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ORIGINAL SCIENTIFIC PAPER
IZVORNI ZNANSTVENI RAD

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Comparison of Three Different Orthodontic Adhesives Bonded to Metallic and Ceramic Brackets: SEM and SEM/EDX Analysis (*In Vitro* Study)

Usporedba triju različitih ortodontskih adheziva za pričvršćivanje metalnih i keramičkih bravica: SEM i SEM/EDX analiza (istraživanje in vitro*)*

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Abstract

Objectives: To compare three different orthodontic adhesives (Transbond XT Light Cure Adhesive, Heliosit Orthodontic, Fuji Ortho LC) bonded to two types of orthodontic brackets: ceramic brackets (Fascination Roth 0.22) and metallic brackets (Topic Roth 0.22, Dentaurum). **Materials and methods:** The study was performed on 18 human teeth (6 for each adhesive). The prepared teeth were divided into three groups according to the examination time. Subsequently, they were observed after 1, 2 and 3 weeks following bonding. After the experimental procedure, the teeth samples were cut in half along the longitudinal axis in the vestibulo-oral direction, fixed with conductive carbon cement, placed in a high-vacuum evaporator and then coated with carbon. One half of each sample was observed under a Field-emission gun scanning electron microscope (FEG-SEM Hitachi SU 8030, Japan), while on the second half of the samples qualitative (X-ray line-scans) and semi-quantitative point X-ray energy dispersive analyses (EDX) were performed with Thermo Noran (USA) NSS System 7, equipped with Ultra Dry detector (30 mm^2 window). **Results:** Transbond XT had an ideal bond with the enamel and the bracket base, with rare presence of microgaps and cracks in the enamel. Heliosit Orthodontic demonstrated a better bond relationship with the bracket base than the enamel, whereas in the latter the presence of microgaps in the bond was observed. The microphotographs of Fuji Ortho LC demonstrated many cracks inside the adhesive, and some of them continued to move forward into the enamel surface. Therefore, an impression of a very solid bond relationship with the enamel exists, with cracks being present in the enamel surface and never at the enamel-adhesive interface. Microgaps also appeared at the bracket-adhesive interface. **Conclusion**: Transbond XT is a highly filled composite resin and is an ideal orthodontic adhesive in each aspect examined, with an ideal enamel-adhesive and bracket-adhesive interface. Heliosit Orthodontic provides better bracket-adhesive interface compared to the enamel. Fuji Ortho LC as a solid resin-modified GIC provides a better enamel-adhesive interface, compared to the bracket base.

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Introduction

Direct bonding of orthodontic brackets has opened a new chapter in orthodontics, with manufacturers of dental materials under daily pressure to find dental adhesives with

Uvod

Direktno povezivanje ortodontskih bravica otvorilo je novo poglavlje u ortodonciji, pri čemu su proizvođači dentalnih materijala svakodnevno pod pritiskom da pronađu adheoptimal properties. Also, recently, many adult patients have demanded orthodontic treatment and superior esthetics, therefore, along with the conventional metallic brackets, ceramic brackets were introduced (1).

Apart from esthetics, the critical question regarding the metallic brackets is whether the connection will be too weak to withstand the forces of orthodontic treatment. On the contrary, when ceramic brackets are employed, the concern is whether the connection will be too strong for safe removal of the brackets after the completion of orthodontic therapy.

Current ceramic brackets are made of monocrystalline or polycrystalline aluminum oxide (2). Additionally, the base of the ceramic brackets is silanized, hence they form a very strong chemical bond with the adhesive, which poses a problem during their removal. The brittle, rigid nature of ceramic brackets and the enamel itself leads to poor stress absorption during debonding of the bracket, and, since ceramic brackets do not bend during debonding, fractures can occur either on the ceramic bracket, but also at the resin/enamel interface, often causing cracks to appear on the very surface of the enamel (3). If the bond between the adhesive and the enamel is stronger than the enamel itself, then the enamel will fracture when the bracket is debonded (4, 5).

The adhesives used for bonding of the brackets, are based on composite resins or glass ionomer cements (GIC). The composite resins-based, due to their higher strength, are used more often. However, today, with the modification of the GICs, their mechanical properties are also improved, whereby they meet the conditions for successful retention in orthodontic therapy. Additionally, GIC adhesives are known to release fluoride ions that migrate into the tooth and contribute to remineralization of the tooth structure. (5-7). Unlike conventional composite resins, GICs have the properties of being physically and chemically bonded to enamel and with dentin, through the affinity of calcium in the tooth structure with the carboxylate group in cement, but at the same time they bind to metals and plastics. They can be bonded to the enamel without the need for acid etching (4).

To improve the shortcomings of the conventional GICs (low bond strength compared o composite resins, high bracket de-bonding rate, poor mechanical properties in the early stages after bonding and susceptibility to moisture during the initial reaction) (8), and in order to improve the bond strength, hybrid materials known as resin-modified GICs - a combination of the properties of GIC and composites, have been introduced. These materials release fluoride ions in the same way as the conventional GICs. Yet they possess improved physical and mechanical properties, as well as the ability to set rapidly by light polymerization (4, 5).

Upon application of orthodontic adhesives (composite resin or resin-modified GICs), etching with 37% orthophosphoric acid is the most commonly used conditioning method (9). Several authors claim that the orthophosphoric acid etching results in loss of about 10-30 µm from the enamel, but the adhesive can penetrate up to 50 µm into the enamel. After removing the bracket and cleaning the adhesive, between 50-55.6 µm of enamel is lost (10).The purpose of this study was to compare the interface between different types of

ziv s optimalnim svojstvima. Također, danas mnogi odrasli pacijenti traže uz ortodontski tretman i superiornu estetiku, stoga su, uz konvencionalne metalne bravice, uvedene i keramičke (1).

Osim estetike, ključno pitanje u vezi s metalnim bravicama jest hoće li veza biti preslaba da izdrži sile ortodontskog liječenja. S druge strane, kada se koriste keramičke bravice, zabrinjava hoće li veza biti prejaka za sigurno uklanjanje bravica poslije završetka ortodontskog tretmana.

Sadašnje keramičke bravice izrađene su od monokristalnog ili polikristalnog aluminijeva oksida (2). Uz to, njihova je baza silanizirana i tvori vrlo snažnu kemijsku vezu s adhezivom, što je problem tijekom njihova uklanjanja. Krhka, čvrsta priroda keramičkih bravica i same cakline dovodi do loše apsorpcije stresa tijekom odvajanja bravice, zato što se one ne savijaju pri odvajanju, pa se može dogoditi puknuće kako na keramičkoj bravici, tako i na samom spoju smole/cakline, često uzrokujući pukotine na površini cakline (3). Ako je veza između adheziva i cakline jača od cakline, tada će pri odvajanju bravice caklina puknuti (4, 5).

Adhezivi koji se koriste za lijepljenje bravica temelje se na kompozitnim smolama ili stakleno-ionomernim cementima (SIC). Kompozitne smole češće se koriste zbog veće čvrstoće, ali danas, s modifikacijom SIC-ova, poboljšana su i njihova mehanička svojstva, što zadovoljava uvjete za uspješnu retenciju u ortodontskoj terapiji. Osim toga, SIC adhezivi poznati su po oslobađanju fluoridnih iona koji migriraju u zub i pridonose remineralizaciji strukture zuba (5 – 7). Za razliku od konvencionalnih kompozitnih smola, SIC-ovi imaju svojstva da su fizički i kemijski povezani s caklinom i dentinom putem afiniteta povezivanja kalcija u strukturi zuba s karboksilnom skupinom u cementu, ali istodobno se vežu i za metal i plastiku. Mogu se povezati s caklinom bez potrebe za jetkanjem kiselinom (4).

Kako bi se poboljšali nedostatci konvencionalnih SIC-ova (niska čvrstoća veza u usporedbi s kompozitnim smolama, visoka stopa odvajanja bravica, loša mehanička svojstva u ranim fazama poslije povezivanja i osjetljivost na vlagu tijekom početne reakcije) (8) i kako bi se poboljšala čvrstoća veze, uvedeni su hibridni materijali poznati kao smolom modificirani SIC-ovi – kombinacija svojstava SIC-a i kompozita. Ti materijali oslobađaju fluoridne ione kao i konvencionalni SIC-ovi, ali poboljšana su im fizička i mehanička svojstva i brzo se stvrdnjavaju svjetlosnom polimerizacijom (4, 5).

Pri primjeni ortodontskih adheziva (kompozitna smola ili smolom modificirani SIC-ovi), najčešće korištena metoda kondicioniranja jest korištenje 37-postotne ortofosforne kiseline (9). Pojedini autori tvrde da jetkanje ortofosfornom kiselinom rezultira gubitkom cakline od 10 do 30 µm, ali da adheziv može prodrijeti do 50 µm u caklinu. Nakon uklanjanja bravice i čišćenja adheziva, izgubljeno je između 50 i 55,6 µm cakline (10). Cilj ovog istraživanja bio je usporediti vezu između različitih vrsta ortodontskih adheziva i cakline, odrediti kvalitetu povezivanja različitih vrsta bravica s ortodontskim adhezivima i, konačno, ocijeniti elementarnu kompoziciju adheziva i razinu inkorporacije iona iz adheziva u strukturu cakline.

orthodontic adhesives and the enamel; to determine the quality of bonding of different types of brackets with orthodontic adhesives; and, finally, to assess the elemental composition of the adhesives and the level of ion incorporation from the adhesives in the enamel structure.

Material and methods

A total of 18 human teeth extracted for orthodontic reasons were used in the study, six per each group, bonded with three different types of light-polymerizing orthodontic adhesives: Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, USA), Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Liechtenstein), Fuji Ortho LC (GC Corporation, Japan) with two different types of brackets: Dentaurum Topic Roth 0.22 and Dentaurum Fascination Roth 0.22 (both Dentaurum. Langhorne, United States).

2.1. Preparation of samples

The extracted teeth, each with intact labial surface, were stored in physiological saline and used within 1 month after extraction. The teeth were cut 1-2 mm above the cemento-enamel junction.

Before bonding of the brackets, the teeth were cleaned by pumice and polishing paste. Prior to bonding and following bonding, the teeth were kept in saline to maintain a moist environment, thus preventing their dehydration.

The teeth bonded with Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Liechtenstein) and Fuji Ortho LC (GC Corporation, Japan) were conditioned with 37% orthophosphoric acid, while the teeth bonded with Transbond XT Light Cure adhesive (3M Unitek Orthodontic Products, USA) were conditioned with Transbond Plus Self-Etching Primer (3M Unitek Orthodontic Products, USA).

The prepared teeth were divided into three groups according to the examination time and were observed after 1, 2 and 3 weeks following bonding.

2.2. SEM (Scanning Electron Microscopy)

After the experimental procedure, the teeth samples were cut in half along the longitudinal axis in the vestibulo-oral direction with a separator (Superflex 605.524.220, NTI-KAHLA GmbH, Germany).

The cut samples were placed in an incubator at 37°C for 12 hours. The samples were fixed with conductive carbon cement (Leit-C conductive carbon cement, Neubauer Chemikalien) in special holders. Subsequently, the samples were placed in a high-vacuum evaporator and coated with carbon (Model S105, Edwards Co., UK). The process took place in 2 phases: the first phase lasted 5 min at 10^{-5} Torr, while the duration of the second phase was 60 min.

One half of each sample was observed under a Field-emission gun scanning electron microscope (FEG-SEM Hitachi SU 8030, Japan).

2.3. SEM / EDX (Scanning Electron Microscopy / Energy Dispersive Analysis with X-rays)

The second half of the samples was carbon-coated (Model S105, Edwards Co., UK). X-ray energy analysis (EDX)

Materijali i metode

U istraživanju je korišteno ukupno 18 humanih zuba ekstrahiranih iz ortodontskih razloga – po šest zuba za svaku skupinu – spojenih trima različitim vrstama svjetlosnopolimerizirajućih ortodontskih adheziva: Transbond XT Light Cure Adhesiveom (3M Unitek Orthodontic Products, SAD), Heliosit Orthodonticom (Ivoclar Vivadent, Schaan, Lihtenštajn), Fuji Ortho LC-om (GC Corporation, Japan) s dvama različitim tipovima bravica: Dentaurum Topic Roth 0.22 i Dentaurum Fascination Roth 0.22 (oboje Dentaurum, Langhorne, SAD).

2.1. Priprema uzoraka

Ekstrahirani zubi, svaki s netaknutom labijalnom površinom, pohranjeni su u fiziološkoj otopini i korišteni unutar jednog mjeseca poslije vađenja. Zubi su rezani 1 do 2 mm iznad caklinsko-dentinskoga spoja.

Prije lijepljenja bravica zubi su očišćeni polirnom gumicom i pastom za poliranje. Prije i poslije postupka lijepljenja zubi su držani u fiziološkoj otopini da bi se održala vlažna sredina i spriječila dehidracija.

Zubi lijepljeni Heliosit Orthodonticom (Ivoclar Vivadent, Schaan, Lihtenštajn) i Fuji Ortho LC-om (GC Corporation, Japan) bili su kondicionirani 37-postotnom ortofosfornom kiselinom, a zubi spojeni adhezivom Transbond XT Light Cure (3M Unitek Orthodontic Products, SAD) bili su kondicionirani Transbond Plus Self-Etching Primerom (3M Unitek Orthodontic Products, SAD).

Pripremljeni zubi podijeljeni su u tri skupine prema vremenu ispitivanja i promatrani su jedan, dva i tri tjedna poslije lijepljenja.

2.2. SEM (skenirajući elektronski mikroskop)

Nakon eksperimentalnog postupka uzorci zuba prerezani su na pola uzdužno u vestibulo-oralnom smjeru separatorom (Superflex 605.524.220, NTI-KAHLA GmbH, Njemačka).

Prerezani uzorci stavljeni su u inkubator na 37 °C tijekom 12 sati. Zatim su fiksirani provodljivim karbonskim cementom (Leit-C provodljivi karbonski cement, Neubauer Chemikalien, Njemačka) u posebnim držačima. Nakon toga stavljeni su u visokovakuumski evaporator i premazivani slojem ugljika (Model S105, Edwards Co., UK). Proces je imao dvije faze: prva je trajala 5 minuta pri 10^{-5} torra, a druga 60 minuta.

Jedna polovina svakog uzorka promatrana je s pomoću pištolja za emisiju polja skenirajućega elektronskog mikroskopa (FEG-SEM Hitachi SU 8030, Japan).

2.3. SEM / EDX (skenirajući elektronski mikroskop/ energijska disperzivna analiza rendgenskih zraka)

Druga polovina uzoraka bila je premazana slojem ugljika (Model S105, Edwards Co., UK). Energijska disperzivna was performed with Thermo Noran (USA) NSS System 7, equipped with Ultra Dry detector (30 mm^2 window).

Qualitative X-ray energy analysis (EDX) was performed by collecting X-ray line-scans along the line which goes from the bracket, through the adhesive interface, into the enamel in order to determine the elemental distribution of the surface enamel.

Finally, a semi-quantitative EDX point analysis was performed on the enamel surface in order to determine the elemental level (%) of carbon (C), hydrogen (O), sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), fluorine (F), phosphates (P) and calcium (Ca). For each sample, 10 points adjacent to the tooth surface were randomly selected, plus 3 additional points randomly selected away from the surface, and mean values were calculated.

2.4. Statistical analysis

Statistical analysis was performed in statistical programs: STATISTICA 7.1; SPSS 20.0. Processing was performed using standard descriptive and analytical bivariate and multivariate methods.

Numerical series were analyzed by central tendency measures and data dispersion measures. In the numerical series, where there is no deviation from the normal distribution, the significance of the difference was tested with the Student t - test. The statistical significance of the differences between more than three numerical variables was analyzed using ANOVA test, and in case of significant differences, the post-hoc Tukey's test was applied. For CI (confidence interval 95% -CI), the statistical significance for an error level of less than 0.05 (p) was defined.

Results

Figure 1 represents microphotographs of samples tested 1 week after bonding. The microphotographs (Figure 1a, Figure 1b) show the presence of micro-spaces between the Transbond XT and the enamel, and several micro-cracks can be noticed inside the enamel. Excellent interface between the adhesive and the bracket is visible. Figure 1c shows a clear crack inside the adhesive Heliosit Orthodontic itself, which runs tangentially along the outer part of the retention net of the metallic bracket, while the part of the adhesive located on the inner net is well attached to the bracket. The bond between the adhesive and the enamel is not ideal, and cracks at the interface between the adhesive and the enamel, and occasionally inside the enamel, are present. Figure 1d shows the cracks between the adhesive and the enamel, while the yellow arrow shows a fracture at the enamel-dentin junction which probably occurred during preparation of the samples. Figure 3e shows numerous cracks inside the Fuji Ortho LC adhesive some of which extend in the direction of the enamel, while microfractures can be seen on the very surface of the enamel. Fractures in the adhesive itself that continue to move forward inside the enamel are shown in Figure 1f.

In Figure 2a and Figure 2b, the connection of the Transbond XT adhesive with the enamel and the bracket on the other side looks ideal without the appearance of any microspaces. Figure 2b shows a strong and ideal connection be-

analiza rendgenskih zraka (EDX) obavljena je Thermo Noran (SAD) NSS Systemom 7, opremljenim detektorom Ultra Dry (prozor od 30 mm^2).

Kvalitativna energijska disperzivna analiza rendgenskih zraka (EDX) obavljena je prikupljanjem rendgenskih linijskih skeniranja duž linije koja prolazi od bravice kroz površinu adheziva do cakline kako bi se odredila elementarna distribucija površine cakline.

Konačno, provedena je polukvantitativna analiza EDX točaka na površini cakline da bi se odredila razina elementa (%) kao što su ugljik (C), vodik (O), natrij (Na), magnezij (Mg), aluminij (Al), silicij (Si), fluor (F), fosfati (P) i kalcij (Ca). Za svaki uzorak odabrano je 10 točaka uz rub zuba, plus 3 dodatne nasumično odabrane točke udaljene od površine, te su izračunate srednje vrijednosti.

2.4. Statistička analiza

Statistička analiza provedena je u programu STATISTICA 7.1; SPSS 20.0. Obrada je obavljena standardnim deskriptivnim i analitičkim bivarijantnim i multivarijantnim metodama.

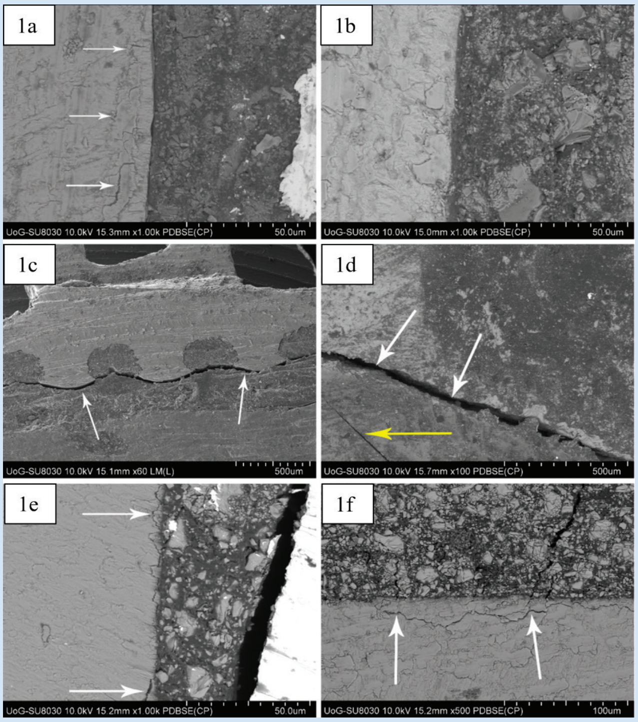
Numeričke serije analizirane su mjerama središnje tendencije i mjerama disperzije podataka. U numeričkim serijama gdje nema odstupanja od normalne distribucije, značajnost razlike testirana je Studentovim t-testom. Statistička značajnost razlika između više od triju numeričkih varijabli analizirana je ANOVA testom, a u slučaju značajnih razlika primijenjen je post-hoc Tukeyjev test. Za interval pouzdanosti od 95 % (IP) definirana je statistička značajnost za razinu pogreške manju od 0,05 (p).

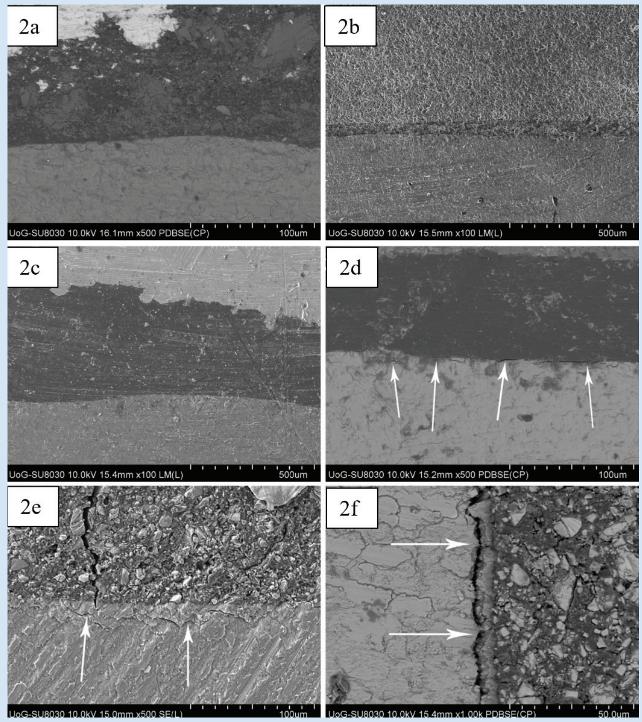
Rezultati

Na slici 1. su mikrofotografije uzoraka testiranih tjedan dana nakon lijepljenja. Mikrofotografije (slika 1. a, slika 1. b) pokazuju prisutnost mikroprostora između Transbonda XT i cakline, te se mogu primijetiti mnogobrojne mikropukotine unutar cakline. Izvrsno su vidljivi spojevi između adheziva i bravice. Na slici 1. c jasno se vidi pukotina unutar adheziva Heliosit Orthodontic koja se pruža tangencijalno duž vanjskog dijela retencijske mreže metalne bravice, a unutarnji dio adheziva dobro je povezan s bravicom. Veza između adheziva i cakline nije idealna – vidljive su pukotine na spoju između adheziva i cakline te povremeno unutar cakline. Slika 1. d prikazuje pukotine između adheziva i cakline, a žuta strelica pokazuje gdje je prijelom na spoju cakline i dentina koji je vjerojatno nastao tijekom pripreme uzoraka. Na slici 1. e uočava se mnogo pukotina unutar adheziva Fuji Ortho LC – neke se protežu prema caklini, a mikropukotine se mogu vidjeti i na površini cakline. Prisutni su prijelazi u adhezivu koji se nastavljaju unutar cakline na slici 1. f.

Na slici 2. a i na slici 2. b, veza adheziva Transbond XT s caklinom i bravicom na drugoj strani izgleda idealno, bez pojave mikroprostora. Slika 2. b prikazuje jaku i idealnu vezu između adheziva i cakline, te između adheziva i keramičke bravice, bez ikakvih mikroprostora.

Slika 2. c također prikazuje idealno sučelje između adheziva Heliosit Orthodontic s caklinom i bravicom bez ikakvih





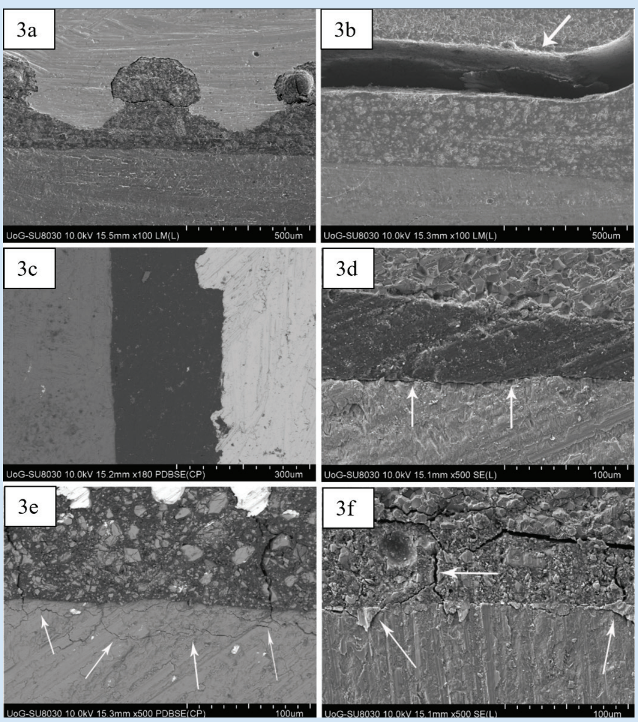


Figure 1 Micro-photographs of samples tested 1 week after bonding: 1a) Metallic bracket bonded with Transbond XT; 1b) Ceramic bracket bonded with Transbond Xt; 1c) Metallic bracket bonded with Heliosit Orthodontic; 1d) Ceramic bracket bonded with Heliosit Orthodontic; 1e) Metallic bracket bonded with Fuji Ortho LC adhesive; 1f) Ceramic bracket bonded with Fuji Ortho LC adhesive.

Slika 1. Mikrofotografije uzoraka testiranih tjedan dana nakon vezivanja: 1. a) Metalna bravica lijepljena Transbond XT-om; 1. b) Keramička bravica lijepljena Transbond XT-om; 1.c) Metalna bravica lijepljena Heliosit Orthodonticom; 1. d) Keramička bravica lijepljena Heliosit Orthodonticom; 1. e) Metalna bravica lijepljena Fuji Ortho LC-om 1. f) Keramička bravica lijepljena Fuji Ortho LC-om

Figure 2 Microphotographs of samples tested 2 weeks after bonding: 2a) Metallic bracket bonded with Transbond XT; 2b) Ceramic bracket bonded with Transbond XT; 2c) Metallic bracket bonded with Heliosit Orthodontic; 2d) Ceramic bracket bonded with Heliosit Orthodontic; 2e) Metallic bracket bonded with Fuji Ortho LC adhesive; 2f) Ceramic bracket bonded with Fuji Ortho LC adhesive.

Slika 2. Mikrofotografije uzoraka testiranih dva tjedna nakon vezivanja: 2. a) Metalna bravica lijepljena Transbond XT-om; 2. b) Keramička bravica lijepljena Transbond Xt-om; 2. c) Metalna bravica lijepljena Heliosit Orthodonticom; 2. d) Keramička bravica lijepljena Heliosit Orthodonticom; 2. e) Metalna bravica lijepljena Fuji Ortho LC-om; 2. f) Keramička bravica lijepljena Fuji Ortho LC-om

Figure 3 Micro-photographs of samples tested 3 weeks after bonding: 3a) Metallic bracket bonded with Transbond Xt; 3b) Ceramic bracket bonded with Transbond Xt; 3c) Metallic bracket bonded with Heliosit Orthodontic; 3d) Ceramic bracket bonded with Heliosit Orthodontic; 3e) Metallic bracket bonded with Fuji Ortho LC adhesive; 3f) Ceramic bracket bonded with Fuji Ortho LC adhesive.

Slika 3. Mikrofotografije uzoraka testiranih tri tjedna nakon vezivanja: 3. a) Metalna bravica lijepljena Transbond XT-om; 3, b) Keramička bravica lijepljena Transbond XT-om; 3. c) Metalna bravica lijepljena Heliosit Orthodonticom; 3. d) Keramička bravica lijepljena Heliosit Orthodonticom; 3, e) Metalna bravica lijepljena Fuji Ortho LC-om; 3. f) Keramička bravica lijepljena Fuji Ortho LC-om

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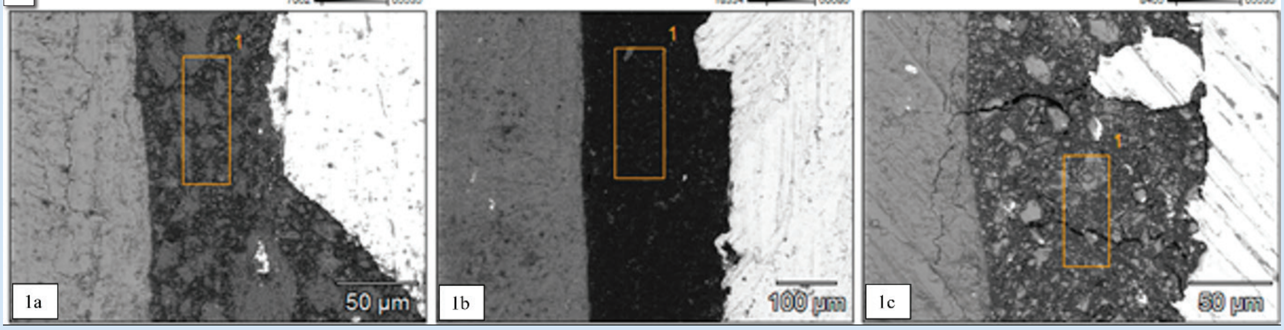
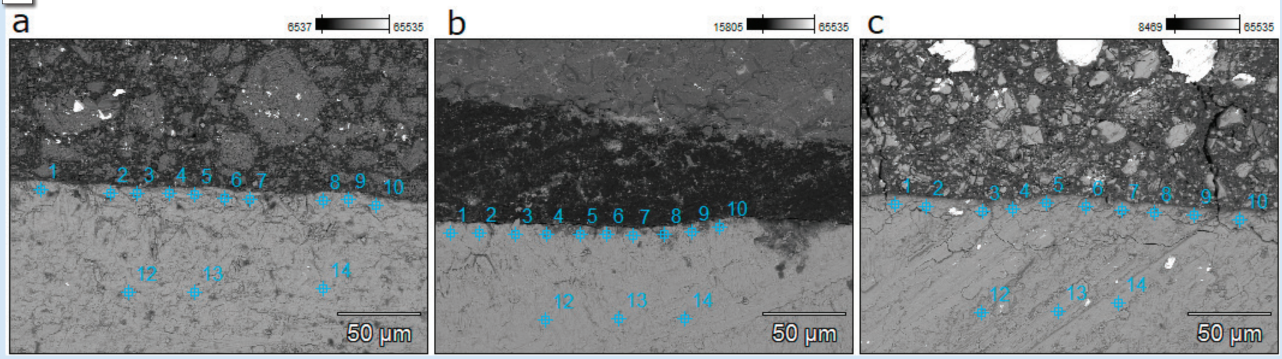


5



6

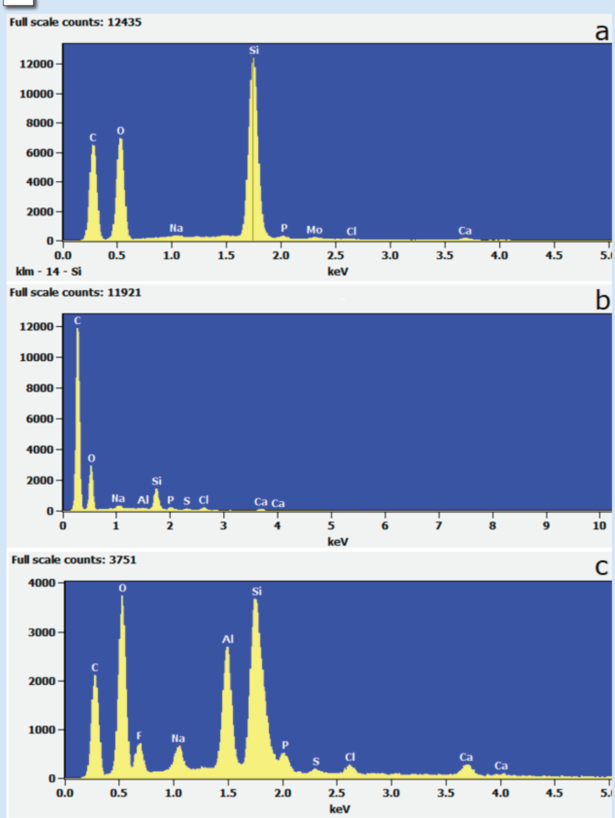


Figure 4 Micro-photographs of the selected areas for semi-quantitative EDX analysis of the adhesives: a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, USA) b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Liechtenstein) c) Fuji Ortho LC (GC Corporation, Japan.

Slika 4. Mikrofotografije odabranih područja za polukvantitativnu EDX analizu adheziva: a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, SAD) b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Lihtenštajn) c) Fuji Ortho LC (GC Corporation, Japan)

Figure 5 Micro-photographs of the point selection in the enamel for the semi-quantitative EDX analysis: a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, USA) b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Liechtenstein) c) Fuji Ortho LC (GC Corporation, Japan)

Slika 5. Mikrofotografije odabira točaka na caklini za polukvantitativnu EDX analizu: a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, SAD) b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Lihtenštajn) c) Fuji Ortho LC (GC Corporation, Japan)

Figure 6 Overview of the elements found in the selected surface of the samples (y-axis: number of counts, x-axis: energy in keV: a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, USA): presence of carbon (C), oxygen (O), sodium (Na), silicon (Si), phosphorus (P), molybdenum (Mo), chlorine (Cl) and calcium (Ca); b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Liechtenstein): presence of carbon (C), oxygen (O), sodium (Na), aluminum (Al), silicon (Si), phosphorus (P), sulphur (S), chlorine (Cl) and calcium (Ca); c) Fuji Ortho LC (GC Corporation, Japan): presence of carbon (C), oxygen (O), fluorine (F), sodium (Na), aluminum (Al), silicon (Si), phosphorus (P), sulphur (S), chlorine (Cl) and calcium (Ca).

Slika 6. Pregled elemenata pronađenih na odabranoj površini uzoraka (y-os: broj očitanja, x-os: energija u keV): a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, SAD): prisutnost ugljika (C), kisika (O), natrija (Na), silicija (Si), fosfora (P), molibdena (Mo), klora (Cl) i kalcija (Ca); b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Lihtenštajn): prisutnost ugljika (C), kisika (O), natrija (Na), aluminija (Al), silicija (Si), fosfora (P), sumpora (S), klora (Cl) i kalcija (Ca); c) Fuji Ortho LC (GC Corporation, Japan): prisutnost ugljika (C), kisika (O), fluora (F), natrija (Na), aluminija (Al), silicija (Si), fosfora (P), sumpora (S), klora (Cl) i kalcija (Ca).

tween the adhesive and the enamel, as well as the adhesive and the ceramic bracket, without any microspaces.

Figure 2c also displays an ideal interface between the adhesive Heliosit Orthodontic with the enamel and the bracket without any micropores after 2 weeks after bonding. In the microphotograph the interface between Heliosit Orthodontic adhesive and the enamel in certain segments is excellent, and cracks can be seen inside the enamel (Figure 2d) as a result of tight connection. Finally, the interface of the adhesive with the ceramic bracket is adequate.

Figure 2e shows microcracks in Fuji Ortho LC adhesive itself that extend into the enamel. The interface between the adhesive and the enamel looks good, while microspaces are present at the interface between the adhesive and the bracket. In Figure 2f, the cracks inside the adhesive can be clearly seen, but there is also a microfracture along the entire surface of the enamel, where the fractured superficial part of the enamel is firmly attached to the adhesive.

After 3 weeks, the microphotographs of the samples bonded with Transbond XT show that the connection between the adhesive and the enamel is ideal, while in the retention net of the metallic bracket, cracks appear in the adhesive itself. In Figure 3b, the ceramic bracket is debonded partially from the tooth, which is probably a result of the trauma caused during separation and sample preparation. However, the fracture line is in the adhesive itself.

Heliosit Orthodontic is very well connected to the enamel and the metallic bracket (Figure 3c), while in Figure 3d, a microcrack is observed that goes along the entire contact surface between the adhesive and the enamel, while the interface with the ceramic bracket is acceptable.

Figure 1e shows cracks in the adhesive which penetrate into enamel. The bond with the enamel appears strong, without microspaces between the adhesive and the enamel, likewise the bond between the adhesive and the metallic bracket. After 3 weeks, (Figure 3f), microcracks are noticed in the adhesive. They penetrate deep into the enamel. The interface between the adhesive and the ceramic bracket is inadequate and microspaces are visible.

For each adhesive used, a semi-quantitative SEM/EDX surface analysis of a selected area in the adhesive itself was performed in order to determine their elemental composition (Figure 4a, 4b, 4c. and Figure 6.)

SEM/EDX semi-quantitative line-scan analysis was performed to explore the elemental distribution in the surface enamel (Figure 5a, 5b and 5c). The average values of elements expressed in % mass in all three groups, analyzed by the semi-quantitative EDX point analysis in samples are presented in Table 1. The difference registered between the average values of the elements for the adhesive Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, USA) bonded to ceramic and metallic brackets, in the group tested 2 and 3 weeks after bonding, according to the t- test is statistically insignificant for $p > 0.05$. In the group tested 1 week after bonding, the difference is statistically significant for $p < 0.05$ for Si, P, Ca and S.

The difference registered between the average values of the elements for the adhesive Heliosit Orthodontic (Ivoclar

mikropora dva tjedna nakon spajanja. Na mikrofotografiji je spoj između adheziva Heliosit Orthodontic i cakline u određenim segmentima izvrstan, a unutar cakline vide se pukotine (slika 2. d) kao rezultat čvrste veze. Na kraju, spoj adheziva s keramičkom bravicom je adekvatan.

Na slici 2. e uočavaju se mikropukotine u samom adhezivu Fuji Ortho LC koje se protežu u caklinu. Spoj između adheziva i cakline izgleda dobro, no između adheziva i bravice postoje mikroprostori. Na slici 2. f jasno se vide pukotine unutar adheziva, ali i mikropukotina duž cijele površine cakline gdje je slomljeni površinski dio cakline čvrsto povezan adhezivom.

Nakon tri tjedna mikrofotografije uzoraka spojenih Transbond XT-om pokazuju da je veza između adheziva i cakline idealna, no u retencijskoj mreži metalne bravice ima pukotina u adhezivu. Na slici 3. b keramička se bravica djelomično odvojila od zuba, što je vjerojatno rezultat traume tijekom odvajanja i pripreme uzoraka, ali linija prijeloma je u samom adhezivu.

Heliosit Orthodontic vrlo dobro povezuje i caklinu i metalnu bravicu (slika 3. c), a na slici 3. d možemo primijetiti mikropukotinu duž cijele kontaktne površine između adheziva i cakline, dok je spoj s keramičkom bravicom prihvatljiv.

Slika 3. e prikazuje pukotine u adhezivu koje prodiru u caklinu. Veza s caklinom čini se snažna, bez mikroprostora između adheziva i cakline, jednako kao i veza između adheziva i metalne bravice. Nakon tri tjedna (slika 3. f) primjećuju se mikropukotine u adhezivu koje prodiru duboko u caklinu. Spoj između ljepila i keramičke bravice je neadekvatan, a mikropukotine su vidljive.

Za svaki korišteni adheziv, obavljena je polukvantitativna SEM/EDX analiza odabrane površine adheziva kako bi se odredila njihova elementarna kompozicija (slike 4. a, 4. b, 4. c i slika 6.).

SEM/EDX polukvantitativna analiza linije obavljena je da bi se istražila elementarna distribucija na površini cakline (slike 5. a, 5. b i 5. c). Prosječne vrijednosti elemenata izražene u % mase u svim trima skupinama, analizirane polukvantitativnom EDX analizom točaka u uzorcima, prikazane su u tablici 1. Razlika između prosječnih vrijednosti elemenata za adheziv Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, SAD) spojen s keramičkim i metalnim bravicama, u skupini testiranoj dva i tri tjedna nakon lijepljenja, prema t-testu statistički nije značajna za $p > 0,05$.U skupini testiranoj jedan tjedan nakon lijepljenja, razlika je statistički značajna za $p < 0,05$ za Si, P, Ca i S.

Razlika između prosječnih vrijednosti elemenata za adheziv Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Lihtenštajn) spojen s keramičkim i metalnim bravicama, u skupini testiranoj tri tjedna nakon lijepljenja prema t-testu statistički je značajna za $p < 0,05$ između Mg-a i P-a, a u skupini testiranoj jedan tjedan nakon lijepljenja statistički je značajna za $p < 0,05$ za Si i S.

Razlika među prosječnih vrijednosti elemenata za adheziv Fuji Ortho LC (GC Corporation, Japan) spojen keramičkim i metalnim bravicama, u skupini testiranoj tri tjedna nakon lijepljenja prema t-testu statistički je značajna za $p < 0,05$ samo za Si; u skupini testiranoj dva tjedna nakon spajanja sta-

Table 1 Average values of elements found in the enamel enamel expressed in % mass in all three groups (semi-quantitative EDX point analysis): Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, USA) b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Liechtenstein) c) Fuji Ortho LC (GC Corporation, Japan), bonded to metallic and ceramic brackets

Tablica 1. Prosječne vrijednosti elemenata pronađenih u caklini izražene u masenom % u svim trima grupama (polukvantitativna EDX analiza točaka): a) Transbond XT Light Cure Adhesive (3M Unitek Orthodontic Products, SAD) b) Heliosit Orthodontic (Ivoclar Vivadent, Schaan, Lihtenštajn) c) Fuji Ortho LC (GC Corporation, Japan), vezane za metalne i keramičke bravice

	Transbond XT						Heliosit Orthodontic						Fuji Ortho LC					
	Metallic bracket • Metalne bravice			Ceramic bracket • Keramičke bravice			Metallic bracket • Metalne bravice			Ceramic bracket • Keramičke bravice			Metallic bracket • Metalne bravice			Ceramic bracket • Keramičke bravice		
	Nr.	Avg.	StD	Nr.	Avg.	StD	Nr.	Avg.	StD	Nr.	Avg.	StD	Nr.	Avg.	StD	Nr.	Avg.	StD
Group tested 1 week after bonding • Grupa 1 testirana tjedan dana nakon lijepljenja																		
Mg	13	**0.09**	0.03	13	**0,1**	0,037	12	**0.1**	0.1	13	**0.1**	0.0	10	**0.1**	0.05	12	**0.1**	0.0
Si	13	**0.6**	0.2	13	**0,4**	0,1	13	**0.5**	0.2	13	**0.1**	0.1	12	**0.2**	0.1	13	**0.2**	0.1
P	13	**19.2**	3.9	13	**15,3**	1,3	13	**16.1**	2.4	13	**14.6**	1.6	13	**16.8**	2.2	13	**14.5**	1.5
S	13	**0.8**	0.3	13	**0,6**	0,1	13	**0.8**	0.3	13	**0.3**	0.1	13	**0.3**	0.1	13	**0.3**	0.1
Ca	13	**43.9**	12.5	13	**31.0**	3,1	13	**33.7**	6.1	13	**29.8**	3.8	13	**37.6**	6.3	13	**30.9**	3.5
F	/	/	/	/	/	/	/	/	/	/	**/**	/	13	**0.7**	0.2	13	**0.7**	0.5
Group 2 tested 2 weeks after bonding • Grupa 2 testirana dva tjedna nakon lijepljenja																		
Mg	5	**0.1**	0.0	13	**0,1**	0,0	11	**0.1**	0.0	12	**0.1**	0.0	7	**0.1**	0.0	11	**0.1**	0.03
Si	10	**0.1**	0.0	11	**0,2**	0,2	11	**0.1**	0.0	13	**0.1**	0.0	13	**0.6**	1.5	13	**0.5**	0.1
P	13	**14.7**	0.9	13	**15,5**	1,8	13	**17.9**	2.03	13	**17.6**	1.9	13	**15.7**	2.3	13	**15.5**	3.2
S	8	**0.2**	0.1	10	**0,1**	0,042	4	**0.1**	0.0	7	**0.1**	0.0	6	**0.1**	0.1	13	**1.05**	0.3
Ca	13	**30.4**	2.4	13	**31,1**	4,6	13	**37.2**	4.9	13	**37.0**	3.9	13	**34.9**	6.8	13	**33.4**	12.5
F	/	/	/	/	/	/	/	**/**	/	/	**/**	/	12	**1.0**	0.4	12	**0.5**	0.4
Group tested 3 weeks after bonding • Grupa 3 testirana tri tjedna nakon lijepljenja																		
Mg	12	**0.1**	0.0	12	**0,1**	0,0	10	**0.2**	0.0	12	**0.1**	0.0	5	**0.2**	0.1	9	**0.1**	0.1
Si	11	**0.2**	0.2	12	**0,1**	0,1	6	**0.1**	0.1	10	**0.2**	0.07	13	**0.2**	0.1	13	**0.1**	0.04
P	13	**17.1**	2.2	13	**17,8**	1.0	10	**18.6**	2.7	13	**15.6**	1.8	13	**17.0**	2.2	13	**16.4**	2.7
S	13	**0.2**	0.2	13	**0,1**	0,1	5	**0.2**	0.1	11	**0.2**	0.1	11	**0.2**	0.1	9	**0.1**	0.1
Ca	13	**38.4**	9.4	13	**38,1**	3,2	10	**39.1**	8.0	13	**33.7**	7.6	13	**40.6**	8.3	13	**38.2**	9.7
F	/	/	/	/	/	/	/	**/**	/	/	**/**	/	9	**1.5**	1.1	9	**1.3**	0.8

Vivadent, Schaan, Liechtenstein) adhesive bonded to ceramic and metallic brackets, in the group tested 3 weeks after, the bonding according to the t-test is statistically significant for $p <0.05$ between Mg and P, and in the group tested 1 week after, the bonding is statistically significant for $p <0.05$ between Si and S.

The difference between the mean values of the elements for the Fuji Ortho LC (GC Corporation, Japan) adhesive bonded to ceramic and metallic brackets, in the group tested 3 weeks after, the bonding according to the t-test is statistically significant for $p <0.05$ only for Si; while in the group tested 2 weeks after, the bonding is statistically significant for $p <0.05$ only between F and S, and in the group tested 1 week after, the bonding is statistically significant for $p <0.05$ between Mg, P and Ca.

Both in the surface and in the point analysis, i.e. in the adhesives themselves and in the surface enamel, fluoride ion was found in the samples of/or bonded with Fuji Ortho LC (Table 1).

tistički je značajna za $p < 0,05$ samo za F i S, a u skupini testiranoj jedan tjedan nakon spajanja statistički je značajna za $p < 0,05$ za Mg, P i Ca.

Također, kako u površinskoj tako i u točkastoj analizi, odnosno u samim adhezivima i na površini cakline, fluoridni ioni pronađeni su u uzorcima spojenima adhezivom Fuji Ortho LC (tablica 1.).

Discussion

In the current study, the adhesion of three orthodontic adhesives, commonly used in clinical practice, was evaluated in *in vitro* conditions. In *in vitro* studies, the effects of force during mastication, bad habits, type of food and beverages consumed during therapy, chemical and physical degra-

Rasprava

U ovom istraživanju procijenjena je adhezija triju ortodontskih adheziva koji se uobičajeno koriste u kliničkoj praksi, ali u uvjetima *in vitro*. Važno je napomenuti da istraživanja *in vitro* ne mogu potpuno replicirati kompleksne interakcije koje se događaju u oralnom okružju tijekom žvakanja, izladation, saliva pH, oral hygiene and bacterial activity, are just a small part of the complex interaction of the processes that cannot be reproduced (11-13), especially when it comes to fixed orthodontic appliances.

During the preparation of the samples for electron-microscopic observation, they are exposed to trauma while cutting the tooth in longitudinal direction; whereas, on the other hand, when placing the samples in a vacuum, the tooth and the adhesive become dehydrated and contraction between them is possible, which was visible on the examined samples (Figure 1a, 1c, 1d, 1e, 1f, 2d, 2e, 2f, 3b, 3d, 3e, 3f); and according to previous studies this line does not show (14). In the current study, even with the shortcomings of the preparation protocol, all adhesives remained bonded to the teeth, except for one of the specimens with ceramic bracket bonded with Transbond XT Light Cure Adhesive, which was partially detached.

The two interfaces, namely 1) the one between the bracket and the adhesive, or 2) the area between the enamel and the adhesive, are subject to damage (5, 15).

Firstly, *the analysis of the interface between the bracket and the adhesive* indicates that the specimens bonded to ceramic brackets with Transbond XT Light Cure Adhesive show an ideal connection with the bracket, without appearance of microspaces, which is in line with other studies (1, 15-17), claiming that ceramic brackets have a significantly stronger bond compared to metallic brackets. The use of ceramic brackets significantly improves esthetics (18), but they are very brittle and their dimensional change is less than 1%, therefore, during debonding, the possibility remains for fractures to appear in the ceramic brackets themselves. (4,5). Regarding both Fuji Ortho LC and Heliosit Orthodontic ceramic bracket bonded specimens, the interface was of similar quality as in the metallic bracket bonded specimens, again, without micropores.

Transbond XT Light Cure Adhesive and Heliosit Orthodontic in all metallic bracket bonding specimens was always filled to the smallest retention points of the bracket, without appearance of bubbles, with rare occurrence of microfractures in the adhesive itself, which probably occurred during vacuum placement. Fuji Ortho LC adhesive in metallic bracket bonded specimens was relatively well-filled, with frequent fractures in the adhesive itself and a rare appearance of bubbles, probably due to the mixing of the powder and liquid. This is in line with other findings, which claim that Transbond XT Light Cure Adhesive is a golden standard, and is a better adhesive than Fuji Ortho LC. (19)

Secondly, *the analysis of the interface between the adhesive and the enamel*, found that Transbond XT Light Cure Adhesive ideally adheres to the enamel, without appearance of micropores, while Heliosit Orthodontic has a relatively good interface with the enamel, with frequent micropores in the adhesive and microcracks in the enamel. The space between the tooth and the bracket appeared filled to the smallest retention points of the bracket, without appearance of bubbles, and with rare microfractures in the adhesive itself. Similarly, previous reports (17-19) have stated that Transbond XT Light Cure Adhesive and Heliosit Orthodontic showed results that are clinically acceptable.

ganja različitim tvarima i drugim čimbenicima povezanima s fiksnim ortodontskim aparatom (11 – 13).

Pri pripremi uzoraka za elektronsko-mikroskopsko promatranje uzorci su izloženi traumi tijekom uzdužnog rezanja zuba, a i njihovim stavljanjem u vakuum; zub i adheziv postaju dehidrirani što može prouzročiti kontrakciju između njih, što je vidljivo na ispitivanim uzorcima (slike 1. a, 1. c, 1. d, 1. e, 1. f, 2. d, 2, e, 2. f, 3. b, 3. d, 3. e, 3. f); no sukladno dosadašnjim istraživanjima, ova linija nije spomenuta u istraživanju i kako se uspoređuju s prethodnima, što može biti zbunjujuće (14). Unatoč nedostatcima u protokolu pripreme, svi su adhezivi ostali povezani sa zubima, osim jednog uzorka s keramičkom bravicom vezanog Transbond XT Light Cure Adhesiveom koji se djelomično odvojio.

Dva ključna spoja – 1) između bravice i adheziva te 2) područje između cakline i adheziva – podložna su oštećenjima (5, 15).

Prvo, *analiza spoja između bravice i adheziva* pokazuje da preparati koji su lijepljeni Transbond XT Light Cure Adhesiveom s keramičkim bravicama postižu idealni spoj bez mikrorazmaka, što se slaže s drugim istraživanjima (1, 15 – 17) u kojima se tvrdi da keramičke bravice imaju značajno jaču vezu u usporedbi s metalnima. Upotreba keramičkih bravica znatno poboljšava estetiku (18), ali veoma su lomljive, a njihova dimenzijska promjena manja je od 1 %, pa tijekom uklanjanja mogu puknuti (4, 5). Kad je riječ o uzorcima keramičkih bravica spojenih s pomoću adheziva Fuji Ortho LC i Heliosit Orthodontic, spoj je bio slične kvalitete kao i kod uzoraka s metalnim bravicama, ponovno bez mikropora.

Transbond XT Light Cure Adhesive i Heliosit Orthodontic – u svim uzorcima s metalnim bravicama spojevi su uvijek bili ispunjeni do najmanjih retencijskih točaka bravice, bez pojave mjehurića, uz rijetku pojavu mikropukotina u adhezivu, što se vjerojatno dogodilo pri postavljanju pod vakuumom. Adheziv Fuji Ortho LC u uzorcima metalnih bravica bio je relativno dobro ispunjen, s čestim pukotinama u adhezivu i rijetkom pojavom mjehurića, vjerojatno zbog miješanja praha i tekućine. To je u skladu s drugim saznanjima prema kojima je Transbond XT Light Cure Adhesive zlatni standard i bolji adheziv od Fuji Ortho LC-a (19).

Drugo, *analiza spoja između adheziva i cakline* pokazala je da Transbond XT Light Cure Adhesive idealno prianja uz caklinu, bez pojave mikropora, a Heliosit Orthodontic ima razmjerno dobar spoj s caklinom, ali s čestim mikroporama u adhezivu i mikropukotinama u caklini. Prostor između zuba i bravice činio se ispunjenim do najmanjih retencijskih točaka bravice, bez pojave mjehurića i s rijetkim mikropukotinama u adhezivu. Slično tomu, u prethodnim izvješćima (17 – 19) navodi se da su Transbond XT Light Cure Adhesive i Heliosit Orthodontic pokazali klinički prihvatljive rezultate.

Fuji Ortho LC pokazao je relativno dobar spoj s caklinom, prikazujući mikropukotine unutar cakline, ali nikada u adhezivu. To upućuje na vrlo snažnu vezu adheziva s caklinom. Rezultati Fuji Ortho LC-a u nekim dosadašnjim istraživanjima bili su statistički inferiorni u usporedbi s drugim adhezivima, iako su zadovoljavali za kliničku uporabu (11, 20, 21). No u ovom istraživanju pokazano je vrlo snažno prianjanje uz caklinu, što se može dogoditi zbog korištenja

Fuji Ortho LC showed a relatively good interface with the enamel, exhibiting microcracks inside the enamel, but never in the adhesive itself. This indicates a very strong bond of the adhesive with the enamel. The results of Fuji Ortho LC in some of the previous studies were statistically inferior to other adhesives, although they were still satisfactory for clinical purposes (11, 20, 21). In this study, on the contrary, a very strong bond with the enamel was demonstrated, which may be because 37% orthophosphoric acid etching was used, according to the protocols reported previously. (19,22) In a study by Cheng et al. (23), the results showed that under the same etching procedure, the bond strength was statistically higher on the Fuji Ortho LC compared to Transbond XT Light Cure Adhesive.

In most studies, conventional etching has shown a stronger bond, and this might be due to greater penetration into the enamel and decomposition of hydroxyapatite crystals by 37% orthophosphoric acid (3, 18, 24-26). Sfondrini et al. (27) did not observe a significant difference in bond strength between composite resin bonding and resin-modified GIC when acid etching was performed, unlike the situation where the brackets were bonded to resin-modified GIC without etching, when the bond strength was statistically higher in the composite resin. These studies are in accordance with the previously reported findings of the present study.

Undoubtedly, the orthodontic appliances increase the caries risk (28). Therefore, it is important to emphasize that of all tested adhesives, only in Fuji Ortho LC, the elemental composition obtained by surface SEM/EDX analyses, as well as the semiquantitative SEM/EDX line-scan analysis of randomly selected points in the surface of the enamel, showed the presence of fluorides, which is known to have anticariogenic properties (29). The latter indicates the incorporation of fluorides into enamel due to the fluoride release from the resin-modified GIC adhesive, and formation of an ion-exchange layer, typical of GICs, a property reported in previous studies (30).

Conclusions

Within the limitations of this *in vitro* study, the obtained results lead to conclusions that:

Transbond XT Light Cure Adhesive showed an ideal bond to the enamel without the presence of micro-spaces. However, Fuji Ortho LC established a strong bond to the enamel due to the presence of the ion-exchange layer. The combination of conventional orthophosphoric acid etching and silane-based ceramic bracket bonding establishes the strongest adhesion, but this bond can cause enamel fracture during debonding. Fuji Ortho LC released fluoride ions in the surface layers of the enamel, which could contribute to its anticariogenic properties.

Author Contributions: Conceptualization - **A.A., E.G.**; methodology - **I.G., N.C**; software - **N.C.**; validation - **M.V., D.G.**; formal analysis - **A.A., N.C.**; investigation - **A.A., I.G.**; resources - **E.G.**; data curation - **M.V.**; writing original draft preparation - **A.A.**; writing review and editing - **E.G., M.V.**; visualization - **D.G.**; supervision - **E.G.**; project administration - **I.G.**; funding acquisition - **I.G.** All authors have read and agreed to the published version of the manuscript.

37-postotne ortofosforne kiseline za jetkanje, prema do sada objavljenim protokolima (19, 22). Cheng i suradnici (23) u svojem su istraživanju pokazali da je, u jednakom postupku jetkanja, čvrstoća veze bila statistički veća kod Fuji Ortho LC-a u usporedbi s Transbond XT Light Cure Adhesiveom.

U većini istraživanja konvencionalno je jetkanje pokazalo jaču vezu, što može biti zbog većeg prodiranja u caklinu i razgradnje hidroksiapatitnih kristala s 37-postotnom ortofosfornom kiselinom (3,18, 24 – 26). Sfondrini i suradnici (27) nisu uočili značajnu razliku u čvrstoći veze između kompozitne smole i smolom modificiranoga stakleno-ionomernog cementa (SIC) kad je provedeno jetkanje kiselinom. To se slaže s prethodno objavljenim saznanjima u tom istraživanju.

Neupitno je da ortodontski aparati povećavaju rizik od pojave karijesa (28). Zato je važno istaknuti da je, od svih testiranih adheziva, samo u Fuji Ortho LC-u, analizama površine s pomoću SEM/EDX tehnike, dobivena elementarna kompozicija koja pokazuje prisutnost fluorida. Uz to, polukvantitativna analiza linije s pomoću SEM/EDX tehnike na nasumično odabranim točkama na površini cakline pokazala je prisutnost fluorida koji je poznat po svojstvima sprječavanja nastanka karijesa (29).To upućuje na ugradnju fluorida u caklinu zbog njegova otpuštanja iz adheziva na bazi smolom modificiranoga stakleno-ionomernog cementa (SIC) i formiranje sloja ionizacije tipičnoga za SIC-ove, što je svojstvo objavljeno u prethodnim istraživanjima (30).

Zaključci

U sklopu ograničenja ovog istraživanja *in vitro*, dobiveni rezultati dovode do sljedećih zaključaka: Transbond XT Light Cure Adhesive pokazao je idealno prianjanje za caklinu bez pojave mikroprostora; međutim, Fuji Ortho LC snažno prianja za caklinu zbog prisutnosti ionskog sloja izmjene, kombinacija konvencionalnog jetkanja ortofosfornom kiselinom i vezivanja keramičke bravice na bazi silana omogućuje najjače prianjanje, ali to prianjanje može prouzročiti lom cakline tijekom uklanjanja. Fuji Ortho LC oslobađa ione fluorida u površinskim slojevima cakline, što bi moglo pridonijeti njegovim antikariogenim svojstvima.

Doprinosi autora: **A.A., E. G.** – koncepcija; **I. G., N. C.** – metodologija; **N.C.** – softver; **M. V., D. G.** – validacija; **A. A., N. C.** – formalna analiza; **A. A., I. G.** – istraživanje; **E. G.** – resursi; **M. V.** – izračun podataka; priprema originalnog teksta – **A. A.**; **E. G., M. V.** – pregled i uređivanje; **D. G.** – vizualizacija; **E. G.** – nadzor; **I. G.** – upravljanje projektom; **I.G.** – financiranje. Svi autori pročitali su tekst i suglasili se s objavljenom verzijom.

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Sažetak

Ciljevi: Željelo se usporediti tri različita ortodontska adheziva (Transbond XT Light Cure Adhesive, Heliosit Orthodontic, Fuji Ortho LC) za pričvršćivanje dviju vrsta ortodontskih bravica: metalnih (Fascination Roth 0.22) i keramičkih (Topic Roth 0.22. Dentaurum). **Materijali i metode:** Istraživanje je provedeno na 18 ljudskih zuba (po 6 za svaki adheziv). Podijeljeni su u tri skupine prema vremenu ispitivanja i promatrani su poslije jednoga, dva i tri tjedna nakon pričvršćivanja. Nakon eksperimentalnog postupka uzorci zuba prerezani su na pola duž longitudinalne osi u vestibulo-oralnom smjeru, fiksirani provodnim karbonskim cementom, postavljeni u visokovakuumski evaporator i zatim premazani ugljikom. Jedna polovina svakog uzorka promatrana je pištoljem za emisiju polja skenirajućeg elektronskog mikroskopa (FEG-SEM Hitachi SU 8030, Japan), a na drugoj polovini provedena je kvalitativna (x-zrake analizirajućih linija) i polukvantitativna energijska disperzivna analiza rendgenskih zraka (EDX) Thermo Noran (SAD) NSS Systemom 7, opremljenim Ultra Dry detektorom (prozor od 30 mm^2). **Rezultati:** Transbond XT idealno se povezao s caklinom i bazom bravice pa su mikrorazmaci i pukotine u caklini bili rijetki. Heliosit Orthodontic pokazao je bolji odnos povezivanja s bazom bravice nego s caklinom gdje su kasnije uočeni mikrorazmaci u vezi. Mikrofotografije Fuji Ortho LC-a pokazale su mnogo pukotina unutar adheziva, a neke od njih nastavile su se na površini cakline. Zato postoji dojam o vrlo čvrstom povezivanju s caklinom i s pukotinama na površini cakline, ali ne i na spoju caklina-adheziv. Mikrorazmaci su se pojavili i na spoju bravica i adheziva. **Zaključak:** Transbond XT visoko je punjena kompozitna smola i idealni ortodontski adheziv u svakom ispitivanom aspektu, s idealnim spojem caklina-adheziv i spojem bravica-adheziv. Heliosit Orthodontic pruža bolji spoj bravica-adheziv nego s caklinom. Fuji Ortho LC kao smolom modificirani stakleno-ionomerni cement pruža bolji spoj caklina-adheziv u usporedbi s bazom bravice.

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