

# Bonding of orthodontic ceramic brackets: optimal conditioning method of lithium disilicate restorations

Stefan Kuzmanovski<sup>1</sup>, Cvetanka Bajraktarova Mishevska<sup>2</sup>, Aneta Mijoska<sup>1</sup>, Natasha Stavreva<sup>1</sup>, Emilija Bajraktarova Valjakova<sup>1</sup>

<sup>1</sup>University „Ss Cyril and Methodius“, Faculty of Dentistry, Department of Prosthodontics, Skopje, Republic of North Macedonia;

<sup>2</sup>University „Ss Cyril and Methodius“, Faculty of Dentistry, Department of Orthodontics, Skopje, Republic of North Macedonia

## SUMMARY

**Introduction** According to the data obtained from the American Orthodontic Society survey conducted in 2015, the number of adult patients that requested orthodontic treatment increased from 14% to 27% between 2010 and 2014. Because of the increased age of the patients, fixed orthodontic treatment is expected to be performed on restored teeth, including all ceramic restorations. The objectives of the present study were to evaluate the influence of different surface treatments on the bond strength of ceramic brackets to lithium disilicate ceramic and to determine the mode of fracture.

**Material and method** 45 ceramic rectangular specimens were obtained from lithium disilicate CAD/CAM blocks which were divided in five groups according to the performed surface conditioning method: 1. grinding with a fine diamond bur (control group); 2. sandblasting with 29 µm alumina (Al<sub>2</sub>O<sub>3</sub>); 3. etching with 4% hydrofluoric acid (HF); 4. sandblasting followed by conditioning with a universal primer – silanization (Al<sub>2</sub>O<sub>3</sub> + S); 5. HF acid etching followed by silanization (HF + S). Shear bond strength test was performed after storing the samples in water bath for 7 days. All of the fractured samples were analyzed with optical microscope to determine the type of fracture.

**Results** HF acid etching followed by silanization performed the highest bond strength of 8.03 MPa, while sandblasting followed by silanization – 6.69 MPa. Mechanical surface conditioning with either HF acid etching or sandblasting resulted in significantly lower bond strength (2.65 MPa and 1.51 MPa respectively). Mainly adhesive mode of fractures was noticed after sandblasting and silanization, indicating minor chance of damaging the ceramic restoration after debonding, at the end of the orthodontic treatment, unlike the ceramic specimens in the Group 5 (HF + S) with 42.8% mixed and 14.4% cohesive fractures; 100% adhesive fractures were observed after mechanical treatments.

**Conclusion** According to the SBS test results and fracture type, sandblasting followed by the application of a universal primer can be considered as an adequate method for conditioning the lithium disilicate ceramic restorations before the bonding of ceramic orthodontic brackets.

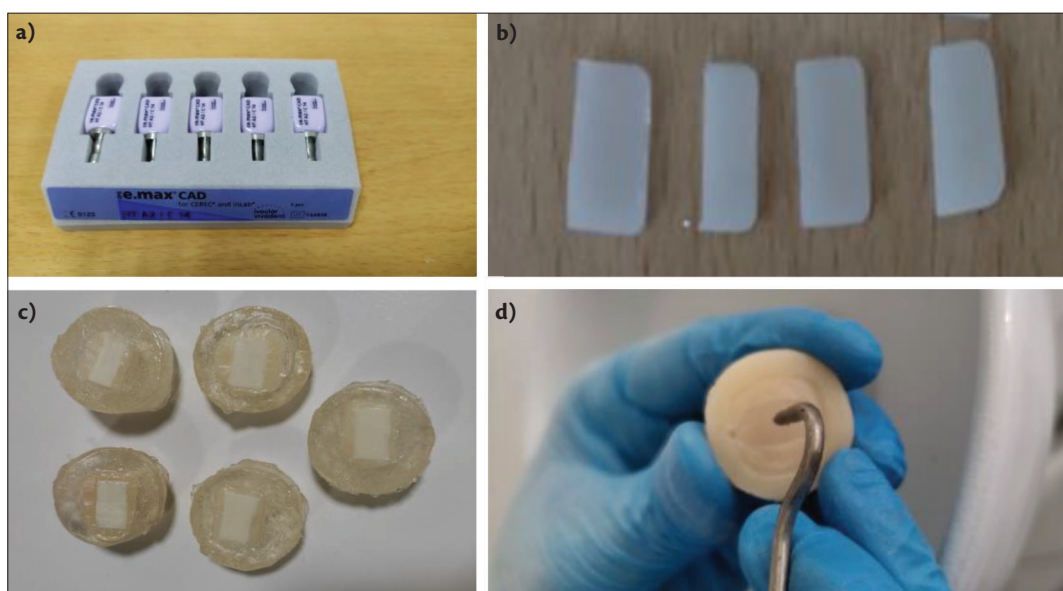
**Keywords:** ceramic bracket; conditioning methods; lithium disilicate restoration; sandblasting; hydrofluoric acid; universal primer

## INTRODUCTION

According to the data obtained from the survey of the American Orthodontic Society conducted in 2015, the number of adult patients that requested orthodontic treatment increased from 14% to 27% between 2010 and 2014; this means that the number of adult orthodontic patients has doubled in just 4 years [1]. Therefore, in daily clinical practice, treatment with orthodontic brackets can be expected to be performed on a dentition previously treated with fixed-prosthetic restorations – metal-ceramic or all-ceramic crowns or veneers. On the other hand, debonding of the brackets during the orthodontic treatment is an often problem, resulting in patients' frustration and dental appointments that are scheduled more frequently, with performing of the entire procedure for re-bonding the brackets. Obtaining an adequate bonding of the brackets to

the restorations is of a great importance for the successful orthodontic treatment. However, the bond strength should not be too strong in order to avoid damage of the ceramic restorations during the debonding process of the brackets at the end of the orthodontic treatment [1].

The introduction of glass ceramics as restorative materials in prosthodontics, as well as the advantages of adhesive cementation, contributed to the development of the concept of ceramic etching with hydrofluoric acid (HFA). However, HFA possesses corrosive potential causing burns and necrosis of the tissue at the contact site, while if absorbed into the bloodstream, fluoride ions are distributed to all organs and tissues, provoking potential life-threatening systemic toxicity and organs failure. All this imposes an extraordinary caution and great awareness of health consequences when HF acid is used intraorally [2]: exposure of the oral mucosa to a concentration of 0.1%



**Figure 1.** a) Lithium disilicate presintered CAD/CAM blocks; b) Ceramic samples after sintering; c) Prepared acrylic molds with ceramic samples; d) Air abrasion

**Slika 1.** a) Litijum-disilikatni presinterovani CAM/CAM blokovi; b) Keramički uzorci nakon sinterovanja; c) Pripremljeni akrilni kalupi sa keramičkim uzorcima; d) Vazдушna abrazija



**Figure 2.** a) Etching the samples with 4% HFA; b) Prepared samples from group 4; c) Prepared samples from group 5

**Slika 2.** a) Nagrizanje četvoroprocentnom fluorovodoničnom kiselinom; b) Pripremljeni uzorci Grupe 4; c) Pripremljeni uzorci Grupe 5

HFA can lead to mucosal necrosis [3]. When etching glass ceramics with 9.6% HFA, a double reaction occurs: primary - between the acid and the ceramic glass phase, and secondary - between the acid and the crystalline phase, leaving the larger crystals intact. In this way, an irregular surface is created with microscopic pores that enable the micromechanical interlocking of the adhesive resin. The more the crystalline phase is present, and the less the glass phase in the composition of the material, the greater is resistance of the ceramic to the acid [4].

Sandblasting using aluminum oxide ( $Al_2O_3$ ) particles is another method for mechanical conditioning of the bonding surface of ceramic restorations causing an irregularity, which is required for micromechanical retention of the bonding agent. However, air abrasion may cause irreversible damage of the ceramic restorations. Therefore, it is recommended that  $Al_2O_3$  sandblasting should be performed under low pressure (1-2 bars), using grain size less than  $50 \mu m$  and at a distance of 10 mm perpendicular to the ceramic surface [5].

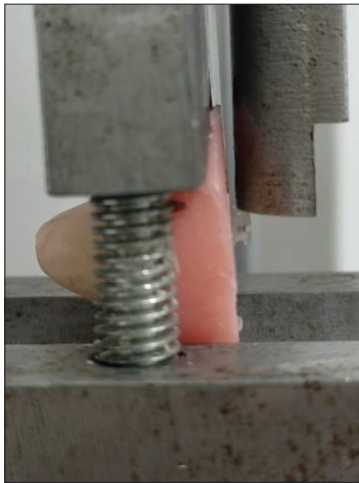
After mechanical treatment, in order to achieve an optimal bond with the composite luting cement, chemical conditioning with primers that contain silane is recommended. The silane molecule has two different functional groups: the -SiOH group that bonds to the silicon dioxide molecules

and another organic functional group that bonds to the methacrylate resin of the composite cement [6].

The aim of the present study was to evaluate the influence of different surface conditioning methods on the bond strength of ceramic brackets to lithium disilicate ceramic and to determine the mode of fracture.

## MATERIAL AND METHOD

Ceramic sections (2 mm thick) were obtained from presintered lithium disilicate CAD/CAM blocks IPS e.max CAD (Ivoclar Vivadent, Schaan Liechtenstein) with a diamond blade (Figure 1a) using a Minitom precise cutting machine (Struers, Denmark) (Figure 1b); cutting was performed with permanent water cooling to prevent overheating of the ceramic material that may cause microcracks. All the ceramic sections were sintered in a ceramic furnace Programat EP 5000 (Ivoclar Vivadent, Schaan Liechtenstein). Sintered ceramic sections were immersed in the middle of the metal ring molds ( $d = 30 \text{ mm}$ ) filled with freshly mixed cold-polymerizing acrylate polymethyl methacrylate - PoliTEMP (PoliDent, Slovenia), with an exposed ceramic surface that was used as a bonding surface for the ceramic brackets (Figure 1c). After that, the



**Figure 3.** Shear bond strength test  
**Slika 3.** Ispitivanje čvrstoće veze na smicanje

bonding surface of all ceramic samples was grinded with a fine diamond bur. All ceramic samples were randomly divided into 5 groups according to surface treatment:

1. Control group = no further treatment;
2.  $\text{Al}_2\text{O}_3$ : the bonding surfaces were sandblasted with 29- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles – Sandman (Innovative Micro Dentistry, Poland), perpendicular to the bonding surface, for 10 seconds, under a pressure of 1 bar and at 10 mm distance (Figure 1d). Surfaces were cleaned with air blowing for 5 sec.
3. HF: the bonding surfaces were etched with 4% hydrofluoric acid – IPS Ceramic etching gel (Ivoclar Vivadent, Shaan Liechtenstein) for a period of 20 seconds. Surfaces were thoroughly rinsed with water for 60 sec to remove the residual acid and dried with compressed air (Figure 2a).
4.  $\text{Al}_2\text{O}_3$  + S: the bonding surfaces were sandblasted with 29- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  particles using a blasting procedure as in Group 2. After that, a universal primer – Monobond Plus (Ivoclar Vivadent, Shaan Liechtenstein) was applied in a thin coat and left to react for 60 seconds (Figure 2b). Any remaining excess was dispersed with a gentle stream of air.
5. HF + S: the bonding surfaces were etched with 4% hydrofluoric acid for 20 s, rinsed and dried. A universal primer – Monobond Plus was applied and left to react for 60 seconds (Figure 2c).

### Bonding procedure

After surface treatment, ceramic brackets for maxillary central incisors – Cosmetic 20/40 UR Central (American Orthodontics, USA) were bonded to a treated surfaces using orthodontic composite luting cement No Mix:30™ One step Adhesive – (American Orthodontics, USA) with a constant vertical load of 1 kg, for 1 min. The composite cement was left to set in its self-curing mode. The samples were stored in distilled water at 37°C for 7 days – Biobase Water Tank WT-42 (Biobase Biodustry, Shandong, China) thus imitating the conditions in the oral cavity (moisture and temperature).

### Shear bond strength test

The shear bond strength test (SBS) was carried out using a universal testing machine – Shimadzu AGS-X (Shimadzu Co., Japan), at a speed of 0.5 mm/min, for determining bond strength between orthodontic brackets and ceramic surfaces (Figure 3). The SBS was expressed in MPa, derived by dividing the imposed force (N) at the time of fracture by the bonding area of the ceramic bracket ( $\text{mm}^2$ ) ( $\text{MPa} = \text{N}/\text{mm}^2$ ).

### Mode of fracture

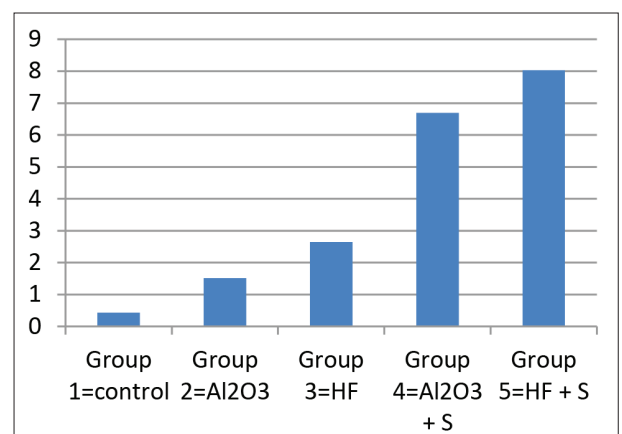
The mode of fracture (adhesive, cohesive in luting cement or ceramic bracket or mixed) for each specimen was determined using a light microscopy – Levenhuk Zeno Cash ZC6 (Levenhuk Inc., USA) at a magnification of 60 $\times$ .

### Statistical Analysis

The analysis of categorical variables was done by determining coefficient of relationships, proportions and rates. Continuous variables were analyzed with measures of central tendency (mean, median, minimum and maximum values), as well as with measures of dispersion (standard deviation). Shapiro-Wilk W test was used to determine the normality of the frequency distribution of the studied variables. Pearson Chi square test was used to determine the association between certain attributive dichotomous features.

### RESULTS

The SBS results after different surface treatments are recorded in Figure 4 and Table 1. The highest bond strength,  $8.04 \pm 0.61$  MPa, was recorded when ceramic brackets were bonded to ceramic surfaces that were etched with hydrofluoric acid followed by silanization (Group 5).



**Figure 4.** Mean SBS (MPa) after different conditioning treatments  
**Slika 4.** Srednje vrednosti SBS-a (MPa) posle različitih metoda kondicioniranja površine

Insignificantly lower bond strength ( $p = 0.1477$ ) was achieved when sandblasting the bonding surfaces with  $29\text{-}\mu\text{m Al}_2\text{O}_3$  followed by silanization (Group 4) –  $6.69 \pm 1.20$  MPa. When mechanical treatments were performed only, HF acid etching (Group 3) or sandblasting (Group 2), the bond strength was significantly lower,  $2.65 \pm 0.84$  MPa and  $1.52 \pm 0.43$  MPa, respectively. The lowest SBS was recorded after grinding the ceramic surfaces with fine diamond burr (control group),  $0.43 \pm 0.23$  MPa.

Adhesive fractures after debonding were exclusively present in the first three groups (100%), whereas in the Group 4 it showed dominant presence (71.4%). Mixed and cohesive modes of fracture in ceramic brackets were found only in the Groups 4 and 5 (Table 1).

**Table 1.** Mode of fracture after debonding  
**Tabela 1.** Tip frakture nakon debondiranja

	Mode of fracture Tip frakture		
	Adhesive Adhezivni (%)	Cohesive Kohezivni (%)	Mixed Pomešani (%)
Group 1 Grupa 1	100	/	/
Group 2 Grupa 2	100	/	/
Group 3 Grupa 3	100	/	/
Group 4 Grupa 4	71.4	14.3	14.3
Group 5 Grupa 5	42.8	14.4	42.8

## DISCUSSION

The aim of the present study was to evaluate the influence of different surface treatments over the bonding efficacy of ceramic brackets to the lithium disilicate ceramic. The results showed that the most efficient treatments were mechanical treatments when followed by chemical conditioning: sandblasting or etching with hydrofluoric acid, followed by conditioning with a primer ie. silanization. Low bond strength was achieved when using mechanical alteration methods only (air abrasion or etching with hydrofluoric acid), without primer conditioning. The lowest bond strength was observed in the control group, meaning that if the ceramic bonding surfaces are ground with a diamond burr only, a sufficient bond strength cannot be expected.

According to Yang et al. [7] air abrasion used as a sole treatment achieved low bond strength. On the other hand, primer conditioning performed after sandblasting provided an adequate bond strength to composite cements. However, when primer was used as the only conditioning method, initially adequate bond strength with the composite cement was achieved, but the bond was proved to be non-water resistant, which resulted in drastically decreased bond strength over time. Hence, the authors concluded that although chemical conditioning methods (primers) are responsible for building the bond with ceramics, such a bond can only be made after previously conducted mechanical conditioning of the bonding surface. In the process of air

abrasion, the ceramic surface roughness increases, it gets cleaned of organic molecules and becomes receptive for a connection with chemical agents [7].

In the literature, the application of hydrofluoric acid when bonding the brackets, is described in combination with silanization (a silane-based primer) as a method of conditioning ceramic surfaces, which results in both micromechanical and chemical retention of orthodontic brackets. Hydrofluoric acid dissolves the glass matrix at the bonding surface of the ceramic restoration, resulting in irregular, and pronounced surface micromorphology. However, due to the high toxicity and corrosive effects of hydrofluoric acid, its use in *in vivo* conditions is limited [8]. Mehmeti et al. concluded that the use of hydrofluoric acid to roughen the surface of zirconium dioxide or lithium disilicate ceramics did not lead to a significantly stronger bond compared to orthophosphoric acid followed by primer application. Furthermore, hydrofluoric acid can weaken the surface structure of ceramics, and considering the corrosive and toxic effect on the oral mucosa, the authors believe that hydrofluoric acid is not the optimal method for conditioning lithium disilicate or zirconium dioxide ceramics [9].

Schmage et al. suggested that bond strength between 6 and 10 MPa of orthodontic brackets to ceramic restorations is sufficient. The conditioning method that provides higher bond strength may lead to damage during the process of debonding; in some cases, if the bond strength exceeds 13 MPa, fracture of the ceramic restoration may be expected [10].

Determining optimal conditioning treatment for lithium disilicate ceramic restorations is largely dependent on the analysis of the fractured surfaces after brackets debonding and determination of the fracture mode. The adhesive type of fracture usually occurs when weaker bond is built between orthodontic brackets and ceramic restorations. Sample analysis with light microscopy showed no residue of the bonding cement on the ceramic bonding surfaces. On the other hand, cohesive or mixed mode of fracture is more prevalent if higher bond strength is achieved and a residue of the bonding cement is detected on the ceramic bonding surface. In some samples, fragments of the fractured ceramic brackets that remained bonded to the ceramic surfaces were observed. In this study, adhesive mode of fractures was exclusively represented in the groups where treatment consisted of mechanical conditioning methods only (sandblasting or etching with hydrofluoric acid), without silanization. Contrary, cohesive or mixed mode of fractures were present in the groups with mechanical and chemical conditioning of the bonding ceramic surfaces (sandblasting or hydrofluoric acid etching followed by silanization) (Table 1).

Conditioning method that provides the strongest bond strength of the brackets to ceramic restorations is not always considered as optimal conditioning method due to the appearance of cohesive fractures after debonding of the orthodontic brackets. Removing the fractured bracket or residual luting cement (which remains adherent to the ceramic surface) may result in irreversible damage of the prosthetic restoration.

## CONCLUSION

Considering the corrosive and toxic effects that hydrofluoric acid has on the oral mucosa, as well as dominantly cohesive and mixed mode of fractures after debonding, the authors suggest that sandblasting with 29- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  followed by silanization may be considered as an optimal conditioning method for lithium disilicate ceramic restorations before bonding the orthodontic ceramic brackets.

## REFERENCES

1. Alzainal HA, Shehab Majud A, Al-Ani MA, Mageet OA. Orthodontic bonding: Review of the literature. *Int J Dent.* 2020;2020:8874909. [DOI: 10.1155/2020/8874909] [PMID: 32733564]
2. Bajraktarova Valjakova E, Korunoska Stevkovska V, Georgieva S, Ivanovski K, Bajraktarova Misevska C, Mijoska A, et al. Hydrofluoric Acid: burns and systemic toxicity, protective measures, immediate and hospital medical treatment. *Open Access Maced J Med Sci.* 2018;6(11):2257–69. [DOI: 10.3889/oamjms.2018.429] [PMID: 30559898]
3. Harvard University DoCaCB. Guidelines for the Safe Use of Hydrofluoric Acid. Cambridge, MA, USA: Harvard University; 2013.
4. Yucel MT, Aykent F, Akman S, Yondem I. Effect of surface treatment methods on the shear bond strength between resin cement and all-ceramic core materials. *J Non-Crystalline Solids.* 2012;358(5):925–30. [DOI: 10.1016/j.jnoncrysol.2012.01.006]
5. Lung CY, Liu D, Matinlinna JP. Silica coating of zirconia by silicon nitride hydrolysis on adhesion promotion of resin to zirconia. *Mater Sci Eng C Mater Biol Appl.* 2015;46: 103–10. [DOI: 10.1016/j.msec.2014.10.029] [PMID: 25491965]
6. Xie H, Li Q, Zhang F, Lu Y, Tay FR, Qian M, et al. Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better. *Dent Mater.* 2016;32(3):403–11. [DOI: 10.1016/j.dental.2015.12.014] [PMID: 26754430]
7. Yang B, Barloi A, Kern M. Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin. *Dent Materials.* 2010;26(1):44–50. [DOI: 10.1016/j.dental.2009.08.008] [PMID: 19766300]
8. Wolf DM, Powers JM, O'Keefe KL. Bond strength of composite to etched and sandblasted porcelain. *Am J Dent.* 1993;6(3):155–8. [PMID: 8240779]
9. Mehmeti B, Kelmendi J, Ilijazi D, Bleron Azizi S, Jakovljević S, Haliti F, et al. Comparison of Shear Bond Strength Orthodontic Brackets Bonded to Zirconia and Lithium Disilicate Crowns. *Acta Stomatol Croat.* 2019;53(1):17–27. [DOI: 10.15644/asc53/1/2] [PMID: 31118529]
10. Schmage P, Nergiz NV, Herrmann W, Ozcan M, Nergiz I. Influence of various surface-conditioning methods on the bond strength of metal brackets to ceramic surfaces. *Am J Orthod Dentofacial Orthop.* 2003;123(5):540–6. [DOI: 10.1067/mod.2003.S0889540602569110] [PMID: 12750673]

---

Received: 25.9.2023 • Accepted: 11.12.2023

# Bondiranje ortodontskih keramičkih breketa: optimalna metoda kondicioniranja litijum-disilikatnih restauracija

Stefan Kuzmanovski<sup>1</sup>, Cvetanka Bajraktarova Miševska<sup>2</sup>, Aneta Mijoska<sup>1</sup>, Nataša Stavreva<sup>1</sup>, Emilija Bajraktarova Valjakova<sup>1</sup>

<sup>1</sup>Univerzitet „Sv. Kiril i Metodij“, Stomatološki fakultet, Katedra stomatološke protetike, Skoplje, Republika Severna Makedonija;

<sup>2</sup>Univerzitet „Sv. Kiril i Metodij“, Stomatološki fakultet, Katedra ortodontije, Skoplje, Republika Severna Makedonija

## KRATAK SADRŽAJ

**Uvod** Prema podacima dobijenim iz istraživanja Američkog ortodontskog društva sprovedenog 2015. godine, broj odraslih pacijenata koji su zatražili ortodontski tretman povećan je sa 14% na 27% u periodu od 2010. do 2014. godine. Zbog povećanja starosti pacijenta očekuje se izvođenje fiksnog ortodontskog tretmana na restauriranim zubima, uključujući i bezmetalne keramičke nadoknade. Ciljevi ovog istraživanja bili su da se evaluira uticaj različitih površinskih tretmana na čvrstoću veze keramičkih breketa sa litijum-disilikatnom keramikom i da se utvrdi tip frakture nakon debondiranja breketa.

**Materijal i metod** Dobijeno je 45 keramičkih uzoraka iz litijum-disilikatnih CAD/CAM blokova koji su prema izvršenoj metodi kondicioniranja podeljeni u pet grupa: 1. brušenje finim dijamantskim borerom (kontrolna grupa); 2. peskarenje sa 29 µm aluminijum-oksida ( $Al_2O_3$ ); 3. jetkanje sa četvoroprocentnom fluorovodoničnom kiselinom (HF); 4. peskarenje praćeno kondicioniranjem univerzalnim prajmerom – silanizacija ( $Al_2O_3 + S$ ); 5. jetkanje HF kiselinom praćeno silanizacijom (HF + S). Ispitivanje čvrstoće bondiranja je izvršeno nakon čuvanja uzoraka u vodenom kupatilu sedam dana. Svi slomljeni uzorci su analizirani optičkim mikroskopom da bi se odredio tip preloma.

**Rezultati** Jetkanje HF kiselinom praćeno silanizacijom ima najveću snagu bondiranja, od 8,03 MPa, dok peskarenje praćeno silanizacijom ima 6,69 MPa. Mehaničko kondicioniranje površine bilo HF kiselinom ili peskarenjem rezultiralo je značajno nižom čvrstoćom bondiranja (2,65 MPa i 1,51 MPa). Nakon peskarenja i silanizacije uočen je uglavnom adhezivni tip preloma, što ukazuje na manju mogućnost oštećenja keramičke nadoknade nakon debondiranja, za razliku od keramičkih uzoraka u Grupi 5 (HF + S) sa 42,8% mešovitih i 14,4% kohezivnih preloma. Uočeni su 100% adhezivni prelomi nakon mehaničkih tretmana.

**Zaključak** Prema rezultatima SBS testa i vrsti preloma, peskarenje sa nanošenjem univerzalnog prajmera može se smatrati adekvatnim metodom za kondicioniranje litijum-disilikatnih keramičkih nadoknada pre bondiranja keramičkih ortodontskih breketa.

**Cljučne reči:** keramički breketi; metode kondicioniranja; litijum-disilikatne restauracije; vazдушna abrazija; fluorovodonična kiselina; univerzalni prajmer

## UVOD

Prema rezultatima dobijenim anketom Američkog ortodontskog društva sprovedenom 2015. godine, broj odraslih pacijenata koji su zatražili ortodontski tretman povećan je sa 14% na 27% između 2010. i 2014. godine, što znači da se broj odraslih ortodontskih pacijenata udvostručio za samo četiri godine [1]. Zbog toga se u svakodnevnoj kliničkoj praksi može očekivati da se ortodontski tretman ortodontskim breketama izvodi na zubima koji su prethodno tretirani fiksno-protetskim restauracijama – metal-keramičkim ili potpuno keramičkim krunama ili faseta. S druge strane, debondiranje breketa je čest problem, zbog čega se pregledi kod ortodonta češće zakazuju i povezuju se sa ponovnim sprovođenjem celokupne procedure za rebondiranje breketa. Od primarne važnosti za uspešan ortodontski tretman je postizanje adekvatnog vezivanja breketa za protetske nadoknade. Pri tome, čvrstoća veze ne bi trebalo da bude ni previše jaka kako bi se izbeglo oštećenje keramičkih površina tokom procesa debondiranja breketa na kraju ortodontskog tretmana [1].

Uvođenje staklokeramike kao restaurativnog materijala u stomatološku protetiku, kao i dokazane prednosti adhezivnog cementiranja doprineli su razvoju koncepta jetkanja fluorovodoničnom kiselinom (HFA). Međutim, HFA poseduje korozivni potencijal koji izaziva opekotine i nekrozu tkiva na mestu kontakta, a ako se apsorbuje u krvotok, joni fluora se distribuiraju u sve organe i tkiva, izazivajući moguću sistemsku toksičnost opasnu po život i otkazivanje organa. Sve ovo nameće izuzetan oprez i svest o zdravstvenim posledicama kada se HF kiselina primenjuje intraoralno [2]: izlaganje oralne sluzokože u

koncentraciji od 0,1% HFA može dovesti do nekroze sluzokože [3]. Prilikom jetkanja staklokeramike sa 9,6% HFA dolazi do dvostruke reakcije: primarna – između kiseline i staklene faze, i sekundarna – između kiseline i kristalne faze, ostavljajući veće kristale intaktnima. Na ovaj način se stvara nepravilna površina sa mikroskopskim porama koje omogućavaju mikromehaničko retiniranje adhezivne smole. Što je više zastupljena kristalna faza a manje staklena faza u sastavu materijala, veća je otpornost keramike na dejstvo kiseline [4].

Vazдушna abrazija aluminijum-oksida ( $Al_2O_3$ ) još je jedna metoda za mehaničko kondicioniranje bondiranih površina keramičkih restauracija koja uzrokuje iregularnost površine, koja je neophodna za mikromehaničko retiniranje vezivnog sredstva. Sa druge strane, vazдушna abrazija može izazvati ireverzibilno oštećenje keramičkih restauracija. Zbog toga se preporučuje da se peskarenje izvodi pod niskim pritiskom (1-2 bara), korišćenjem zrna aluminijum-oksida veličine manje od 50 µm i na udaljenosti od 10 mm od površine keramike [5].

Nakon mehaničke pripreme keramičke bondirane površine, a da bi se postigla optimalna veza sa kompozitnim cementom, od suštinskog je značaja hemijsko kondicioniranje prajmerima koji sadrže silan u svom sastavu. Molekul silana ima dve različite funkcionalne grupe: -SiOH grupu koja se vezuje za molekule silicijum-dioksida i drugu organsku funkcionalnu grupu koja se vezuje za metakrilatnu smolu kompozitnog cementa [6].

Cilj ove studije bio je da se evaluira jačina bondiranja keramičkih breketa vezanih za litijum-disilikatne keramike nakon različitih metoda kondicioniranja keramičke površine i određivanje tipa preloma nakon debondiranja breketa.

## MATERIJAL I METOD

Keramički uzorci (debljine 2 mm) dobijeni su od presinterovanih litijum-disilikatnih CAD/CAM blokova IPS e.max CAD (Ivoclar Vivadent, Schaan Liechtenstein) (Slika 1a) korišćenjem precizne mašine za sečenje Minitom (Struers, Danska) (Slika 1b). Sečenje je izvedeno uz kontinuirano vodeno hlađenje kako bi se sprečilo pregrevanje keramičkog materijala koje može izazvati mikropukotine. Svi keramički uzorci su sinterovani u keramičkoj peći Programat EP 5000 (Ivoclar Vivadent, Schaan Liechtenstein). Uzorci sinterovane keramike bili su potopljeni u sredinu metalnih prstenastih kalupa ( $d = 30$  mm) ispunjenih hladno-polimerizujućim akrilatom – PoliTEMP (PoliDent, Slovenija), sa ekspaniranom keramičkom površinom koja će biti korišćena kao površina za vezivanje keramičkih breketa (Slika 1v). Nakon toga, površina za vezivanje svih keramičkih uzoraka je izbrušena finim dijamantskim borerom. Svi keramički uzorci su podeljeni u pet grupa prema površinskom tretmanu:

1. Kontrolna grupa = direktno bondiranje ortodontskih breketa.

2.  $Al_2O_3$ : keramički uzorci su peskareni sa  $29\text{-}\mu\text{m}$   $Al_2O_3$  česticama – Sandman (Innovative Micro Dentistry, Poland), u trajanju od 10 sekundi, pod pritiskom od jednog bara i na udaljenosti od 10 mm (Slika 1g).

3. HF: keramičke površine su nagrizane četvoroprocentnom fluorovodoničnom kiselinom – IPS Ceramic etching gel (Ivoclar Vivadent, Schaan Liechtenstein) u trajanju od 20 sekundi. Površine su temeljno ispirane tokom 60 s sa ciljem da se ukloni zaostala kiselina, a nakon toga osušene komprimovanim vazduhom (Slika 2a).

4.  $Al_2O_3 + S$ : keramički uzorci su peskareni sa  $29\text{-}\mu\text{m}$   $Al_2O_3$  česticama postupkom peskarenja kao u Grupi 2. Nakon toga je u tankom sloju nanet univerzalni prajmer – Monobond Plus (Ivoclar Vivadent, Schaan Liechtenstein) i ostavljen da deluje 60 sekundi, nakon čega je sledilo bondiranje breketa (Slika 2b).

5. HF + S: bondirane površine su nagrizane četvoroprocentnom fluorovodoničnom kiselinom tokom 20 s, isprane i osušene. Univerzalni prajmer Monobond Plus je nanet i ostavljen da deluje 60 sekundi nakon, čega je sledilo bondiranje breketa (Slika 2v).

### Postupak bondiranja

Nakon tretmana bondiranih površina, keramičke brekete za maksilarne centralne sekutiće – Cosmetic 20/40 UR Central (American Orthodontics, USA) zalepljene su na tretiranu površinu korišćenjem ortodontskog kompozitnog cementa No Mix:30™ One step Adhesive (American Orthodontics, USA) sa konstantnim vertikalnim opterećenjem od 1 kg u trajanju od 1 min. Nakon procesa bondiranja, uzorci su čuvani u vodenom kupatilu na  $37^\circ\text{C}$  sedam dana – Biobase Water Tank WT-42 (Biobase Biodustry, Shandong, China) imitirajući uslove u usnoj šupljini (vlaga i temperatura).

Ispitivanje čvrstoće veze na smicanje (shear bond strenght test – SBS)

SBS je izveden na univerzalnoj mašini za ispitivanje – Shimadzu AGS-X (Shimadzu Co., Japan), brzinom od  $0,5$  mm/min., u cilju određivanja čvrstoće veze između ortodontskih breketa i keramičkih površina (Slika 3). SBS je izražen u MPa,

izveden deljenjem sile (N) u trenutku preloma sa vezivnom površinom keramičkog breketa ( $\text{mm}^2$ ) ( $\text{MPa} = \text{N}/\text{mm}^2$ ).

### Tip preloma

Tip preloma (adhezivni, kohezivni u nivou cementa ili breketa ili mešoviti) za svaki uzorak određen je pomoću svetlosne mikroskopije – Levenhuk Zeno Cash ZC6 (Levenhuk Inc., USA) pri uvećanju od 60 puta.

### Statistička analiza

Analiza kategoričkih varijabli urađena je utvrđivanjem koeficijenta odnosa, proporcija i stopa. Kontinuirane varijable su analizirane merama centralne tendencije (srednja, medijana, minimalne i maksimalne vrednosti), kao i merama disperzije (standardna devijacija). Za određivanje normalnosti frekvencijske distribucije proučavanih varijabli korišćen je Šapiro–Vilkov test. Pirsonov  $\chi^2$  test je korišćen da se utvrdi povezanost između određenih atributivnih dihotomnih karakteristika.

### REZULTATI

Rezultati SBS-a nakon različitih tretmana keramičkih površina prikazani su na Slici 4 i u Tabeli 1. Najveća čvrstoća veze,  $8,04 \pm 0,61$  MPa, zabeležena je kada su keramičke brekete bondirane na keramičke površine koje su nagrizane fluorovodoničnom kiselinom, a zatim je usledila silanizacija (Grupa 5). Nesignifikantno niža čvrstoća veze ( $p = 0,1477$ ) postignuta je peskarenjem bondiranih površina sa  $29\text{-}\mu\text{m}$   $Al_2O_3$ , praćenom silanizacijom,  $6,69 \pm 1,20$  MPa. Kada su vršeni samo mehanički tretmani, jetkanje HF kiselinom ili peskarenje, jačina veze je bila značajno niža,  $2,65 \pm 0,84$  MPa i  $1,52 \pm 0,43$  MPa. Najniža vrednost SBS-a je zabeležena u kontrolnoj grupi,  $0,43 \pm 0,23$  MPa.

Adhezivni tip preloma nakon debondiranja bio je isključivo prisutan u prve tri grupe (100%), dok je u Grupi 4 bio dominantno prisutan (71,4%). Mešoviti i kohezivni tip preloma konstatovani su samo u Grupi 4 i Grupi 5 (Tabela 1).

### DISKUSIJA

Cilj ove studije bio je da se evaluiira uticaj različitih površinskih tretmana na efikasnost vezivanja keramičkih breketa za litijum-disilikatnu keramiku. Iz dobijenih rezultata može se uočiti da je visoka jačina bondirane sile postignuta u grupama u kojima su sprovedene mehaničko-hemijske metode za alteraciju keramičkih bondiranih površina (vazдушna abrazija ili jetkanje fluorovodoničnom kiselinom, u kombinaciji sa nanošenjem prajmera). Manja jačina bondirane sile je postignuta kod uzoraka koji su podvrgnuti samo metodama mehaničke alteracije (vazдушna abrazija ili jetkanje fluorovodoničnom kiselinom), bez kondicioniranja prajmerom. Najniže srednje vrednosti za jačinu bondirane sile zabeležene su u kontrolnoj grupi, što znači da ako se keramičke bondirane površine ne alteriraju i ne kondicioniraju, onda se ne može očekivati dovoljna jačina bondirane sile.

Autori Yang i saradnici [7] smatraju da vazдушna abrazija bez upotrebe prajmera postiže značajno nisku jačinu bondirane sile. Sa druge strane, vazдушna abrazija keramike praćena kondicioniranjem prajmerom obezbeđuje adekvatnu jačinu bondirane sile za kompozitne cemente. Autori su u istom istraživanju dokazali da kad su prajmeri korišćeni kao jedinstvena metoda kondicioniranja keramike u početku, dobijena je adekvatna jačina bondiranja sa kompozitnim cementom, ali se pokazalo da ona nije vodootporna, što je rezultiralo drastičnom smanjenju jačine bondirane sile tokom vremena. Stoga, autori su zaključili da iako su hemijske metode kondicioniranja (prajmeri) odgovorne za stvaranje veze sa keramikom, takva veza se može ostvariti samo nakon prethodnog mehaničkog kondicioniranja keramičke površine. U procesu vazdušne abrazije povećava se hrapavost površine, ona se čisti od organskih molekula i postaje prijemčiva za stvaranje veze sa hemijskim agensima [7].

U literaturi je opisana primena fluorovodonične kiseline u ortodontske svrhe u kombinaciji sa silanizacijom (prajmer na bazi silana) kao metodom kondicioniranja keramičkih površina, što rezultira i mikromehaničkim i hemijskim retiniranjem ortodontskih breketa. Fluorovodonična kiselina rastvara staklenu matricu keramičke restauracije, zbog čega tretirana površina postaje iregularna, sa izraženom mikromorfologijom. Istovremeno, zbog visoke toksičnosti i korozivnog dejstva fluorovodonične kiseline u kontaktu sa oralnim tkivom i mogućnosti brze nekroze, njena upotreba u *in vivo* uslovima je ograničena [8]. Mehmeti i saradnici zaključili su da upotreba fluorovodonične kiseline u cilju jetkanja površine cirkonijum-dioksida ili litijum-disilikatne keramike nije dovela do značajno jače veze u poređenju sa ortofosfornom kiselinom, nakon čega je usledila primena prajmera. Nadalje, fluorovodonična kiselina može oslabiti površinsku strukturu keramike, a s obzirom na korozivno i toksično dejstvo na oralnu sluzokožu, autori smatraju da fluorovodonična kiselina nije najbolja metoda za kondicioniranje keramike litijum-disilikata i cirkonijum-dioksida [9].

Schmage i saradnici sugerišu da je jačina bondirane sile od 6 do 10 MPa dovoljna da obezbedi adekvatnu vezu između ortodontskih breketa i keramičkih restauracija. Metoda kondicioniranja keramičke površine koja istovremeno obezbeđuje

najjaču bondiranu silu može dovesti do njenog oštećenja tokom debondiranja; ako je jačina bondirane sile veća od 13 MPa, može se očekivati fraktura keramičke površine [10].

Određivanje optimalnog tretmana kondicioniranja za litijum-disilikatne keramičke restauracije u velikoj meri zavisi od određivanja načina preloma. Adhezivni tip preloma se obično javlja kada se između ortodontskih breketa i keramičkih nadoknada izgradi slabija veza. Analiza uzoraka svetlosnom mikroskopijom nije pokazala ostatke vezivnog cementa na keramičkim vezivnim površinama. S druge strane, kohezivni ili mešoviti način loma je preovlađujući ako se postigne veća čvrstoća veze i ako se otkrije ostatak vezivnog cementa na keramičkoj površini. U nekim uzorcima uočeni su fragmenti frakturisanih keramičkih breketa koji su ostali vezani za keramičke površine. U ovoj studiji, adhezivni način preloma bio je isključivo zastupljen u grupama u kojima se tretman sastojao samo od mehaničkih metoda kondicioniranja (peskarenje ili nagrizanje fluorovodoničnom kiselinom), bez silanizacije. Suprotno tome, kohezivni ili mešoviti načini preloma bili su prisutni u grupama sa mehaničkim i hemijskim kondicioniranjem vezivnih keramičkih površina (peskarenje ili jetkanje fluorovodoničnom kiselinom, praćeno silanizacijom) (Tabela 1).

Metoda kondicioniranja koja obezbeđuje najjaču čvrstoću bondiranja breketa za keramičke nadoknade ne smatra se uvek optimalnom metodom kondicioniranja, zbog pojave kohezivnih preloma nakon debondiranja ortodontskih breketa. Uklanjanje slomljenog breketa ili zaostalog cementa (koji ostaje na keramičkoj površini) može dovesti do nepovratnog oštećenja protetske nadoknade.

## ZAKLJUČAK

Uzimajući u obzir korozivne i toksične efekte koje fluorovodonična kiselina ima na oralnu sluzokožu, kao i dominantno kohezivni i mešoviti tip preloma nakon debondiranja, autori sugerišu da se peskarenje sa 29- $\mu\text{m}$   $\text{Al}_2\text{O}_3$ , praćeno silanizacijom, može smatrati optimalnom metodom kondicioniranja litijum-disilikatnih keramičkih restauracija pre bondiranja ortodontske keramičke brekete.