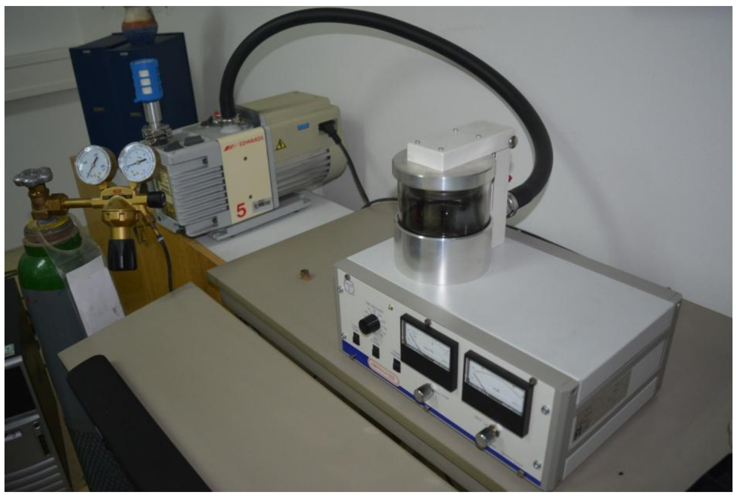


Figure 13. Mini Sputter Coater, Quorum Technologies.

Figure 14. The samples after sputtering with palladium/gold coater.

To determine the adhesive remnant index (ARI; as per Bishara et al.) (61), the measurements were performed, using scores varying from 1 to 5:

1 - All adhesive remaining on the ceramic crown surface with the impression of the bracket base;

2 - More than 90% of the adhesive remaining on the ceramic crown surface;

3 - Less than 90%, but more than 10% of the adhesive remaining on the surface;

4 - Less than 10% of the adhesive remaining on the ceramic crown surface;

5 - No adhesive remaining on the ceramic crown surface.

The damage to the ceramic surface which may have occurred during the shear bond testing was recorded using the Porcelain Fracture Index (PFI; Bourke and Rock, 1999) (8). The index is divided into four scores as follows:

0 - ceramic surface intact or in the same condition as before the bonding procedure;

1 - surface damage limited to glaze layer or very superficial ceramic;

2 - surface damage which features a significant loss of ceramic requiring restoration of the defect by composite resin or replacement of the restoration;

3 - surface damage where the core material has been exposed due to the depth of the cohesive failure.

The study was conducted at the School of Dental Medicine and at the Faculty of Mechanical Engineering and Naval Architecture, Laboratory for testing mechanical properties, University of Zagreb, Croatia.

In certain experiments the data were recorded by the type of material, type of brackets, the type of etching, shear bond strength (SBS) and the two indexes. These data represent the variables of this study, and are systematically presented in Table 1.

Table 1. Variables of the study

Name	Label	Categories	
		Value	Label
T-CER	Type of ceramic	1	Feldspar (Porcelain Fused to Metal)
		2	Zirconium
		3	Lithium Disilicate
T-BRA	Type of bracket	1	Metallic
		2	Ceramic
T-ETC	Type of etchant	1	Phosphoric Acid (PHA)
		2	Hydrofluoric Acid (HFA)
SBS	Shear Bond Strength		
ARI	Adhesive Remnant Index	1	All adhesive remaining on the ceramic crown surface with the impression of the bracket base
		2	More than 90% of the adhesive remaining on the ceramic crown surface
		3	Less than 90%, but more than 10% of the adhesive remaining on the surface
		4	Less than 10% of the adhesive remaining on the ceramic crown surface
		5	No adhesive remaining on the ceramic crown surface
PFI	Porcelain Fracture Index	0	Ceramic surface intact or in the same condition as before the bonding procedure
		1	Surface damage limited to glaze layer or very superficial ceramic
		2	Surface damage which features a significant loss of ceramic requiring restoration of the defect
		3	Surface damage where the core material has been exposed

3.4 **Statistical data processing**

Statistical analysis of the data includes the description of all factors (type of brackets - 2 categories, type of ceramic surface - 3 categories, conditioning of the surface 2 - category), force required for the detachment of the brackets, the index of the remaining material (ARI) and the index of the damage on the ceramic surface (PFI). The subject of data processing was the testing of hypotheses of the research, where a three-factor test was performed as independent variables and with a single dependence variable of measurement level and two nominal variables. For each combination of factors, 12 independent measurements of the dependent variables were performed. Since the dependent variable is in normal distribution and its variance on factors is homogeneous, to test the hypothesis of independence of the dependent variable from factors, the method of triple factorial analysis of variance was chosen.

The difference between the groups, formed by a combination of factors, was tested by the methods of post hoc analysis. Following the implementation of a series of post hoc analysis methods, the results of the Fisher LSD test (Least Significant Difference) were presented as the least strict methods. Due to possible errors in the application of the LSD method, appropriate graphs are provided on which the differences between the groups are illustrated.

The dependency of factors and nominal variables was tested using chi-square test. The level of significance was set at $\alpha = 0.05$ (87). The results are documented in tables and figures.

The data analysis was performed with the STATISTICA 10 software package (StatSoft, Inc. (2011) - STATISTICA (data analysis software system), version 10. (www.statsoft.com.).

4.RESULTS

4.1 Results from Shear Bond Strength (SBS) analysis

The results of this study derived from the tests that determined how different materials and treatments affect the shear bond strength (SBS). The dependent variable is the SBS, the force required to separate the bracket from the substrate. The factors are: the type of ceramic in three different materials (feldspar - porcelain fused to metal, all-contour zirconia and lithium disilicate), the type of bracket in two different materials (metal and polycrystalline ceramic), and the type of etchant done with two different conditioning materials (37% PHA and 5% HFA). Each test was repeated under the same conditions 12 times, which means that the total number of tests performed was $3 \times 2 \times 2 \times 12 = 144$.

The case number, mean, standard deviation and coefficient of variation of SBS for all possible sub-samples, are listed in Table 2. As noted in the table, average values range from 8.52 to 14.75 MPa. Variability of SBS, that is the data scattering around average values, is large and ranges from 30.8 to 61.8%.

The distribution of the SBS is shown in Figure 15. Despite the slight left asymmetry, the SBS distribution is considered to be a normal distribution, as confirmed by the Kolmogorov-Smirnov test ($D = 0.0663$, p = n.s.). With this the conditions are fulfilled, so the impact of the factors (type of ceramic, type of bracket, type of etchant) and their interaction with the SBS can be examined by the three-factorial analysis of variance.

Descriptive statistics for the dependent variable shear bond strength are listed in Table 2.

Table 2. Descriptive statistics of the dependent variable the shear bond strength (SBS).

Type of ceramic	Type of bracket	Type of etchant	Group	Shear Bond Strength (MPa)			
				n[a]	Mean	SD[b]	CV[c] (%)
Feldspar (PFM)	Metallic	Phosphoric Acid (PHA)	**1**	12	9.89	4.95	50.0
		Hydrofluoric Acid (HFA)	**2**	12	10.82	5.92	54.7
		Total		24	10.36	5.36	51.7
	Ceramic	Phosphoric Acid (PHA)	**3**	12	14.10	4.35	30.8
		Hydrofluoric Acid (HFA)	**4**	12	14.75	6.27	42.5
		Total		24	14.43	5.29	36.6
	Total	Phosphoric Acid (PHA)		24	12.00	5.04	42.0
		Hydrofluoric Acid (HFA)		24	12.79	6.29	49.2
		Total		48	12.39	5.65	45.6
Zirconia	Metallic	Phosphoric Acid (PHA)	**5**	12	10.85	5.84	53.8
		Hydrofluoric Acid (HFA)	**6**	12	11.84	7.30	61.7
		Total		24	11.35	6.49	57.2
	Ceramic	Phosphoric Acid (PHA)	**7**	12	8.52	4.72	55.3
		Hydrofluoric Acid (HFA)	**8**	12	8.99	5.36	59.7
		Total		24	8.75	4.94	56.5
	Total	Phosphoric Acid (PHA)		24	9.69	5.33	55.0
		Hydrofluoric Acid (HFA)		24	10.41	6.43	61.8
		Total		48	10.05	5.85	58.2
Lithium disilicate	Metallic	Phosphoric Acid (PHA)	**9**	12	10.20	3.29	32.2
		Hydrofluoric Acid (HFA)	**10**	12	11.95	5.96	49.9
		Total		24	11.08	4.79	43.3
	Ceramic	Phosphoric Acid (PHA)	**11**	12	12.22	6.47	53.0
		Hydrofluoric Acid (HFA)	**12**	12	10.31	5.67	54.9
		Total		24	11.26	6.03	53.5
	Total	Phosphoric Acid (PHA)		24	11.21	5.13	45.7
		Hydrofluoric Acid (HFA)		24	11.13	5.75	51.6
		Total		48	11.17	5.39	48.2
Total	Metallic	Phosphoric Acid (PHA)		36	10.32	4.69	45.5
		Hydrofluoric Acid (HFA)		36	11.54	6.26	54.3
		Total		72	10.93	5.53	50.6
	Ceramic	Phosphoric Acid (PHA)		36	11.61	5.62	48.4
		Hydrofluoric Acid (HFA)		36	11.35	6.14	54.1
		Total		72	11.48	5.85	50.9
	Total	Phosphoric Acid (PHA)		72	10.96	5.18	47.3
		Hydrofluoric Acid (HFA)		72	11.44	6.16	53.8
		Total		144	11.20	5.68	50.7

Legend: [a] number of cases, [b] standard deviation, [c] coefficient of variation

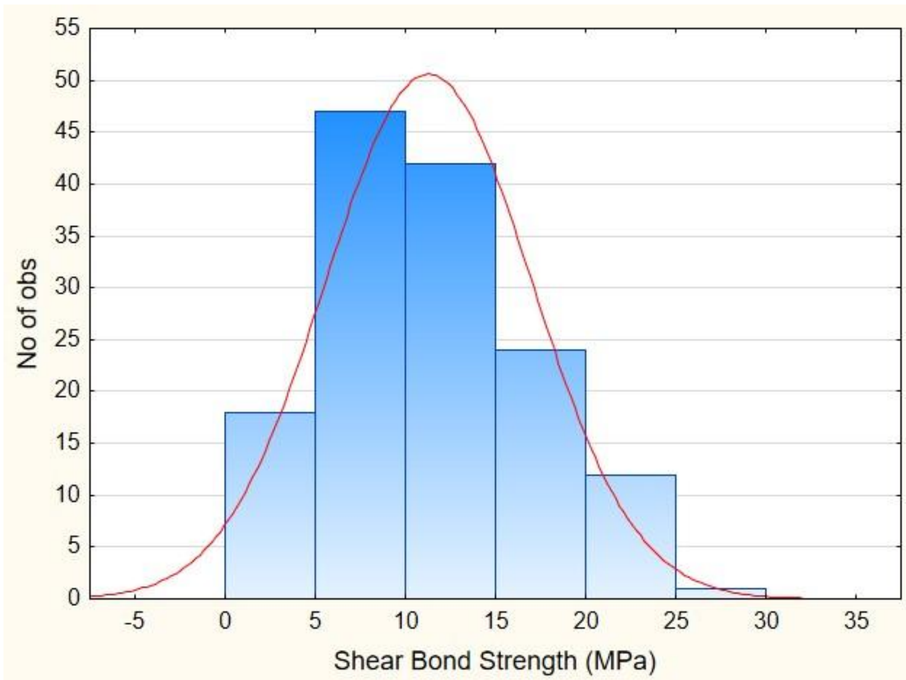


Figure 15. Distribution of the Shear Bond Strength (MPa).

The results from three-factorial analysis of variance of the SBS are presented in Table 3 and shown in Figures 16 to 26.

According to the results of the analysis, the main factors (T-CER, T-BRA, and T-ETC) do not significantly influence the formation of the average of the SBS. The probability of the exclusive influence of T-CER is relatively high (87.3%) with an error of 12.7%. Out of double factor interactions, only the interactive influence of T-CER and T-BRA has a statistically significant influence on the formation of the SBS values ($p = 0.016$). On the other hand, the triple factor interaction does not have a statistically significant effect on the formation of the SBS values (Table 3).

Table 3. The univariate tests of significance for the Shear Bond Strength (MPa).

Effect	SS[a]	df[b]	MS[c]	F[d]	p[e]
Intercept	18077,56	1	18077,56	576,201	<0,001
Type of ceramic (T-CER)	131,73	2	65,87	2,099	0,127
Type of bracket (T-BRA)	11,08	1	11,08	0,353	0,553
Type of etchant (T-ETC)	8,30	0	8,30	0,264	0,608
T-CER × T-BRA	268,53	2	134,26	4,279	**0,016**
T-CER × T-ETC	5,60	2	2,80	0,089	0,915
T-BRA × T-ETC	19,76	1	19,76	0,630	0,429
T-CER × T-BRA × T-ETC	21,29	2	10,65	0,339	0,713
ERROR	4141,33	132	31,37		
TOTAL	22685,18	144			

Legend: [a]sum of squares, [d]egree of freedom, [c]mean square, [d]F-statistics, probability hypotheses that factors do not affect the dependent variable

The average values of SBS for T-CER, T-BRA or T-ETC are illustrated in Figures 16, 17 and 18. For an average SBS value, a 95% confidence interval is set, which means that the actual (population) average of the SBS with 95% probability is within this interval.

The post hoc analysis for the comparison of possible pairs of subgroups induced by the factors and their possible interactions was performed by an LSD test that is the least conservative one out of many tests developed for that purpose.

According to the LSD test, the difference between possible pairs of ceramic types (Table 4) is statistically significant between feldspar and zirconia (p=0.042). The SBS for feldspar material is on average larger than for the other two materials, and compared to zirconia is significantly larger, as shown in the Figure 16 with a rounded frame, that encompasses significantly different materials. It is also visible that 95% of the confident intervals of the SBS for these materials are largely covered, due to the high variation coefficient (Table 2).

Table 4. The LSD test of the Shear Bond Strength – probabilities for Post Hoc test.

Cell. No.	Type of ceramic	(1) 12.39	(2) 10.05	(3) 11.17
1	Feldspar (PFM)		**0.042**	0.288
2	Zirconia	**0.042**		0.328
3	Lithium disilicate	0.288	0.328	

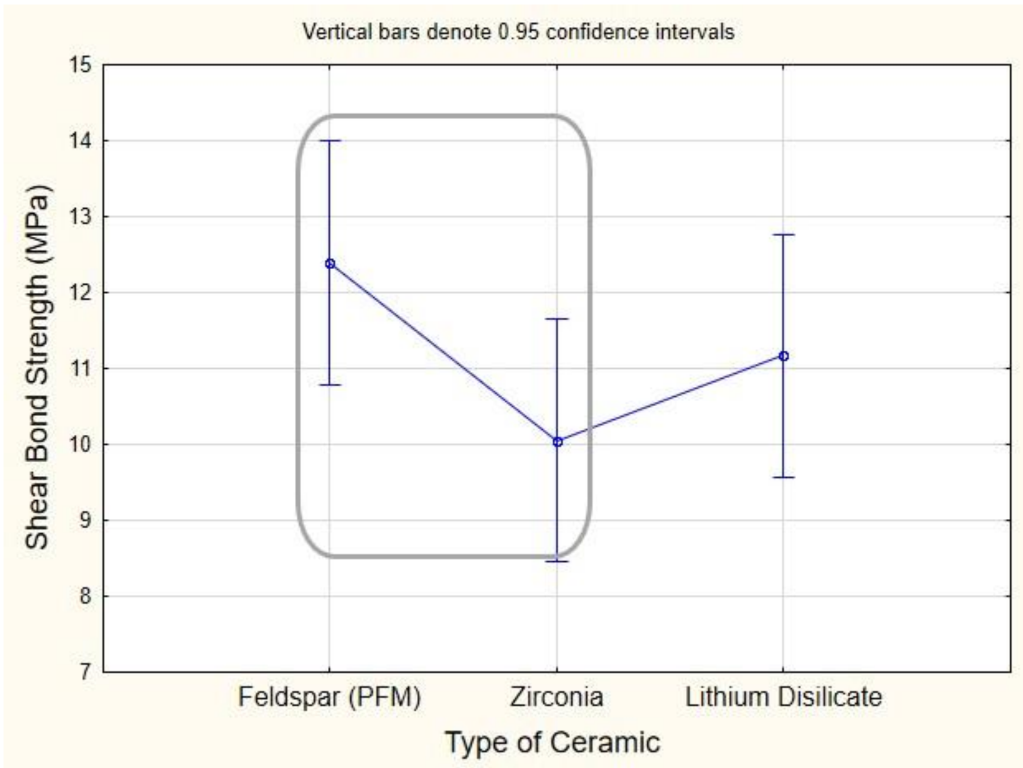


Figure 16. The means of the SBS by the type of ceramic.

The results of the LSD test (Table 5) show that the SBS by type of bracket (T-BRA) does not differ significantly, because both averages are almost equal and are estimated to within a largely covering 95% confident interval of reliability (Figure 17).

Table 5. The LSD test of the SBS – probabilities for Post Hoc test.

Cell. No.	Type of bracket	10.93	11.48
1	Metallic		0.553
2	Ceramic	0.553	

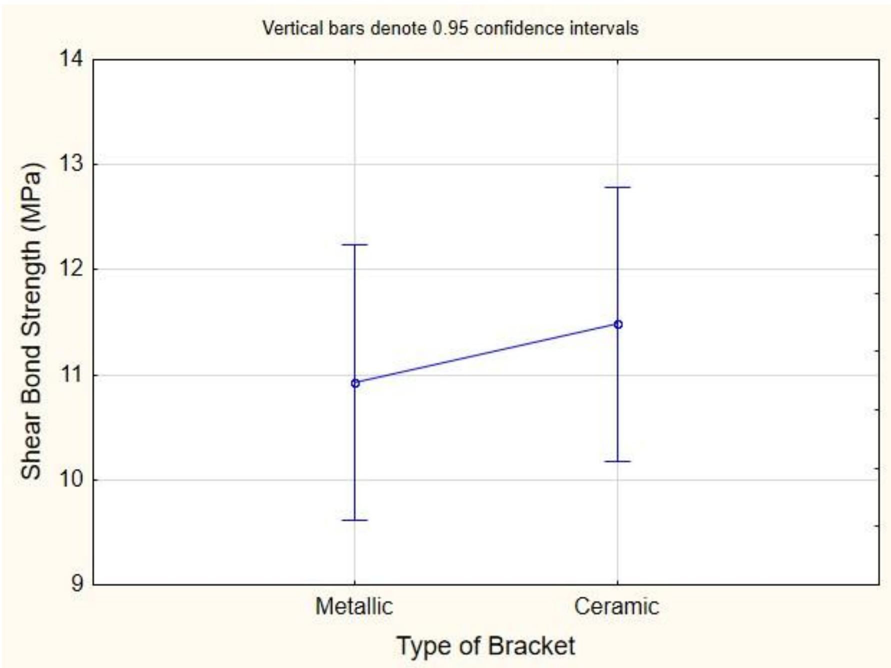


Figure 17. The means of the SBS by the type of bracket.

This is also the case with the surface conditioning (T-ETC), as seen from the results of the LSD test (Table 6) and it is clearly illustrated in Figure 18.

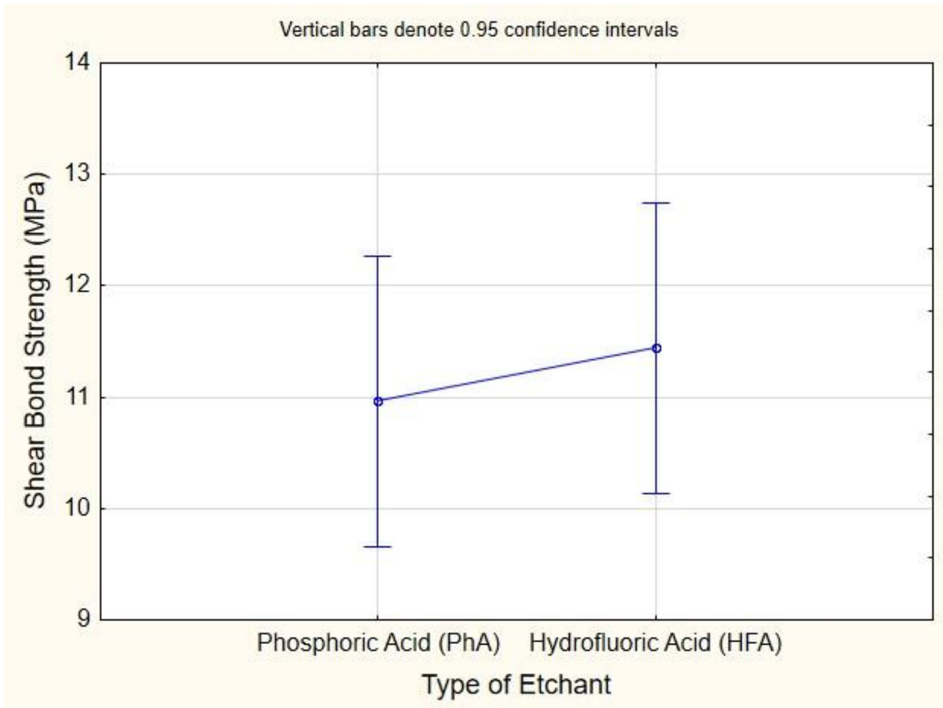


Figure 18. The means of the SBS by the type of etchant.

Table 6. The LSD test of the SBS – probabilities for Post Hoc test.

Cell. No.	Type of etchant	(1) 10.96	(2) 11.44
1	Phosphoric Acid (PHA)		0.608
2	Hydrofluoric Acid (HFA)	0.608	

An illustration of the statistically significant simultaneous effect of T-CER and T-BRA (p = 0.016, Table 3) on the formation of the SBS values is shown in Fig.19 and 20. In Figure 19 on the horizontal axis are the T-CER categories and the colour of the line indicates the categories of T-BRA. In Figure 20 their role was replaced. The details of this significant interaction were also tested by the LSD test for all six pairs of samples from the type of ceramic and type of brackets. In the case of three pairs of samples, the difference of SBS is statistically significant. The SBS is significantly different for feldspar, depending on the type of bracket (p=0.013). Namely, the average SBS of the metallic brackets bonded to feldspar ceramic type is 10.36 MPa, and of the ceramic bracket is statistically significantly higher, 14.43 MPa (Table 7). The conclusion is clearly evident in Figure 19 on which statistically significant different pairs of samples were highlighted by a rounded frame.

Table 7. The LSD test of the SBS – probabilities for Post Hoc test.

Cell. No.	Type of ceramic	Type of bracket	(1) 10.36	(2) 14.43	(3) 11.35	(4) 8.75
1	Feldspar (PFM)	Metallic		0.013	0.542	0.323
2	Feldspar (PFM)	Ceramic	0.013		0.059	0.001
3	Zirconia	Metallic	0.542	0.059		0.111
4	Zirconia	Ceramic	0.323	0.001	0.111	
5	Lithium disilicate	Metallic	0.656	0.040	0.869	0.153
6	Lithium disilicate	Ceramic	0.576	0.053	0.961	0.123

Cell. No.	Type of ceramic	Type of bracket	(5) 11.08	(6) 11.27
1	Feldspar (PFM)	Metallic	0.656	0.576
2	Feldspar (PFM)	Ceramic	0.040	0.053
3	Zirconia	Metallic	0.869	0.961
4	Zirconia	Ceramic	0.153	0.123
5	Lithium disilicate	Metallic		0.908
6	Lithium disilicate	Ceramic	0.908	

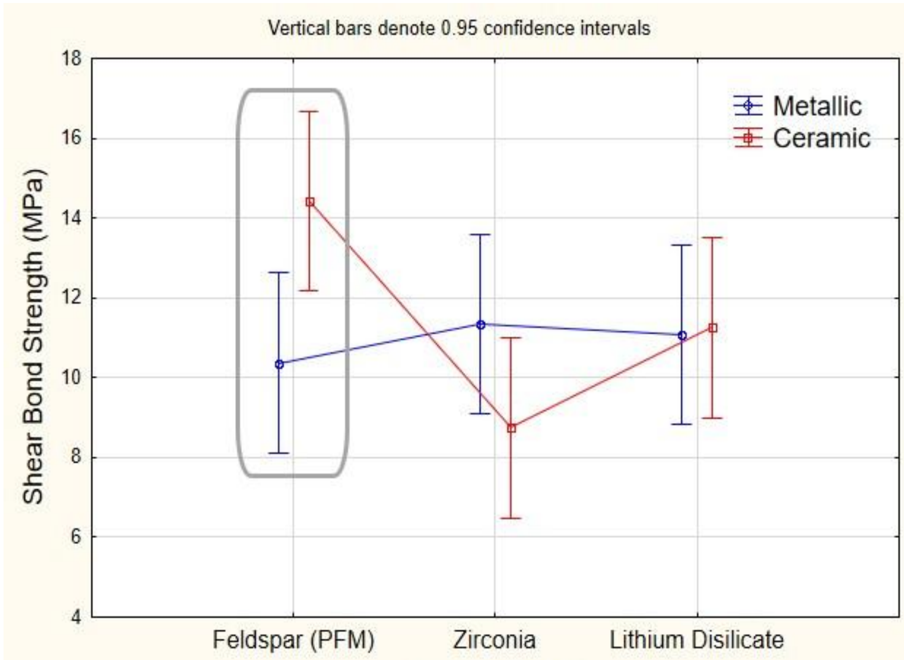


Figure 19. The means of the SBS by the type of ceramic and the type of bracket.

Zirconia combined with a ceramic bracket achieves SBS on average 8.75 MPa, which is significantly smaller ($p = 0.001$) than 14.43 MPa achieved by the feldspar also in combination with a ceramic bracket. In Figure 20 this difference is highlighted with a non-continuous frame. The above mentioned SBS value of feldspar in combination with the ceramic bracket is significantly higher than the value 11.08 MPa of lithium disilicate in combination with a metallic bracket ($p = 0.040$). In Figure 20 this difference is highlighted with a continuous frame.

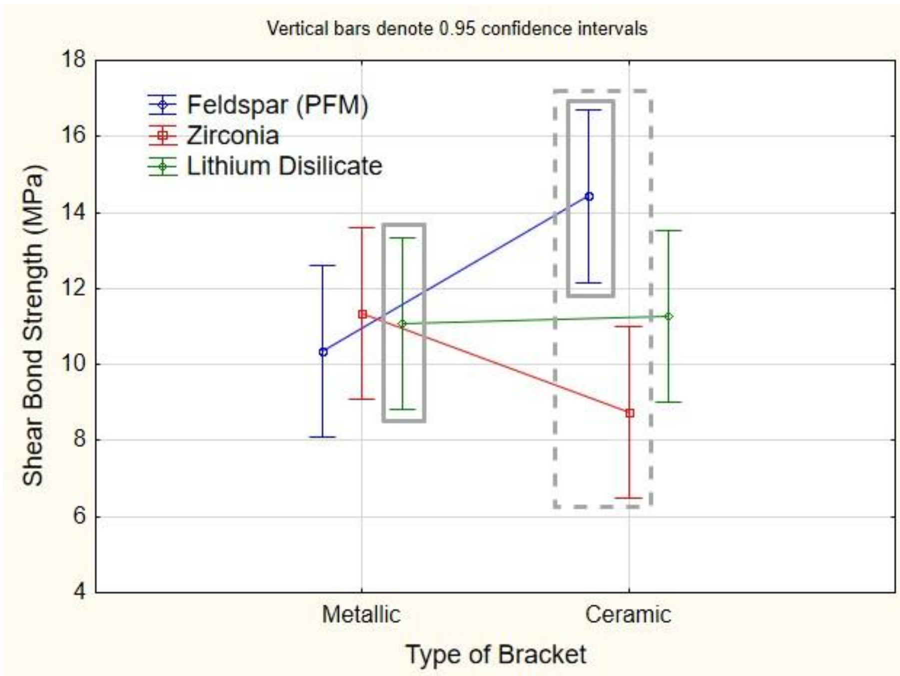


Figure 20. The means of the SBS by the type of bracket and the type of ceramic.

Interaction of the ceramic type to form SBS values in combination with surface preparation is not statistically significant in any of the six possible pairs (Table 8 and Figure 21). The absence of the statistically significant influence is clearly noticed in the large coverage of the corresponding confident intervals. But there is also a noticeable shift of the feldspar ceramic from the other two ceramic materials, equally for both etching methods.

Table 8. The LSD test of the SBS – probabilities for Post Hoc test.

Cell. No.	Type of ceramic	Type of etchant	(1) 12.00	(2) 12.79	(3) 9.69
1	Feldspar (PFM)	Phosphoric Acid (PHA)		0.626	0.156
2	Feldspar (PFM)	Hydrofluoric Acid (HFA)	0.626		**0.057**
3	Zirconia	Phosphoric Acid (PHA)	0.156	**0.057**	
4	Zirconia	Hydrofluoric Acid (HFA)	0.329	0.144	0.654
5	Lithium Disilicate	Phosphoric Acid (PHA)	0.627	0.331	0.348
6	Lithium Disilicate	Hydrofluoric Acid (HFA)	0.595	0.308	0.373
Cell. No.	Type of ceramic	Type of etchant	(4) 10.41	(5) 11.21	(6) 11.13
1	Feldspar (PFM)	Phosphoric Acid (PHA)	0.329	0.627	0.595
2	Feldspar (PFM)	Hydrofluoric Acid (HFA)	0.144	0.331	0.308
3	Zirconia	Phosphoric Acid (PHA)	0.654	0.348	0.373
4	Zirconia	Hydrofluoric Acid (HFA)		0.623	0.656
5	Lithium Disilicate	Phosphoric Acid (PHA)	0.623		0.963
6	Lithium Disilicate	Hydrofluoric Acid (HFA)	0.656	0.963	

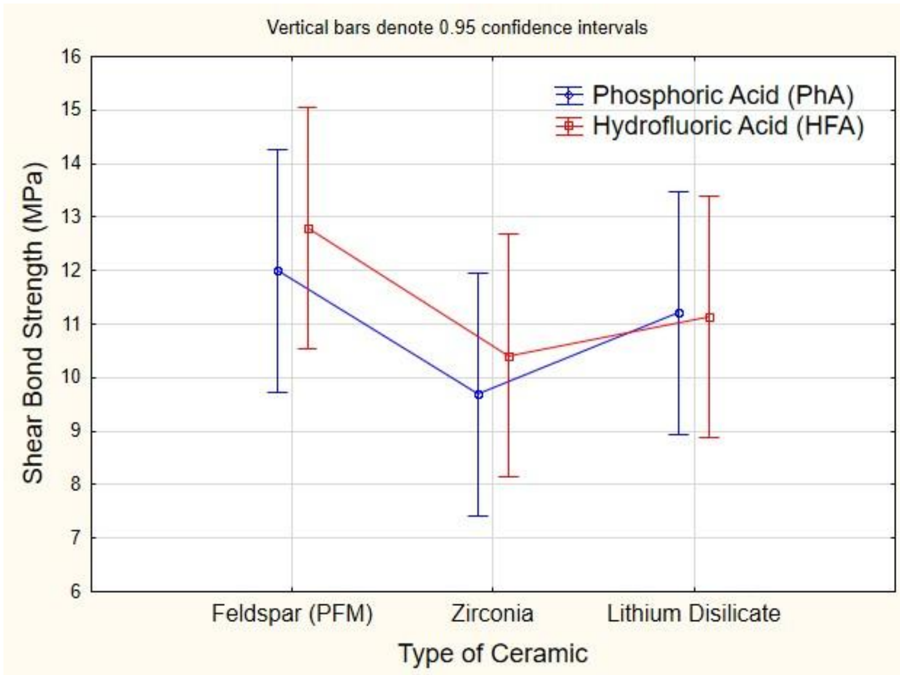


Figure 21. The means of the SBS by the type of ceramic and the type of etchant.

The illustration of the last double interaction relates to the formation of the SBS average under the influence of T-BRA and T-ETC. This is not statistically significant, indicating the probabilities in Table 9 and the position of the SBS average values in Figure 22.

Table 9. The LSD test of the SBS – probabilities for Post Hoc test.

Cell. No.	Type of bracket	Type of etchant	(1) 10.32	(2) 11.54	(3) 11.61	(4) 11.35
1	Metallic	Phosphoric Acid (PHA)		0.357	0.328	0.434
2	Metallic	Hydrofluoric Acid (HFA)	0.357		0.955	0.888
3	Ceramic	Phosphoric Acid (PHA)	0.328	0.955		0.844
4	Ceramic	Hydrofluoric Acid (HFA)	0.434	0.888	0.844	

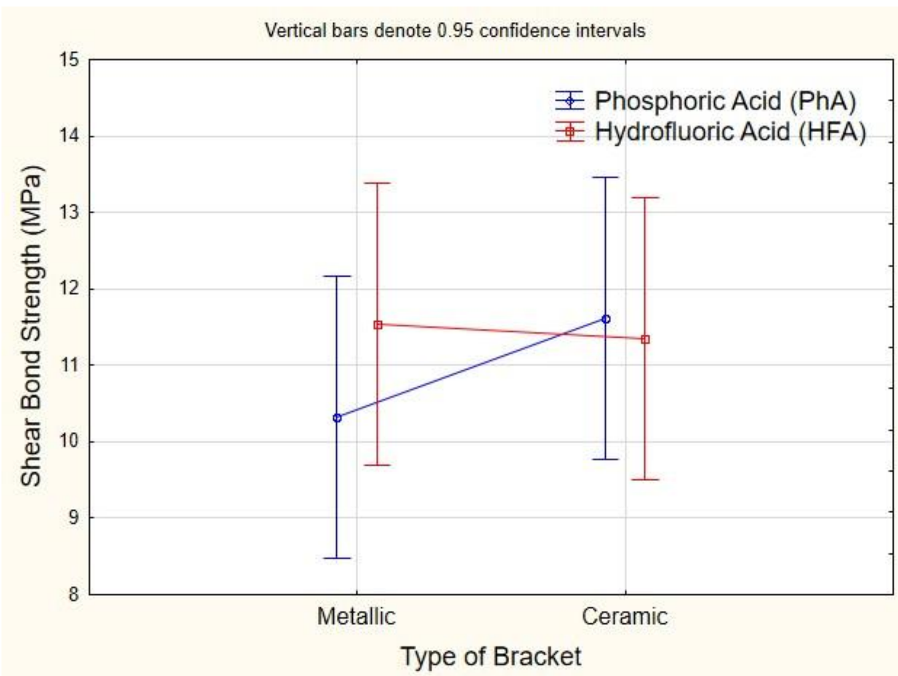


Figure 22. The means of the SBS by the type of bracket and the type of etchant.

The triple interaction of T-CER, T-BRA and T-ETC in the applied model of analysis of variance was not statistically significant (Table 3). However, the post hoc analysis with LSD method reveals six pairs of sub-samples, out of 66 possible, which differ significantly in SBS values. The probabilities of post hoc analysis are shown in Table 10, not including columns 5 to 12 of the combination because they do not contain relevant information, namely the averages of omitted combinations can be read in Table 2.

Table 10. The LSD test of the SBS – probabilities for Post Hoc test.

Cell. No.	Type of beramic	Type of bracket	Type of etchant	(1) 10.32	(2) 11.54	(3) 11.61	(4) 11.35
1	Feldspar (PFM)	Metallic	Phos. Acid (PHA)		0.686	0.068	**0.035**
2	Feldspar (PFM)	Metallic	Hyd. Acid (HFA)	0.686		0.154	0.088
3	Feldspar (PFM)	Ceramic	Phos. Acid (PHA)	0.068	0.154		0.775
4	Feldspar (PFM)	Ceramic	Hyd. Acid (HFA)	**0.035**	0.088	0.775	
5	Zirconia	Metallic	Phos. Acid (PHA)	0.676	0.989	0.158	0.090
6	Zirconia	Metallic	Hyd. Acid (HFA)	0.397	0.657	0.325	0.205
7	Zirconia	Ceramic	Phos. Acid (PHA)	0.550	0.317	**0.016**	**0.007**
8	Zirconia	Ceramic	Hyd. Acid (HFA)	0.692	0.424	**0.027**	**0.013**
9	Lithium Disilicate	Metallic	Phos. Acid (PHA)	0.893	0.788	0.091	**0.049**
10	Lithium Disilicate	Metallic	Hyd. Acid (HFA)	0.370	0.621	0.350	0.223
11	Lithium Disilicate	Ceramic	Phos. Acid (PHA)	0.312	0.543	0.412	0.269
12	Lithium Disilicate	Ceramic	Hyd. Acid (HFA)	0.855	0.825	0.100	**0.054**

The pairs of groups that are significantly different by the SBS are presented in Figures 23 to 26. Therefore, in Figure 23 a statistically significant difference of the feldspar ceramic with metal brackets and etching with PHA (group 1: mean = 9.89 MPa SBS) and the same ceramic with ceramic bracket and etching with HFA (group 4: mean = 14.75 SBS) is marked with a frame, which indicates a better performance of the second group.

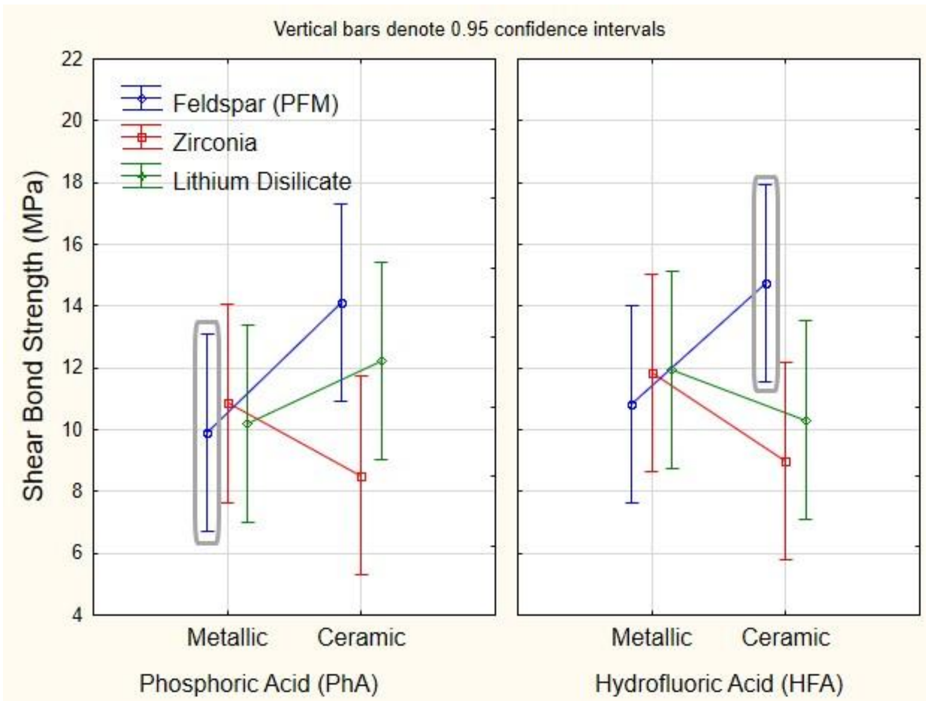


Figure 23. The means of the SBS by the type of ceramic, bracket and etchant.

Figure 24 illustrates significantly higher SBS of feldspar (group 3: mean SBS = 14.10 MPa) versus zirconia (group 7: mean SBS = 8.52 MPa) under the same conditions, i.e. in combination with a ceramic bracket and PHA surface preparation. In the figure it is marked with a non-continuous frame. The continuous frame in the same figure illustrates a statistically significant difference of the combination of lithium disilicate ceramic, metal bracket and PHA surface preparation (group 9: mean SBS = 10.20) compared to feldspar ceramic, ceramic bracket and HFA surface preparation (group 4: mean SBS = 14.75).

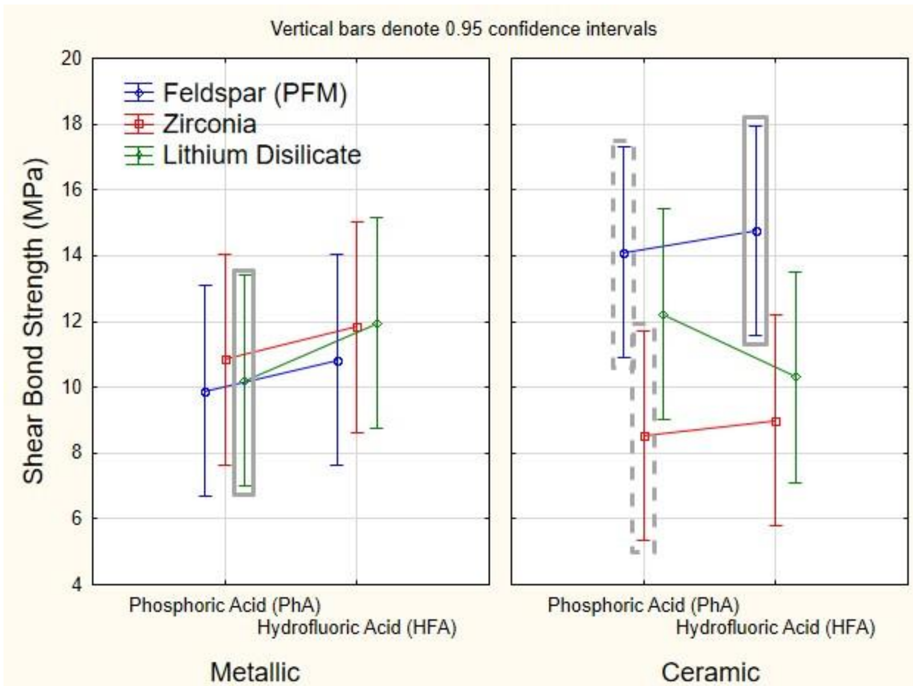


Figure 24. The means of the SBS by the type of ceramic, bracket and etchant.

In Figure 25, a statistically significant lag of zirconia combined with ceramic brackets and PHA surface preparation is presented with a non-continuous frame (group 7: mean SBS = 8.52 MPa) from feldspar ceramic in combination with ceramic brackets and HFA surface preparation (group 4: mean SBS = 14.75 MPa). In the same figure, a statistically significant difference is marked with a continuous frame the SBS of zirconia in combination with ceramic brackets and HFA-surface preparation (group 8: mean SBS = 8.99 MPa) and feldspar in combination with ceramic brackets and PHA surface preparation (group 3: mean SBS = 14.10 MPa).

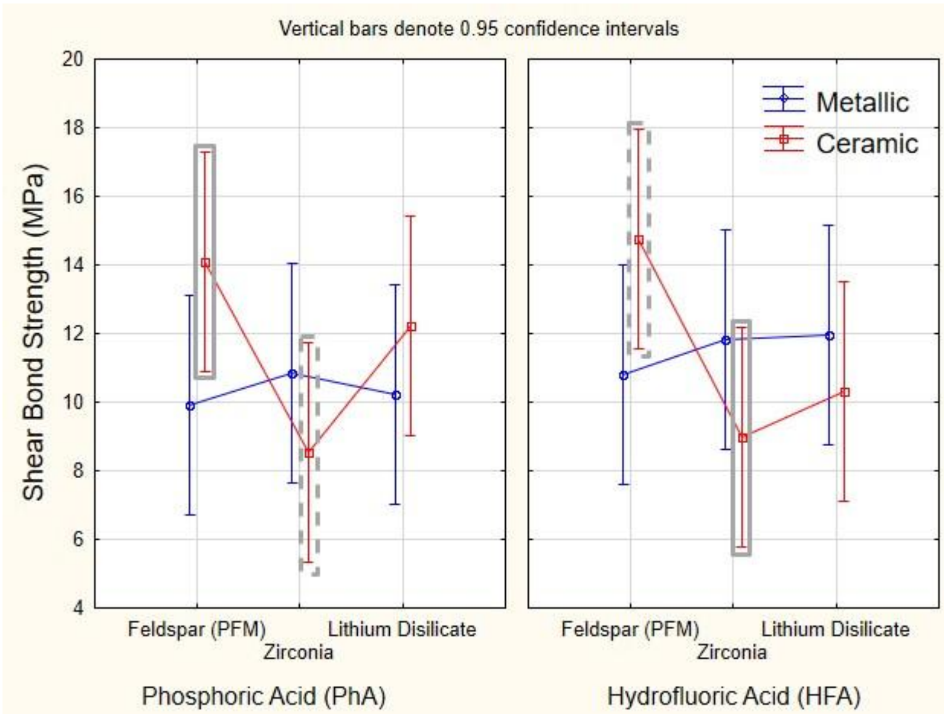


Figure 25. The means of the SBS by the type of ceramic, bracket and etchant.

A statistically significant difference in the SBS for the zirconia surfaces conditioned with HFA in combination with ceramic brackets (group 8: mean SBS = 8.99 MPa) and the feldspar surfaces also conditioned with HFA and combined with ceramic brackets (group 4: mean SBS = 14.75 MPa) is shown in Figure 26 and marked with a frame.

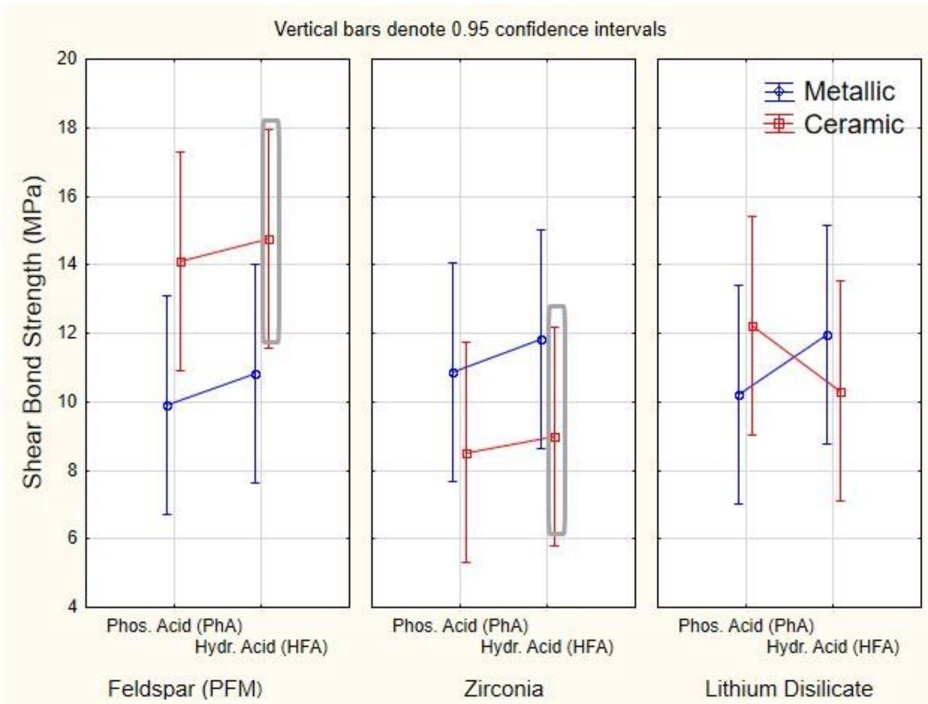


Figure 26. The means of the SBS by the type of ceramic, bracket and etchant.

The results of the test of variance homogeneity of the SBS are presented in Table 11. According to the results of all three tests, it can be argued that the variants are homogeneous and this condition for the application of the subject model is fulfilled. This result is confirmed by the Levene test (Table 12).

Table 11. Tests of Homogeneity of the SBS Variances.

Effect: Type of Ceramic * Bracket * Etchant					
	Hartley - F-max	Cochran – C	Bartlett - Chi-Sqr.	df	P
SBS	4.802353	0.145792	7.246593	11	0.779

Table 12. Levene's Test for Homogeneity of Variances.

Effect: "t_cer"*"t_bra"*"t_etc" Degrees of freedom for all F's: 11, 108				
	MS - Effect	MS - Error	F	p
SBS	8.590976	11.24972	0.763661	0.675

Figure 27 shows the relationship of the average values to the corresponding standard deviations of all 12 sub-samples formed by T-CER, T-BRA and T-ETC factors. The highest average values belong to samples # 3 and # 4 (as indicated in Table 2), and sample # 9 has the smallest variability.

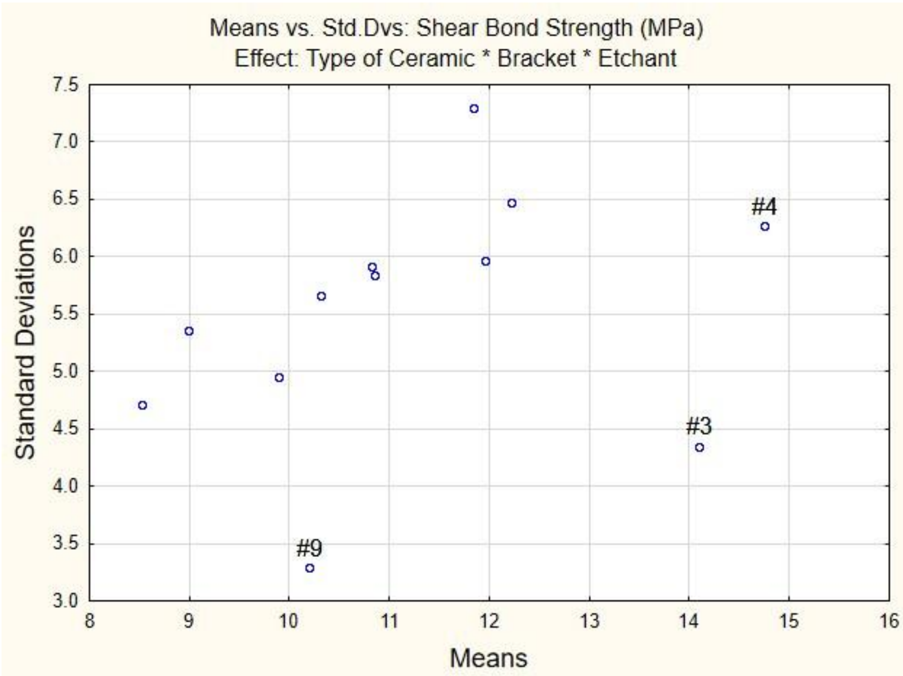


Figure 27. Mean vs. standard deviations plot of the shear bond strength for the type of ceramic, etchant and bracket.

The legitimacy of the applied variance analysis method also confirms the normality of the residual dependent variable (SBS) distribution, namely the normal probability plot of the residuals for the shear bond strength (Figure 28).

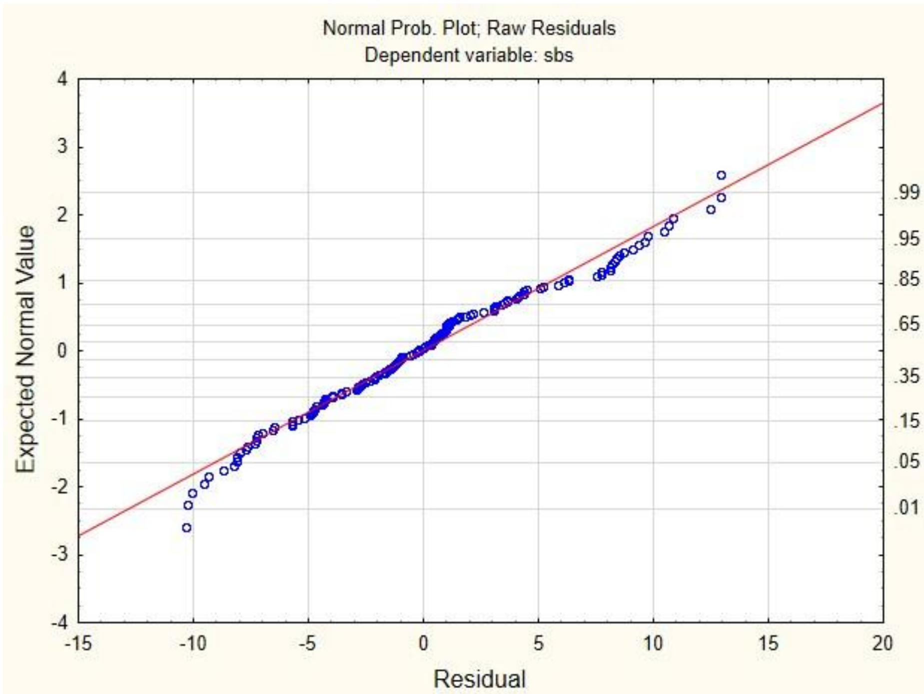


Figure 28. Normal probability plot raw residuals for the shear bond strength.

4.2 **Results from Adhesive Remnant Index (ARI) analysis**

Possible dependence of ARI on the type of ceramics was tested by chi-square test and the results are listed in Table 13. According to the results, the ARI does not depend statistically on the type of ceramics, which means that the occurrence of any of the ARI categories cannot be related to the influence of any type of ceramics.

Table 13. The cross tabulation of the ARI with the type of ceramic and χ^2 – test.

Adhesive Remnant Index		Type of ceramic			Total
		Feldspar (PFI)	Zirconium	Lithium Disilicate	
1- All adhesive remaining on the ceramic surface with the impression of the bracket base	n [a]	5	2	5	12
	hp [b]	41.7%	16.7%	41.7%	100.0%
	vp [c]	10.4%	4.2%	10.4%	8.3%
2 - More than 90 per cent of the adhesive remaining on the ceramic surface	n	7	4	4	15
	hp	46.7%	26.7%	26.7%	100.0%
	vp	14.6%	8.3%	8.3%	10.4%
3 - Less than 90 per cent but more than 10 per cent of the adhesive remaining on the ceramic surface	n	19	11	13	43
	hp	44.2%	25.6%	30.2%	100.0%
	vp	39.6%	22.9%	27.1%	29.9%
4 - Less than 10 per cent of the adhesive remaining on the ceramic surface	n	13	23	21	57
	hp	22.8%	40.4%	36.8%	100.0%
	vp	27.1%	47.9%	43.8%	39.6%
5 - No adhesive remaining on the ceramic surface	n	4	8	5	17
	hp	23.5%	47.1%	29.4%	100.0%
	vp	8.3%	16.7%	10.4%	11.8%
Total	n	48	48	48	144
	hp	33.3%	33.3%	33.3%	100.0%
χ^2 – test		χ^2=9.595		df=8	p=0.295

[a] count, [b] % within adhesive remnant index, [c] % within type of ceramic

The frequencies from Table 13 are visualized in Figure 29.

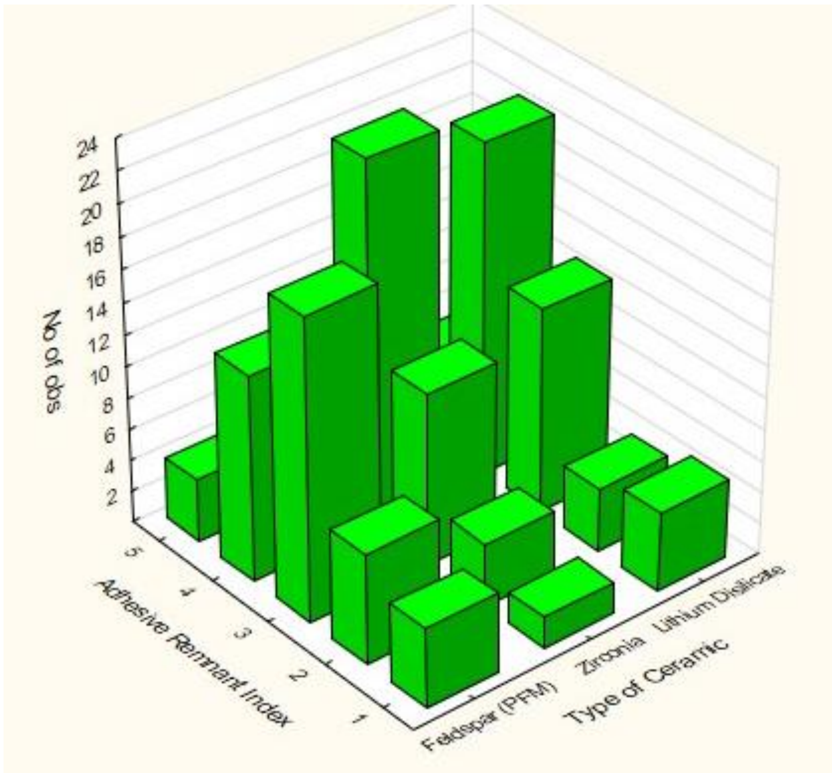


Figure 29. The bivariate histogram of ARI according to the type of ceramic.

The type of brackets significantly affects the appearance of ARI categories. The importance of this dependence is due to the fact that the first two categories (1 and 2) of ARI appear significantly more with metallic brackets: first ARI category occurs only with metallic brackets, and the second also occurs with metallic brackets in 80% of the cases. This significantly deviates from a 50% participation of the metallic brackets in the sample (50%). The 3rd category (60.5%) and 4th (59.6%) category of ARI are more frequent in ceramic brackets (Table 14).

Table 14. The Cross tabulation of the ARI with the type of bracket and χ^2 – test.

Adhesive Remnant Index		Type of bracket		Total
		Metallic	Ceramic	
1- All adhesive remaining on the ceramic surface with the impression of the bracket base	n [a]	12	0	12
	hp [b]	100.0%	0.0%	100.0%
	vp [c]	16.7%	0.0%	8.3%
2 - More than 90 per cent of the adhesive remaining on the ceramic surface	n	12	3	15
	hp	80.0%	20.0%	100.0%
	vp	16.7%	4.2%	10.4%
3 - Less than 90 per cent but more than 10 per cent of the adhesive remaining on the ceramic surface	n	17	26	43
	hp	39.5%	60.5%	100.0%
	vp	23.6%	36.1%	29.9%
4 - Less than 10 per cent of the adhesive remaining on the ceramic surface	n	23	34	57
	hp	40.4%	59.6%	100.0%
	vp	31.9%	47.2%	39.6%
5 - No adhesive remaining on the ceramic surface	n	8	9	17
	hp	47.1%	52.9%	100.0%
	vp	11.1%	12.5%	11.8%
Total	n	72	72	144
	hp	50.0%	50.0%	100.0%
χ^2 – test		χ^2=21.465	df=4	p<0,001

[a] count, [b] % within adhesive remnant index, [c] % within type of bracket

The frequencies from Table 14 are visualized in Figure 30.

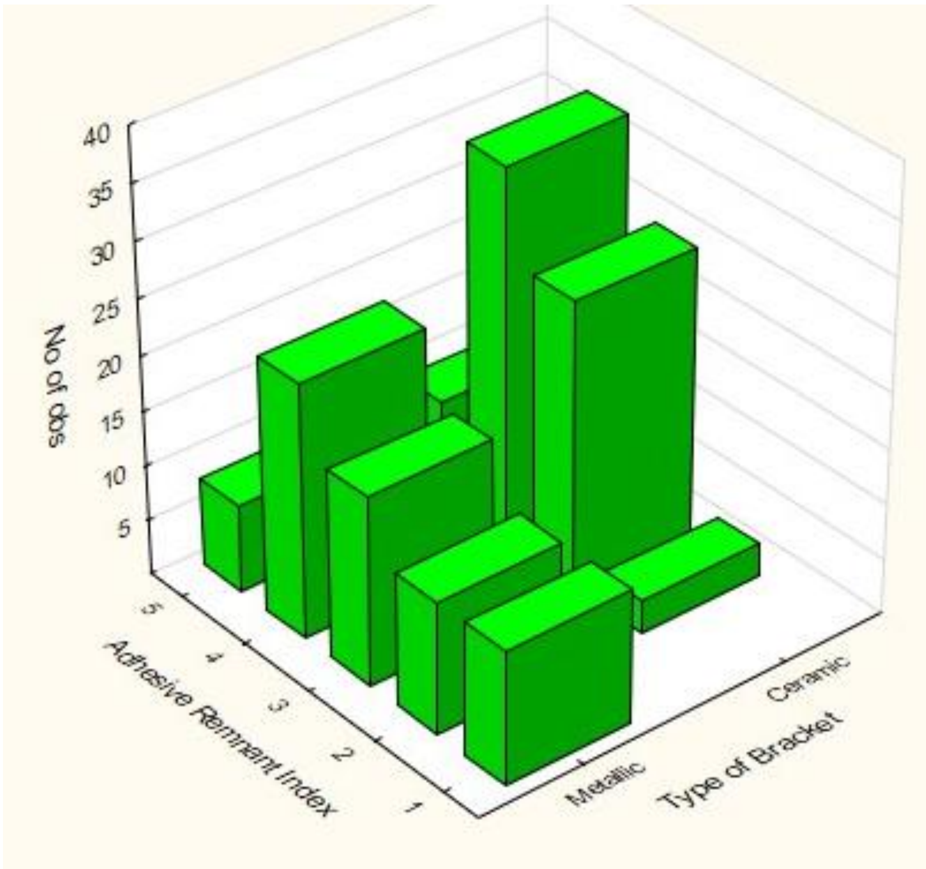


Figure 30. The bivariate histogram of ARI according to the type of bracket.

The etching for surface preparation does not affect significantly the appearance of the ARI categories according to the results presented in Table 15.

Table 15. The Cross tabulation of the ARI with the type of etchant and χ^2 – test.

Adhesive Remnant Index		Type of etchant: Phosphoric Acid (PHA)	Type of etchant: Hydrofluoric Acid (HFA)	Total
1- All adhesive remaining on the ceramic surface with the impression of the bracket base	n [a]	8	4	12
	hp [b]	66.7%	33.3%	100.0%
	vp [c]	11.1%	5.6%	8.3%
2 - More than 90 per cent of the adhesive remaining on the ceramic surface	n	8	7	15
	hp	53.3%	46.7%	100.0%
	vp	11.1%	9.7%	10.4%
3 - Less than 90 per cent but more than 10 per cent of the adhesive remaining on the ceramic surface	n	20	23	43
	hp	46.5%	53.5%	100.0%
	vp	27.8%	31.9%	29.9%
4 - Less than 10 per cent of the adhesive remaining on the ceramic surface	n	31	26	57
	hp	54.4%	45.6%	100.0%
	vp	43.1%	36.1%	39.6%
5 - No adhesive remaining on the ceramic surface	n	5	12	17
	hp	29.4%	70.6%	100.0%
	vp	6.9%	16.7%	11.8%
Total	n	72	72	144
	hp	50.0%	50.0%	100.0%
χ^2 – test		χ^2=4.930	df=4	p=0,295

[a] count, [b] % within adhesive remnant index, [c] % within type of etchant

The frequencies from Table 15 are visualized in Figure 31.

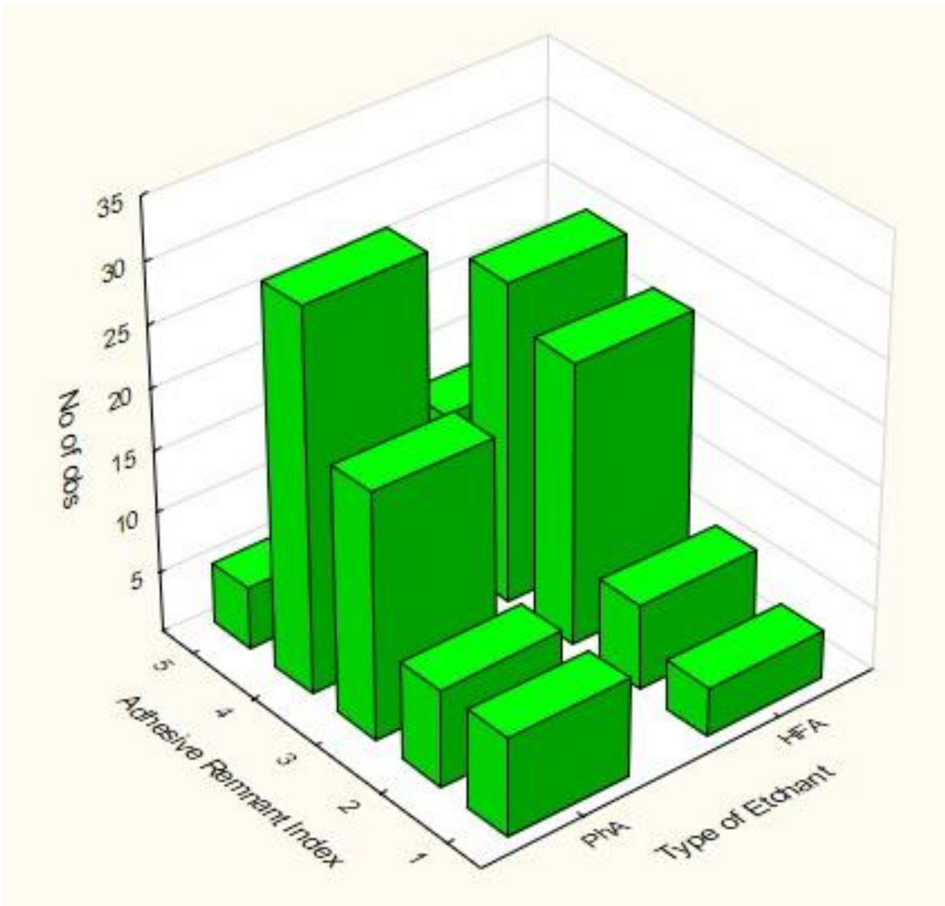


Figure 31. The bivariate histogram of ARI according to the type of etchant.

The connection between the type of bracket and PHA treatment is statistically significant for the appearance of the ARI categories (Chi-Square = 20.613, df = 4 $p < 0.001$). The categories 1 and 2 of ARI occur only with metallic brackets, while the other categories are dominated by ceramic brackets (Table 16).

Table 16. The cross tabulation of the ARI according to the type of bracket for PHA etchant and χ^2 – test.

Adhesive Remnant Index		Type of bracket		Total
		Metallic	Ceramic	
1- All adhesive remaining on the ceramic surface with the impression of the bracket base	n [a]	8	0	8
	hp [b]	100.0%	0.0%	100.0%
	vp [c]	22.2%	0.0%	11.1%
2 - More than 90 per cent of the adhesive remaining on the ceramic surface	n	8	0	8
	hp	100.0%	0.0%	100.0%
	vp	22.2%	0.0%	11.1%
3 - Less than 90 per cent but more than 10 per cent of the adhesive remaining on the ceramic surface	n	7	13	20
	hp	35.0%	65.0%	100.0%
	vp	19.4%	36.1%	27.8%
4 - Less than 10 per cent of the adhesive remaining on the ceramic surface	n	11	20	31
	hp	35.5%	64.5%	100.0%
	vp	30.6%	55.6%	43.1%
5 - No adhesive remaining on the ceramic surface	n	2	3	5
	hp	40.0%	60.0%	100.0%
	vp	5.6%	8.3%	6.9%
Total	n	36	36	72
	hp	50.0%	50.0%	100.0%
χ^2 – test		χ^2=20.613	df=4	p<0,001

[a] count, [b] % within adhesive remnant index, [c] % within type of etchant

The frequencies from Table 16 are visualized in Figure 32.

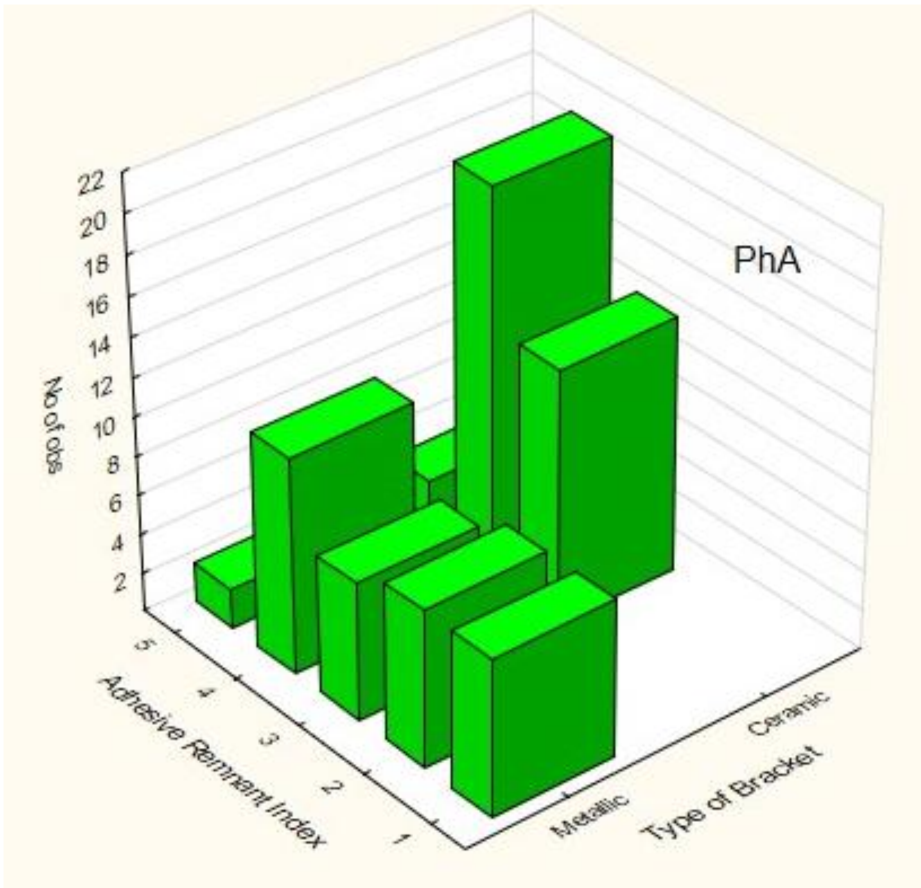


Figure 32. The bivariate histogram of ARI according to the type of bracket for PHA etchant.

As seen from the data in Table 17 and Figure 33, the SBS shows on average an increase in ARI categories. However, the analysis of variance showed that this is not statistically significant (Table 18).

Table 17. Descriptive statistics of the dependent variable SBS.

Adhesive Remnant Index	Shear Bond Strength (MPa)			
	n[a]	Mean	SD[b]	CV[c] (%)
1- All adhesive remaining on the ceramic surface with the impression of the bracket base	12	8.69	4.32	49.7
2 - More than 90 per cent of the adhesive remaining on the ceramic surface	15	10.57	5.58	52.8
3 - Less than 90 per cent but more than 10 per cent of the adhesive remaining on the ceramic surface	43	10.02	5.44	54.3
4 - Less than 10 per cent of the adhesive remaining on the ceramic surface	57	12.14	5.87	48.4
5 - No adhesive remaining on the ceramic surface	17	13.40	5.69	42.5
Total	144	11.20	5.68	50.7

Legend: [a] number of cases, [b] standard deviation, [c] coefficient of variation

Table 18. The univariate tests of significance for the shear bond strength (MPa).

	Sum of Squares	df	Mean Square	F	P
Between Groups	274.479	4	68.620	2.201	**0.072**
Within Groups	4333.143	139	31.174		
Total	4607.622	143			

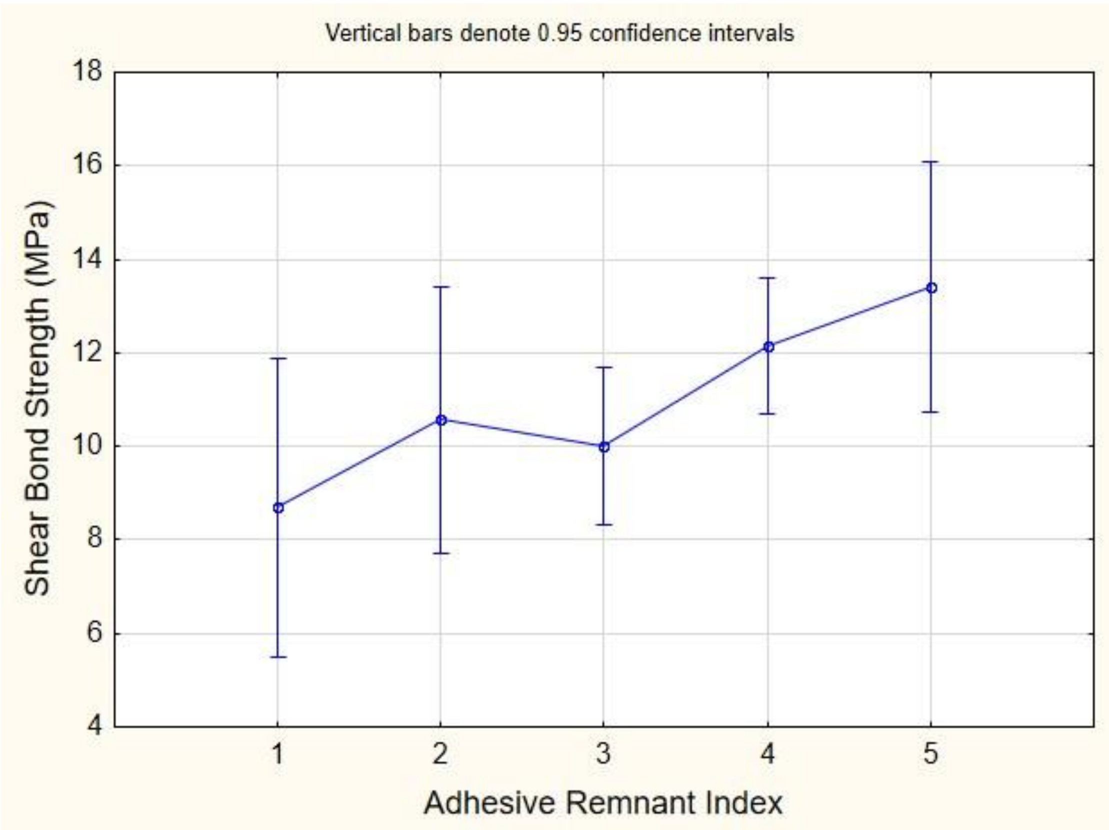


Figure 33. The means of SBS by the ARI.

The adhesive remnant and the type of bond failure are illustrated in representative upcoming figures 34, 35 and 36 for all groups in this research.

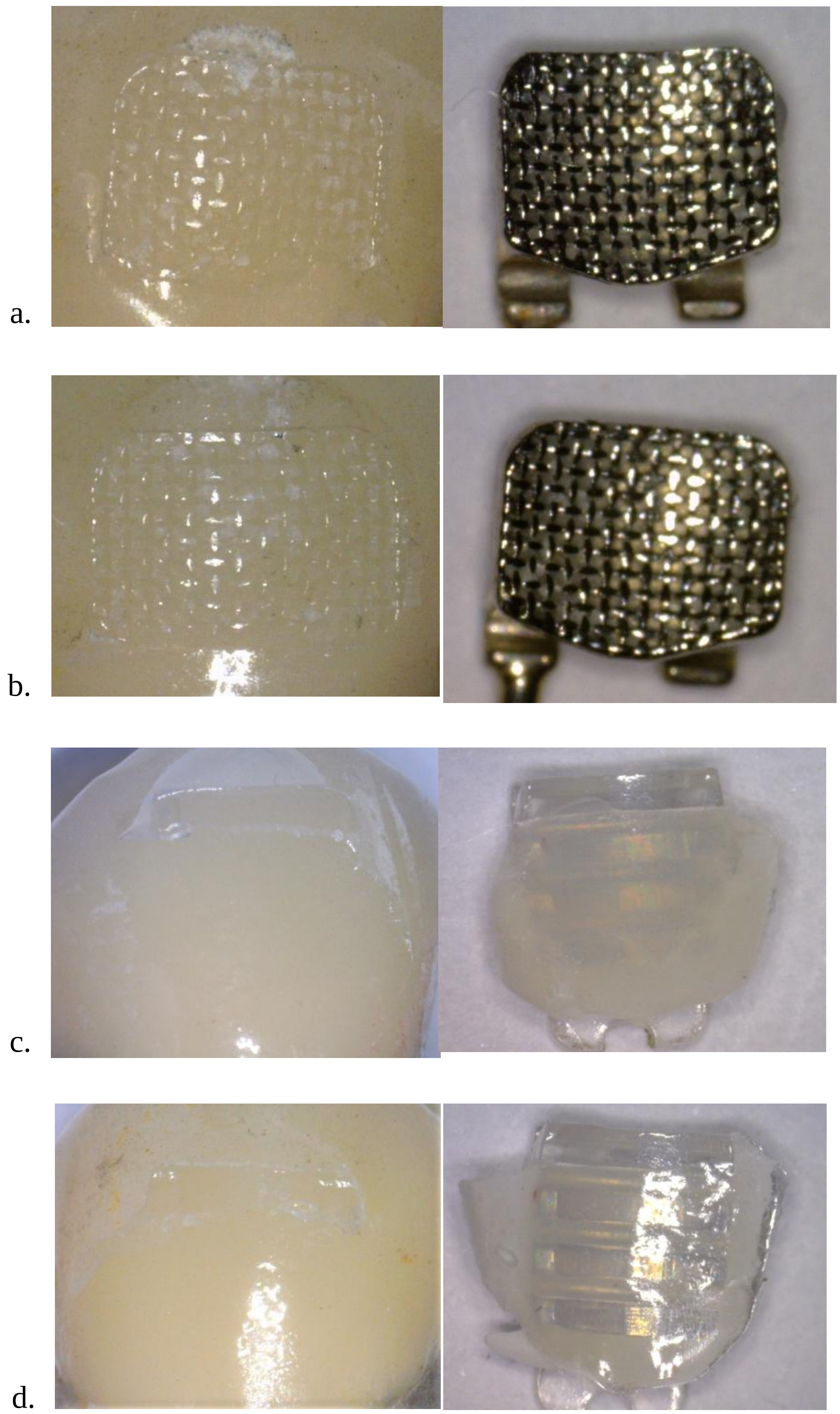


Figure 34. Feldspathic PFM etched with- and bonded: a. HFA - Metallic bracket; b. PHA - Metallic bracket; c. HFA - Ceramic bracket; d. PHA - Ceramic bracket.

a.

b.

c.

d.

Figure 35. Full contour zirconia etched with- and bonded: a. HFA - Metallic bracket; b. PHA - Metallic bracket; c. HFA - Ceramic bracket; d. PHA - Ceramic bracket.

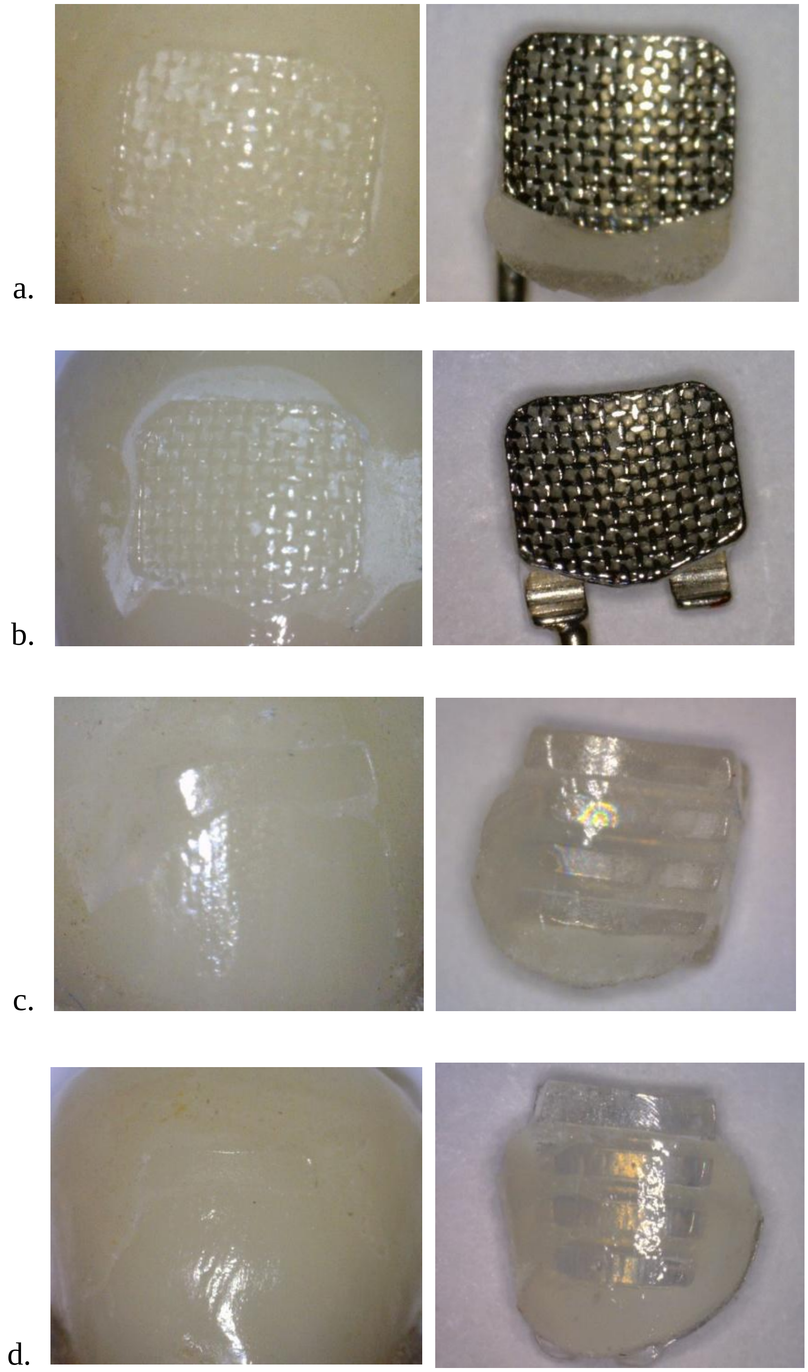


Figure 36. Lithium disilicate etched with- and bonded: a. HFA - Metallic bracket; b. PHA - Metallic bracket; c. HFA - Ceramic bracket; d. PHA - Ceramic bracket.

4.3 Results from Porcelain Fracture Index (PFI) analysis

The results of the dependency of the PFI on the types of ceramics, brackets and etching, are presented in Tables 19-21 and in Figures 37 to 39.

PFI does not depend significantly on the type of ceramic, but a visible shift is noticed indicating better performance of zirconia (Table 19).

Table 19. The cross tabulation of the PFI according to the type of ceramic and χ^2 – test.

Porcelain Fracture Index		Type of ceramic			Total
		Feldspar (PFM)	Zirconium	Lithium Disilicate	
0 - ceramic surface intact or in the same condition as before the bonding procedure	n [a]	12	19	11	42
	hp [b]	28.6%	45.2%	26.2%	100.0%
	vp [c]	25.0%	39.6%	22.9%	29.2%
1 - surface damage limited to glaze layer or very superficial ceramic	n	32	29	36	97
	hp	33.0%	29.9%	37.1%	100.0%
	vp	66.7%	60.4%	75.0%	67.4%
2 - surface damage which features significant loss of ceramic requiring restoration of the defect or replacement of the restoration	n	4	0	1	5
	hp	80.0%	.0%	20.0%	100.0%
	vp	8.3%	.0%	2.1%	3.5%
Total	n	48	48	48	144
	hp	33.3%	33.3%	33.3%	100.0%
χ^2 – test		χ^2=8.677		df=4	p=0,070

[a] count, [b] % within PFI, [c] % within type of ceramic

A statistically significant relationship was not registered in the analysis between PFI and t_bra, which means that PFI does not depend on the type of bracket (Table 20).

Table 20. The cross tabulation of the PFI according to the type of bracket and χ^2 – test.

Porcelain Fracture Index		Type of bracket		Total
		Metallic	Ceramic	
0 - ceramic surface intact or in the same condition as before the bonding procedure	n [a]	23	19	42
	hp [b]	54.8%	45.2%	100.0%
	vp [c]	31.9%	26.4%	29.2%
1 - surface damage limited to glaze layer or very superficial ceramic	n	47	50	97
	hp	48.5%	51.5%	100.0%
	vp	65.3%	69.4%	67.4%
2 - surface damage which features a significant loss of ceramic requiring restoration of the defect or replacement of the restoration	n	2	3	5
	hp	40.0%	60.0%	100.0%
	vp	2.8%	4.2%	3.5%
Total	n	72	72	144
	hp	50.0%	50.0%	100.0%
χ^2 – test		χ^2=0.674	df=2	p=0.714

[a] count, [b] % within PFI, [c] % within type of bracket

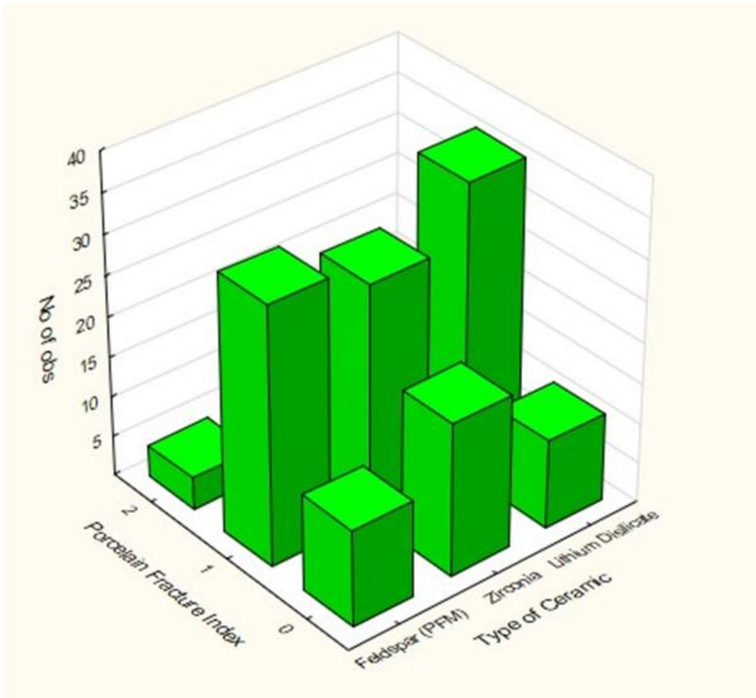


Figure 37. The bivariate histogram of PFI according to the type of ceramic.

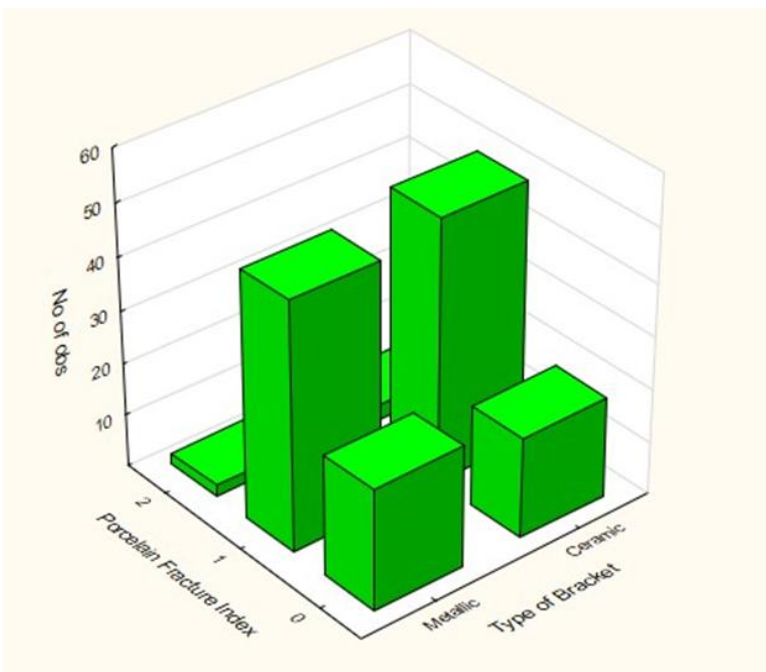


Figure 38. The bivariate histogram of PFI according to the type of bracket.

A significant interaction between PFI and t_etch was registered, meaning that PFI does depend on the type of etchant (Table 21).

Table 21. The cross tabulation of the PFI according to the type of etchant and χ^2 – test.

Porcelain Fracture Index		Type of etchant: Phosphoric Acid (PHA)	Type of etchant: Hydrofluoric Acid (HFA)	Total
0 - ceramic surface intact or in the same condition as before the bonding procedure	n [a]	27	15	42
	hp [b]	64.3%	35.7%	100.0%
	vp [c]	37.5%	20.8%	29.2%
1 - surface damage limited to glaze layer or very superficial ceramic	n	44	53	97
	hp	45.4%	54.6%	100.0%
	vp	61.1%	73.6%	67.4%
2 - surface damage which features a significant loss of ceramic requiring restoration of the defect or replacement of the restoration	n	1	4	5
	hp	20.0%	80.0%	100.0%
	vp	1.4%	5.6%	3.5%
Total	n	72	72	144
	hp	50.0%	50.0%	100.0%
χ^2 – test		χ^2=6.064	df=2	p=0.048

[a] count, [b] % within PFI, [c] % within type of etchant

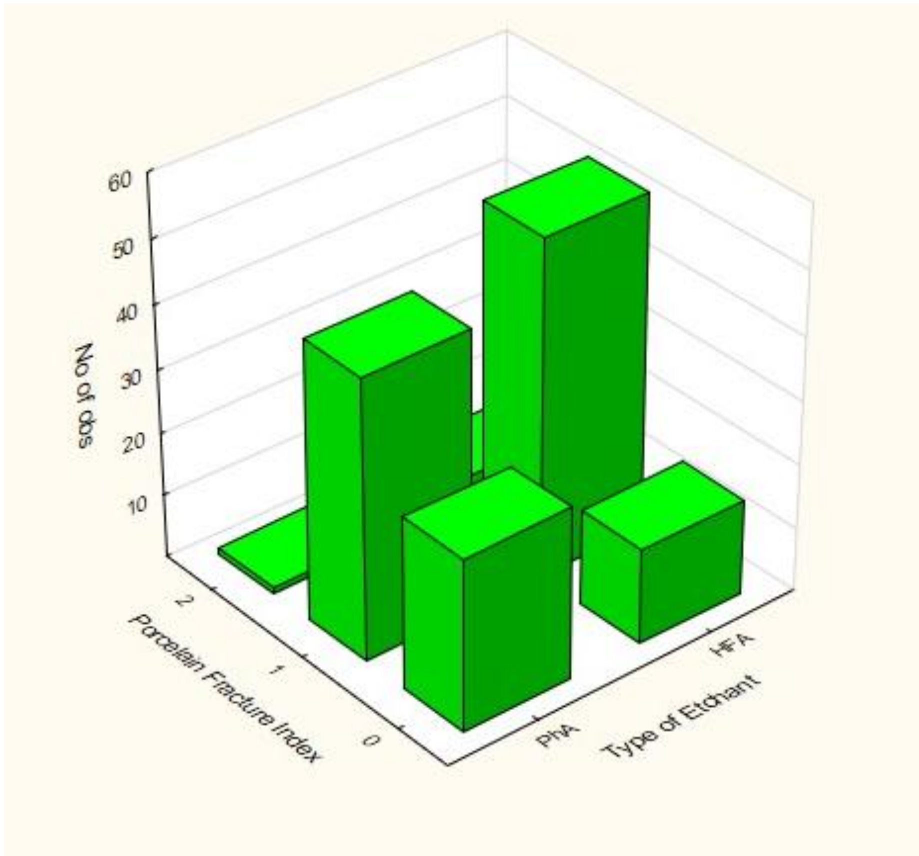


Figure 39. The bivariate histogram of PFI according to the type of etchant.

The SBS is not connected significantly with PFI categories. Namely, according to the results of the variance analysis, differences of the SBS averages by PFI categories are not statistically significant. (Tables 22 and 23). This is clearly evident in Figure 40.

Table 22. Descriptive statistics of the dependent variable SBS.

Porcelain Fracture Index	Shear Bond Strength (MPa)			
	n[a]	Mean	SD[b]	CV[c] (%)
0 - ceramic surface intact or in the same condition as before the bonding procedure	42	10.65	5.32	50.0
1 - surface damage limited to glaze layer or very superficial ceramic	97	11.49	5.79	50.4
2 - surface damage which features significant loss of ceramic requiring restoration of the defect or replacement of the restoration	5	10.39	7.07	68.0
Total	144	11.20	5.68	50.7

Legend: [a] number of cases, [b] standard deviation, [c] coefficient of variation

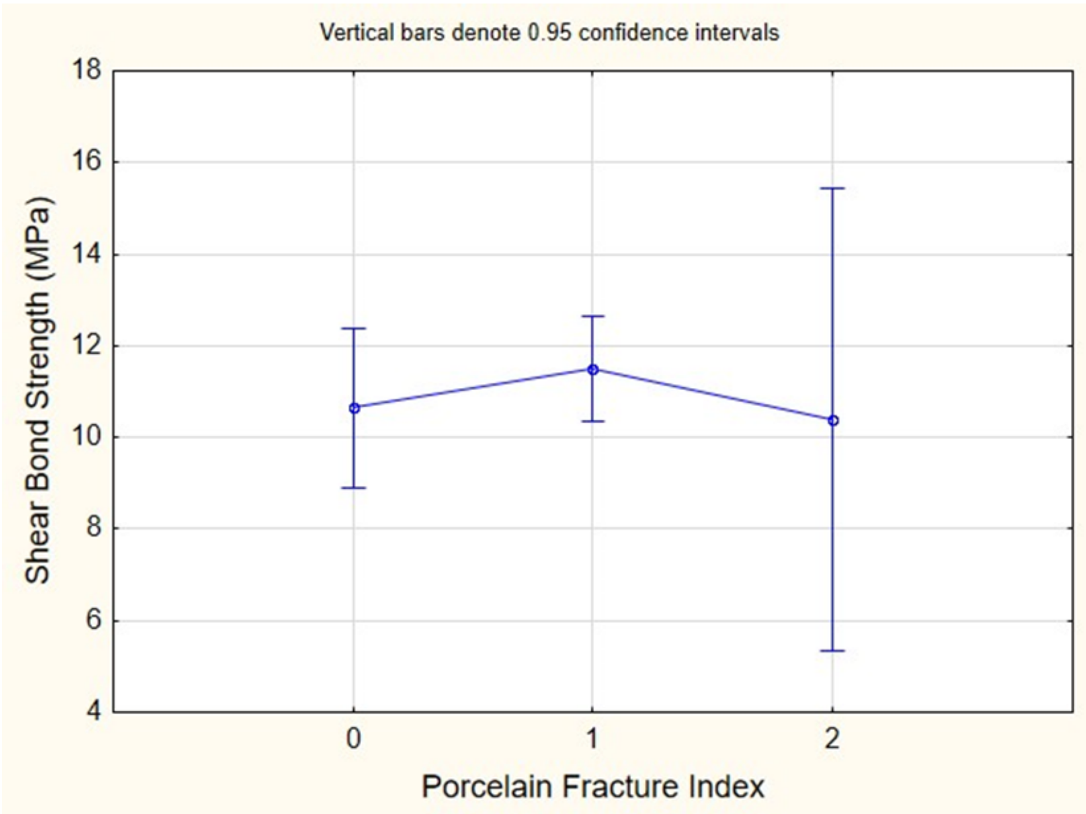


Figure 40. The means of the SBS by categories of the PFI.

Table 23. The univariate tests of significance for the SBS (MPa).

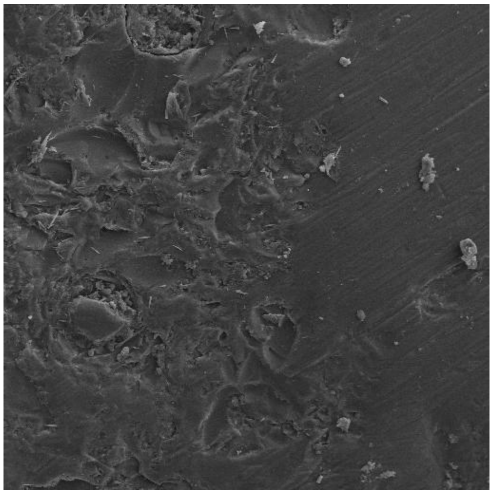
	Sum of Squares	df	Mean Square	F	p
Between Groups	23.937	2	11.969	0.368	0.693
Within Groups	4583.685	141	32.508		
Total	4607.622	143			

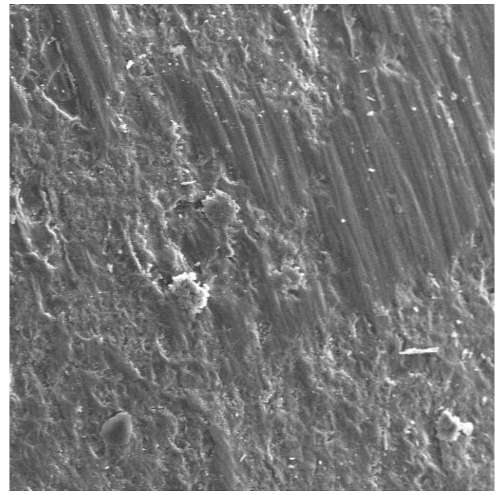
4.4 **Results from SEM analysis**

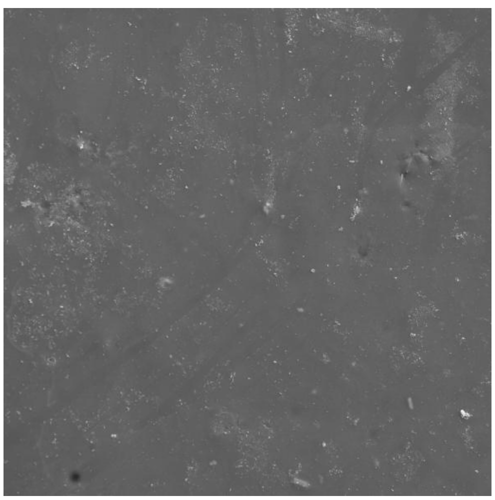
After an analysis of the surfaces before etching, after etching with hydrofluoric acid (HFA) and after etching with phosphoric acid (PHA), before bonding and after debonding the brackets, representative SEM micrographs of the surfaces from the three types of ceramic are investigated and presented in upcoming figures.

The SEM photomicrographs of all three ceramic surfaces etched with HFA revealed different surface morphologies. All-contour zirconia ceramic displayed fewer pits and more unchanged glazed surfaces than the feldspathic-porcelain fused to metal and lithium disilicate glass-ceramic.

In all three types of ceramic crowns etched with PHA, minor losses of the glazed surface and mild roughening were observed. Uniform peeling or an erosive appearance with shallow penetration and undercuts was also observed (Figures 41, 42 and 43)

d.


e.


b.


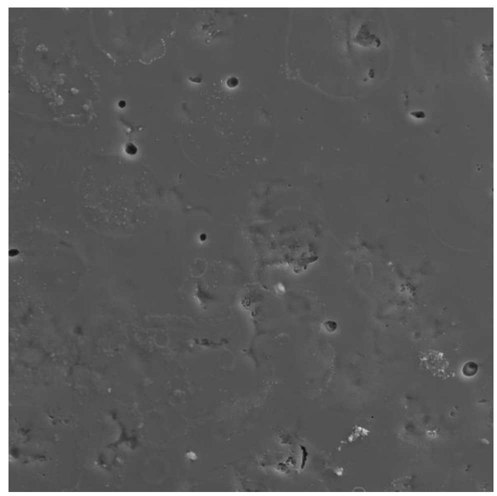
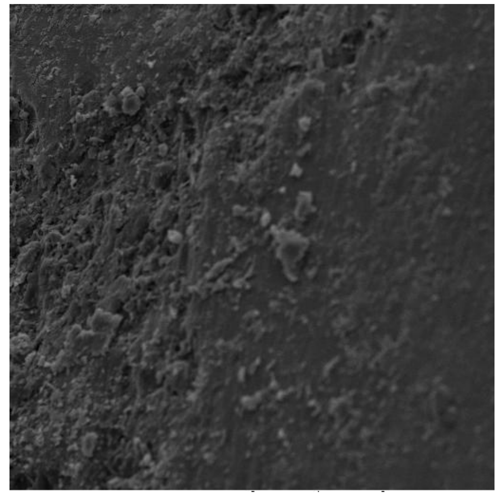
c.


Figure 41. SEM images from feldspathic - PFM surfaces: a. without etching (mag 1000 x); b. etched with PHA before bonding (mag 1000 x); c. etched with HFA before bonding (mag 1000 x); d. etched with PHA after debonding (mag 400 x); c. etched with HFA after debonding (mag 400 x).

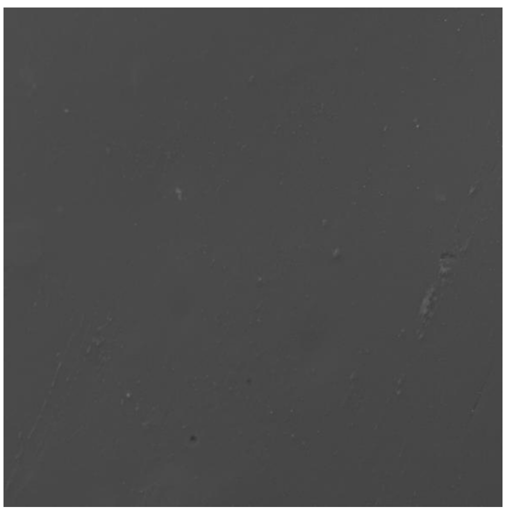
a.


d.

e.



b.



c.

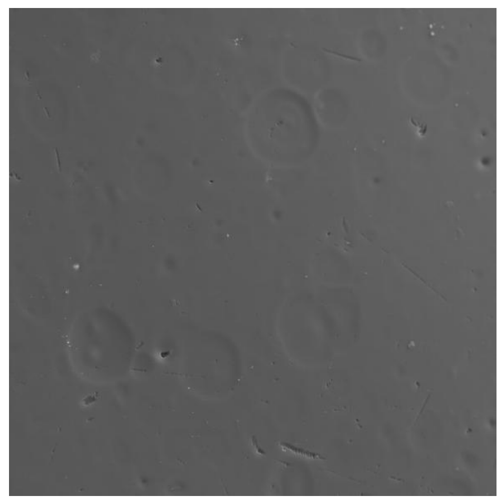
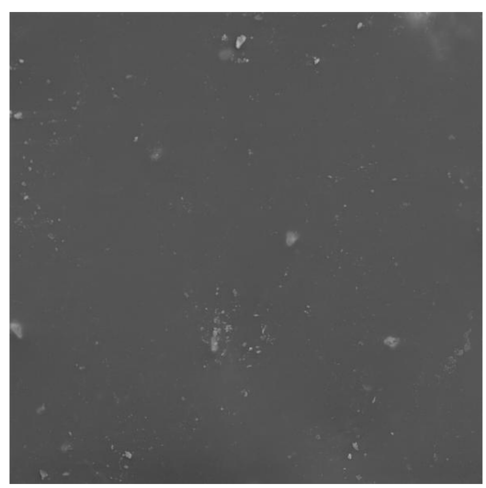
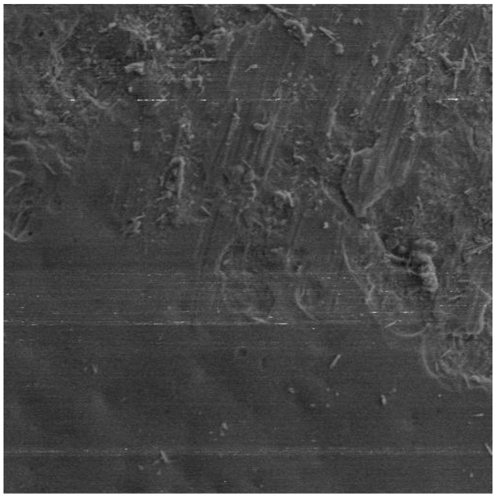
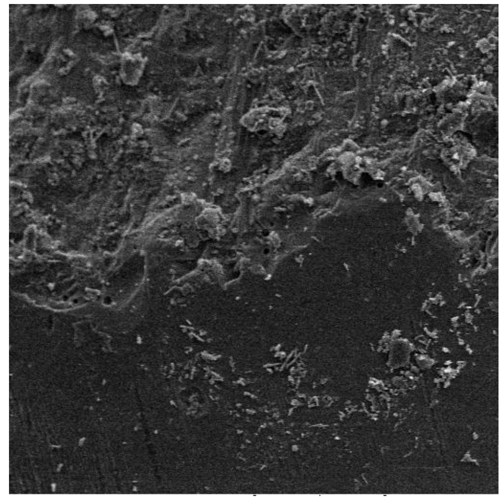


Figure 42. SEM images from full-contour zirconia surfaces: a. without etching (mag 1000 x); b. etched with PHA before bonding (mag 1000 x); c. etched with HFA before bonding (mag 1000 x); d. etched with PHA after debonding (mag 400 x); c. etched with HFA after debonding (mag 400 x).

a.




d.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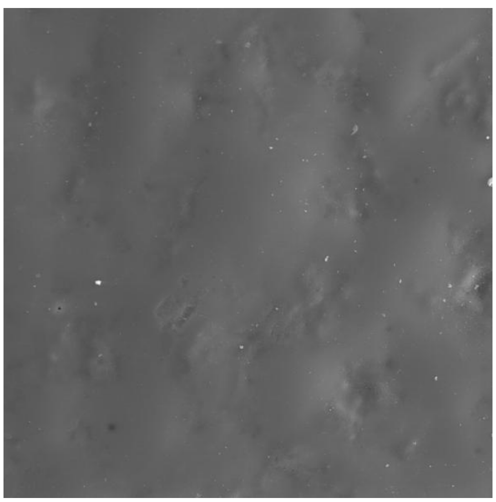
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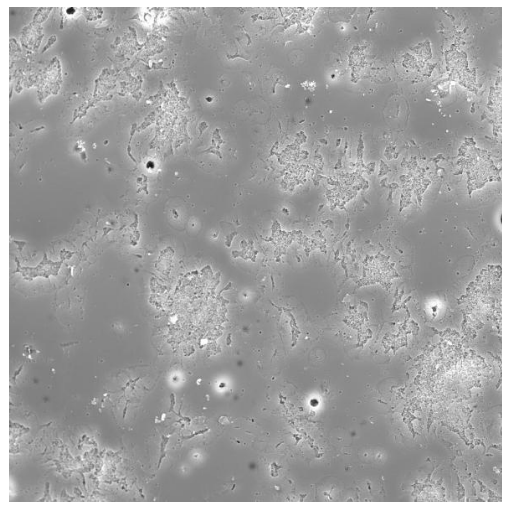
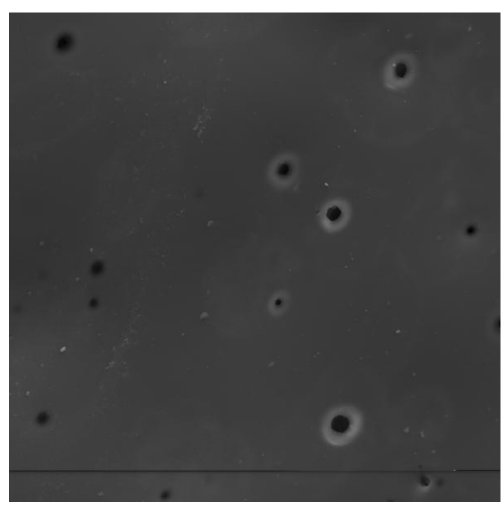

c.

Figure 43. SEM images from lithium disilicate-glass ceramic surfaces: a. without etching (mag 1000 x); b. etched with PHA before bonding (mag 1000 x); c. etched with HFA before bonding (mag 1000 x); d. etched with PHA after debonding (mag 400 x); c. etched with HFA after debonding (mag


a.

5. DISCUSSION

5.1 Study rationale and evaluation of factors interaction

The rapid breakthrough of innovative ceramic materials in dentistry and the rising demand for adult orthodontics have resulted in the need to properly bond orthodontic brackets to various ceramic restorations. Considering the rising demand for an aesthetic facial appearance that led to an increase in the number of adult orthodontic patients with ceramic restorations, there is still no consensus regarding the most efficient ceramic conditioning protocol for gaining optimal bond strength (10,12,88).

The present study evaluated the influence of two different surface etching acids, and two different bracket materials on the SBS of orthodontic brackets bonded to three different ceramic surfaces, which are widely used as restorative material in dentistry. We hypothesized that the SBS of orthodontic brackets bonded to ceramic surfaces is affected by the different ceramic surfaces and by the material the bracket consists of, but it is not affected by the type of etchant applied, and that phosphoric acid in combination with silane is a reliable conditioning alternative for ceramic surfaces prior to bonding.

When bonding brackets to ceramic surfaces, double challenges arise. Optimal bond strength of 6 to 10 MPa is desired to minimize bond failure during the treatment period (13), while on the other hand after debonding procedure, the ceramic restorations should remain in the same condition with their ideal aesthetics and function (11,89). However, the transfer of this value in clinical situations is questionable because of the complex environment of the oral cavity (44). In the present research, most of the groups showed optimal mean values of the SBS. The lowest mean value of the SBS was 8,52 MPa (in the group 7), and the highest mean value of the SBS was 14.75 MPa (in the group 4), which could cause fractures of the ceramic (89).

This research was conducted under in vitro conditions, and it is not always possible to compare it with clinical situations. According to previous research, thermocycling weakened bond strength from a mean of 18.69 – 9.53 MPa (13). However, another study indicated that thermocycling had no significant effect in SBS (90). In the present study, the specimens were thermocycled as a means to artificially age the bond prior to testing, as recommended in previous studies (8,44).

A minimum of 10 specimens is recommended to perform SBS testing (91). However, a greater sample size than 10 per group is recommended for bond strength testing on natural teeth due to variations in tooth shape (80). In this research, different ceramic crowns were divided into groups containing 12 semi-crowns fabricated by a single operator simulating the buccal half of the maxillary first premolar. The premolar tooth form was selected to allow a clinical simulation and to compare the outcome of the present study with previously reported investigations (5,13,20,51).

In addition, because of the complexity of the oral environment, it should be taken into consideration that there are limitations of in vitro studies, and that there might be differences between in vivo and in vitro bond strengths, especially when bonding to other restorative materials. However, despite the limitations, SBS testing remains a relevant methodology to compare bonding protocols by providing important information regarding bracket debonding in clinical situations (82).

5.1.1 Triple interaction between factors

In this research, the triple interaction of factors, which are the type of ceramics, the type of bracket and the type of etchant in the applied model of analysis of variance was not significant. However, the post hoc analysis with LSD method reveals six pairs of groups, which differ significantly in the SBS values.

The first significant difference between groups was noticed between group 1 - feldspar ceramic with metal bracket and etching with PHA (mean SBS = 9.89 MPa) and group 4 - the same ceramic surface with ceramic bracket and etching with HFA (mean SBS = 14.75 MPa), indicating a better performance of the second combination.

Significantly better performance showed group 3 - feldspar (mean SBS = 14.10 MPa) versus group 7 - zirconia (mean SBS = 8.52 MPa) under the same conditions, i.e. in combination with a ceramic bracket with PHA surface preparation.

A significant difference between the group 9 - lithium disilicate ceramic, metal bracket and PHA surface preparation (mean SBS = 10.20) and the group 4 - feldspar porcelain with ceramic bracket and HFA surface preparation (mean SBS = 14.75) was visible, indicating a better performance of the group 4.

Also, a significant lag of the group 7 - zirconia combined with ceramic bracket and PHA surface preparation (mean SBS = 8.52 MPa) from the group 4 - feldspar porcelain in combination with ceramic bracket and HFA surface preparation (mean SBS = 14.75 MPa).

Other significant difference was noticed between the group 8 - zirconia in combination with ceramic bracket and HFA-prepared surface (mean SBS = 8.99 MPa) and with the group 3 - feldspar in combination with ceramic bracket and PHA surface preparation (mean SBS = 14.10 MPa), indicating a better performance of the second combination.

The last significant difference in the SBS between groups was marked with the group 8 - zirconia with ceramic brackets on HFA surface preparation (mean SBS = 8.99 MPa) and the group 4 - feldspar also with ceramic bracket on HFA prepared surface (mean SBS = 14.75 MPa), also indicating a better performance of the second combination.

These findings are in accordance with various studies (13,17,20,23,44,51,76,85,92) revealing the fact that the SBS of orthodontic brackets bonded to ceramic surfaces can be affected by the a combination of different materials, as well as by other procedures prior to bonding. This is not different than bonding to enamel, where a simple procedure like fluorosis reduces bond strength of orthodontic brackets (93).

5.2 Bond strength depending on method of surface conditioning

The results of the triple factorial analysis of variance showed no significant influence ($p = 0.608$) by the type of etchant (5 % hydrofluoric acid and 37 % phosphoric acid) used in this study.

It has been recommended that the methods providing sufficient bond strength with less roughening should be used to avoid microcracks on the ceramic surface (13,23,27). Consequently, in this study no sandblasting or other mechanical roughening was applied. The brackets were bonded to a glassed ceramic surface, after chemical conditioning, in order to determine which etching material is the most appropriate for the use in the clinical work.

Zachrisson *et al.* (1996) advocated the use of strong acids such as 9.6 per cent HFA to etch porcelain in order to increase bond strength (27). However, HFA should be used with great care as it is capable of causing severe trauma to soft tissues and tooth substance (23).

According to Bourk and Rock (1999) removal of the porcelain glaze, or use of hydrofluoric acid, prior to bonding were found to be unnecessary to secure the target bond strength. Also, hydrofluoric acid application was associated with increased porcelain surface damage (13).

Phosphoric acid (37.0%) cannot etch a ceramic surface, but has the ability to neutralize the alkalinity of the absorbed water layer, which is present on ceramic restorations in the mouth and thereby improves the chemical activity of the silane primer that is subsequently applied (12).

It has been reported that silane application after ceramic surface roughening provides a chemical link between porcelain and composite resin, and that it increases the bond strength of orthodontic attachments (8,42,51).

Being aware that in clinical situations the etching with HFA must be used with great caution, as it is extremely corrosive and capable of causing severe trauma (13,23,27,41), we aimed to devise an alternative protocol involving etching with less dangerous materials, such as phosphoric acid in interaction with silane.

Previous studies have shown that optimal bond strength ranges from 6 to 10 MPa (17). Nevertheless, this is not universally accepted in clinical situations, because the bracket-ceramic surface bond is affected by many environmental factors (12).

The present study was performed under in vitro conditions, and in all groups the mean SBS values were higher than 8.5 MPa, and less than 14.8 MPa, which might clinically cause cohesive fractures. Since it was found that the PHA-etched groups had similar bond strengths to those etched with HFA, and the difference between these groups was not significant concerning the etchant where all the groups showed high bond strengths, as well as considering the harmful effects of HFA, our results may therefore indicate that a combination of phosphoric acid with silane is sufficient and that there is no need to use HFA to achieve a higher bond strength. Therefore, our findings indicate that the use of HFA is unnecessary for conditioning the ceramic surface before bonding orthodontic brackets. This is in accordance with previously reported findings (12,13,20,94–96), but in contrast with others (43).

Furthermore, according to the results from the PFI, HFA significantly damages the surface structure of the ceramic, and considering its noxious effect, it is not the best suitable conditioner prior to orthodontic bonding for feldspar, lithium disilicate, and in particular for all-contour zirconia. This has been concluded by also taking into account the crystalline structure of zirconia, and because it showed the weakest bond strength with orthodontic brackets.

5.3 **Bond strength depending on ceramic type**

In this research, according to the triple factorial analysis of variance the type of ceramics did not significantly affect the averages of SBS ($p = 0{,}127$), and only in interaction with the type of bracket the significant difference was noticed.

The absence of a significant influence is clearly noticed in the interaction of the ceramic type to form SBS values in combination with the surface preparation in either of the six possible pairs. However, there is a noticeable shift of the feldspar ceramic from the other two ceramic materials, equally for both etching methods, which means that both these methods tend to give better results to feldspar rather than to lithium disilicate, and even better than to zirconia.

Despite that, according to the post hoc analysis, significant differences between groups with different ceramic types were noted. The highest average values were gained at feldspar - porcelain fused to metal groups (3 and 4) with ceramic brackets and both etchants, PHA and HFA, meaning that SBS for feldspar material is on average larger than the other two materials, and according to the post hoc analysis with LSD method compared to zirconia is significantly larger. These results are not in accordance with other reports (40,85).

The SBS value of feldspar ceramics in combination with the ceramic bracket is significantly higher than the value of lithium disilicate in combination with a metallic bracket. This is in accordance with Alhaija and Wahadni (2007), who observed significant differences between feldspathic and lithium disilicate ceramic restorations, with a higher mean SBS reported in the feldspathic porcelain group (23). However, Turk *et al*. (2016) reported that lithium disilicate had a higher SBS than feldspathic porcelain restorations (85). This may be due to the differences in the processing methods and the molecular structure of the ceramic restorations.

Zirconia combined with a ceramic bracket achieves an average SBS of 8.75 MPa which is significantly smaller than 14.43 MPa achieved by the feldspar with a ceramic bracket. This is assumed to be because the low level of silica on the zirconia surface might have affected the establishment of a siloxane network between the silane coupling and the ceramic surface (67,68).

5.4 Bond strength depending on bracket type

The SBS by type of bracket alone does not differ significantly, because both mean values are almost equal. However, in interaction with the type of ceramics significant differences were noticed. The average SBS of metallic brackets bonded to feldspar ceramic type is 10.36 MPa, while of the ceramic bracket is significantly higher, 14.43 MPa.

Between the groups, feldspar groups (3 and 4) with ceramic brackets showed significantly higher results. The group 11 (lithium disilicate combined with PHA and ceramic bracket), showed high results, but not statistically significant in comparison to metallic bracket groups. In general, ceramic brackets bonded to lithium disilicate samples, compared to those bonded to zirconia, showed slightly, but not significantly higher SBS values. The highest difference between the two ceramics was registered in ceramic bracket and phosphoric acid groups, probably due to the variations of the molecular structures of the two all-ceramic systems.

According to Al-Hity *et al*. (2012), as well as other studies (9,11,16,22,37,43), the bond strength of the ceramic brackets is higher than the bond strength of the metallic brackets, due to a stronger adhesion to ceramics and light transmission, which leads to a higher degree of polymerization and stress reduction on the adhesive-bracket joint. The findings of this research are partly concurrent with the above-mentioned studies. The bond strength of the polycrystalline ceramic brackets bonded to feldspar and partially of those bonded to lithium disilicate ceramic crowns is higher than the SBS of the metallic brackets. This is promising for adult orthodontics, due to better aesthetics of the ceramic brackets during orthodontic treatment.

However, our results indicate that this is not the case for orthodontic brackets bonded to zirconia ceramic crowns. As previously reported by Mehmeti *et al.* (2017) (33) metal brackets, in comparison with ceramic polycrystalline brackets, create better adhesion with all-zirconia surfaces. This might be because mechanical coupling is greater than chemical coupling of the brackets with zirconia ceramic surface, and the base surface design or retention mode of orthodontic brackets plays a determinant role in their bond strength.

5.5 Type of adhesive failure

A modification of the ARI, which divided the scale into 5 scores to provide an accurate evaluation of the adhesive remaining on the ceramic surface, has been previously reported (61).

In the present study, the ARI does not depend statistically on the type of ceramics or on the type of etchant, which means that the occurrence of any of the ARI categories cannot be related to the influence of these two factors.

ARI scores indicated that there was a combined frequency of bond failure at the bracket-adhesive interface and at the adhesive-ceramic interface. These results are in accordance with other reported findings (13,27).

The type of brackets significantly affects the appearance of ARI categories. The importance of this dependence lies in the fact that the first two categories (1 and 2) of ARI appear significantly more with metallic brackets.

Regardless of the ceramic type and their surface conditioning, the samples with metallic brackets have shown mixed adhesive-cohesive failures, with a higher frequency of bond failure at the bracket-adhesive interface in metallic bracket groups, compared to the ceramic bracket groups, independent of the etchant applied. In most of the samples with ceramic brackets, adhesive failures between the ceramic and composite resin were noticed. The 3rd, the 5th, and especially the 4th category of ARI is more frequent in ceramic brackets, which indicates that the bond strength between the composite and the ceramic bracket was stronger than the bond strength between the composite and ceramic crown. These are similar to previously reported findings (27,96), and are different from the study conducted by Abu Alhaija *et al.* (2010) (20).

We gained a significant connection of ARI with the type of bracket treated with PHA. It is argued that categories 1 and 2 of ARI occur only with metallic brackets, while the other categories are dominated by ceramic brackets.

To avoid the ceramic breakage during debonding, adhesive failures at the ceramic/composite interface are preferred (90). According to Zachrisson *et al.* (1996), the debonding strength values may represent the true adhesive force of composite to porcelain only if cohesive fractures can be avoided (27). Our findings are partially in concordance with this. According to a previous study, adhesive failures are usually associated with lower bond strength values (98). Notwithstanding, the type of adhesion (mechanical interlocking or chemical bonds) also have an influence on the failure mode (58).

Although, we found that the SBS shows on average an increase in ARI categories, the variance analysis results show that this is not significant (p=0.072).

5.6 **Ceramic integrity after bracket debonding**

Although in four samples of feldspar the damages of the second scale of the PFI were registered, in one lithium disilicate sample and in none of zirconia samples, as well as in all scores zirconia had better results, still a significant difference was not registered in the analysis, which means that PFI does not depend on the type of ceramics, but it does indicate a better performance of the zirconia.

Also, PFI does not depend on the type of bracket, and SBS is not connected significantly with PFI categories.

Furthermore, the analysis of PFI showed that in none of the all-ceramic types larger fractures or cracks were observed, which is clinically important for the long-term integrity of the restoration.

The significant difference that was noticed between two etchants regarding PFI is in agreement with the studies of Bourke and Rock (1999) (13) and Alhaija *et al.* (2010) (20), what indicates that the use of HFA can also make the surface of all the ceramic materials more vulnerable. This is another argument for avoiding the use of HFA as a surface conditioning method for ceramic surfaces prior to orthodontic bonding.

According to Zachrisson (2000), thermocycling was necessary for testing silane-coupled bonds to porcelain. If thermocycling was not done, the bond strengths to porcelain and the incidence of cohesive porcelain fractures were excessively high (44).

6. CONCLUSION

1. The use of HFA for surface etching of feldspar, all-contour zirconia and/or lithium disilicate, does not cause a significant increase in the SBS values as compared to etching with PHA and silane application.

2. According to the results from the PFI, HFA significantly damages the surface structure of the ceramics, and it is not the best suitable conditioner prior to orthodontic bonding.

3. The SBS of ceramic polycrystalline brackets bonded to feldspar and partialy to lithium disilicate is higher than the SBS of the metallic brackets.

4. Metallic brackets, in comparison with ceramic polycrystalline brackets, create better adhesion with all-zirconia surfaces.

5. Ceramic integrity after debonding of orthodontic brackets does not depend on the type of ceramics, although the results showed a slightly better performance of zirconia.

6. According to the triple factorial analysis of variance there is no significant influence by the type of ceramics on the SBS, but according to the post hoc analysis there is a difference between the results of different ceramic types, and a significant difference between zirconia and feldspar, indicating for a better performance of the last one.

7. Regarding orthodontic point of view, since the SBS values were within the range of optimal bond strength, all three types of ceramic restorations, as well as both types of brackets and both types of etchants, provide sufficiently strong bond strength to realize the treatment.

7.LITERATURE

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8. CURRICULUM VITAE

Blerim Mehmeti was born in the year 1978, in Pristina, Republic of Kosovo, where he finished his studies in Dentistry in the year 2005, attaining the title Doctor of Dental Medicine. In 2010 he finished his specialization in Orthodontics, attaining the title Specialist of Orthodontics.

After graduating he started working in private practice and founded Dental Clinic "Albadent". In the year 2007 he started working as Teaching Assistant of Orthodontics in Medical Faculty - School of Dentistry, University of Pristina, and in the year 2012 as clinical specialist in the Department of Orthodontics, University Dental Clinical Centre of Kosovo.

In October 2014 he started his PhD studies at the School of Dental Medicine, University of Zagreb, Croatia.

He was in the organizing committee of several conferences, as well as he actively participated in numerous international scientific conferences as author and co-author of more than 50 oral and poster presentations. He is also author and co-author of 9 publications, in relevant international journals.

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List of published articles

Scientific articles:

1. **Mehmeti B,** Haliti F, Azizi B, Kelmendi J, Iljazi-Shahiqi D, Jakovljević S, Anić-Milosević S. Influence of different orthodontic brackets and chemical preparations of ceramic crowns on shear bond strength. Australasian Medical Journal. 2018;11(2):107-112..

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