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## Ispitivanje propusnosti kompozitnog ispuna - Pilot studija

### *Microleakage Evaluation of Resin Composite Fillings - A Pilot Study*

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#### Sažetak

Svrha rada bila je ispitati propusnost između restaurativnog materijala i stijenke zuba kod primjene dvaju različitih adhezijskih sustava. Ispitivanje je provedeno na 12 intaktnih trećih kutnjaka ekstrahiranih iz ortodontskih razloga. Zubi su prepiljeni na caklinsko-cementnom spojištu te su dobiveni krunski segmenti. Na okluzalnim površinama izrađeni su kaviteti I. razreda. Uzorci su podijeljeni u dvije skupine - po šest u svakoj. Za izradbu ispuna uporabljen je restaurativni kompozitni materijal "Tetric Ceram" (Vivadent, Schaan, Liechtenstein) i dva različita samojekajuća adhezijska sustava - u jednoj skupini dvofazni adhezijski sustav AdheSE (Vivadent, Schaan, Liechtenstein), a u drugoj jednofazni adhezijski sustav Xeno III (DeTrey, Konstanz, SR Njemačka). Za mjerenje mikropropusnosti koristila su se dva postupka - konstrukcija za prijenos tekućine i tehnika prodora boje. U rezultatima dobivenima konstrukcijom za prijenos tekućine nije ustanovljena statistički znatna razlika između dviju skupina uzoraka ( $p=0,7488$ ). Mjerenje rubnog propuštanja tehnikom prodora boje pokazalo je statistički znatno veće propuštanje ( $p=0,002$ ) u skupini u kojoj je uporabljen adhezijski sustav Xeno III.

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#### Uvod

Adhezijski sustavi su materijali koji povezuju kompozitni materijal s površinom tvrdih zubnih tkiva. Unatoč snažnom razvoju caklinsko-dentinskih adhezijskih sustava, restaurativni smolasti materijali i dalje nepotpuno prijanjaju za zid kaviteta. (1, 2).

Adhezija na caklinu postiže se nakon jetkanja caklinskih rubova kaviteta 32 – 37% ortofosforom kiselinom ili kiselim primerima, te se na taj način pretvara ili uklanja zaostatni sloj i omogućuje mikromehaničko vezivanje (3). Adhezijski susta-

#### Introduction

Adhesive systems are materials that connect resin composite with the surface of the tooth tissue. Despite of various enamel-dentin adhesive systems developed, the bond with cavity walls is still not satisfactory (1, 2).

Adhesion to enamel is enabled by etching of enamel margins of the prepared cavity with 32-37% phosphoric acid or acid primers that remove smear layer providing micromechanical interlocking (3). Adhesive systems are part of a hybrid layer formed

vi postaju dijelom hibridnog sloja koji nastaje tijekom procesa adhezije, bez obzira na to koriste li se tehnikom potpunog jetkanja ili samojetkajućim primerima. Hibridni sloj je područje prožimanja adhezijskog sustava (primera i adheziva), kolagenih vlakana i djelomično demineraliziranog dentina. Ako je infiltracija kolagene mreže nepotpuna, stvara se tzv. hibridoidni sloj sa smanjenom snagom veze (1, 2, 4). Stalnim razvojem adhezijskih sustava teži ih se pojednostavniti sjedinjenjem pojedinih postupaka (jetkanje, priming, bonding) kako bi se olakšala njihova klinička primjena (5). Usporedbom različitih generacija adhezijskih sustava dokazalo se da pojednostavljeni adhezijski sustavi pokazuju lošije kliničke rezultate od višekomponentnih (6).

Glavni problem u adhezijskoj stomatologiji jest čvrstoća veze koju omogućuju moderni adhezijski sustavi te skupljanje kompozitnih (smolastih) materijala tijekom polimerizacije zbog vezanja molekula monomera u polimerne lance, a posljedica je smanjenje volumena kompozitne smole (7). Na početku polimerizacije gubitak volumena kompenziran je "tečenjem" kompozita od slobodnih prema vezanim površinama (8). Tijekom procesa kompozit se stvrdnjava i kompenzacija "tečenjem" više ne slijedi skupljanje materijala. On se odvađa od zida kaviteta, a zbog snage veze dolazi do napetosti u samom materijalu, zubu ili na spoju zub-kompozitnog ispuna. Posljedica toga su adhezijske i kohezijske frakture te mikropukotine (9). Zbog toga nastaje mikropropuštanje, odnosno prodor oralnog fluida koji sadržava bakterije, molekule i ione između stijenke kaviteta i restaurativnoga kompozitnog materijala (10). Ako je rubno pečaćenje dobro, neće nastati rubna pukotina i spriječit će se mikropropusnost, poslijeoperativna preosjetljivost, sekundarni karijes ili iritacija pulpe (11). Današnji caklinsko-dentinski adhezijski sustavi ne mogu zapečatiti rubove i stijenke ispuna, ni dugoročno spriječiti mikropropusnost (12).

Ispitivanja rubnog propuštanja adhezijskih sustava i restaurativnih materijala provode se u laboratorijskim i kliničkim uvjetima (13). U laboratorijskim ispitivanjima *in vitro* postiže se detaljnija provjera pojedinih čimbenika, ali oni ne daju potpunu sliku, jer je gotovo nemoguće postići uvjete kojima je restaurativni materijal izvrgnut u usnoj šupljini. Laboratorijski postupci dijele se na "dvodimenzionalne" (rezani ili lomljeni uzorci) i "trodimenzionalne" (uzorci izvađeni iz kaviteta), a odnose se na mogućnost očitavanja prodora kontrasta uzduž rubne pukotine.

Najčešći postupak za ispitivanje rubne propusnosti jest tehnika prodora boje. To je jedan od kvanti-

during a process of adhesion, either by total-etch or self-etch technique. A hybrid layer is a zone where adhesive system (primer and bond), collagen fibrils and partially demineralized dentin, merge. If the infiltration of collagen network is insufficient, a so called hybridoid layer with decreased bond strength is formed (1, 2, 4). Continuous development of adhesive systems aims at simplification combining etching, priming and bonding that facilitates clinical use (5). Simplified adhesive systems showed poor clinical results when compared to multicomponent systems (6).

The main problem of adhesive dentistry is bond strength of modern adhesive systems, as well as resin composite material shrinkage that develops during polymerization due to formation of polymer chains from monomers into polymers with consequential volume reduction of the composite resin (7). At the start of polymerization the loss of volume is compensated by composite "flow" from the free to the bonded surfaces (8). During polymerization composite resin becomes solid and the flowing is no longer able to compensate for the shrinkage. Material is pulling away from the cavity wall and because of the bonding strength there is tension in the material, tooth or on the tooth-resin composite interface. Consequently, the adhesion and cohesion fractures lead to micro gaps (9) and microleakage i.e. the passage of the oral fluid containing bacteria, molecules and ions between the cavity wall and the restorative material (10). A proper marginal seal prevents a marginal gap formation and microleakage, postoperative sensitivity, secondary caries and pulp irritation (11). The enamel-dentin adhesive systems today are still unable to seal restoration margins and prevent microleakage on a long term basis (12).

Microleakage investigations of adhesive systems and restorative materials are performed by laboratory and clinical procedures (13). *In vitro* laboratory investigations provide more detailed evaluations of certain factors. Still, they do not display the whole situation since it is almost impossible to create the same conditions restorative material is exposed to in oral cavity. Laboratory methods are subdivided into "two-dimensional" (cut or fractured specimens) and "three-dimensional" (specimens removed from the cavities) relating to the ability of detecting a dye penetration along the marginal gap.

Commonly used method for marginal leakage detection is the dye penetration technique, a quantitative method showing the leakage along the restoration margin (1, 2). The dye particles are small enough

tativnih postupaka kojim se dobiva prikaz rubnog propuštanja (1, 2). Tinta – tuš ima dovoljno male molekule boje, pa može prodirati i obojiti mjesto na kojemu nije osigurano prilijeganje materijala uz stijenku kaviteta (2, 14), pa je prikladna za takva istraživanja.

Konstrukcija za prijenos tekućine također omogućuje kvantitativni postupak izmjere rubnog propuštanja. Tlak vode na apikalnu površinu uzorka uzrokuje gibanje tekućine kroz mikroprostor rubne pukotine, što je zabilježeno pomakom mjehurića u mikropipeti (15).

Svrha ovoga rada bila je ispitati propusnost između ispuna od kompozitnog materijala i stijenke zuba kod primjene dvaju samojetkajućih caklinsko-dentinskih adhezijskih sustava: dvofaznoga (AdheSE) i jednofaznog (Xeno III). Za procjenu rubnog propuštanja koristila su se dva postupka - mjerenje konstrukcijom za prijenos tekućine i tehnika prodira boje.

### **Materijal i postupak**

Ispitivanje je provedeno na 12 intaktnih humanih trećih kutnjaka sa završenim razvojem korijena, a ekstrahirani su iz ortodontskih razloga. Zubi su ekstrahirani na Zavodu za oralnu kirurgiju Stomatološkog fakulteta Sveučilišta u Zagrebu, a do istraživanja su čuvani u fiziološkoj otopini na konstantnoj temperaturi od 37° C.

Za postupak istraživanja zubi su mehanički očišćeni, a krune su odvojene dijamantnim brusilom od korjenova u visini caklinsko-cementnog spojišta. Zubna pulpa iz koronarnog dijela uklonjena je eskavatorom. Na okluzalnoj površini dijamantnim su brusilom (ISO 545/018) preparirani kaviteti, uz stalno hlađenje vodom. Bili su standardiziranih dimenzija - 6 mm mezio-distalna širina, 4 mm vestibulo-oralna širina i 3 mm dubina kaviteta, koničnog oblika, divergentnih stranica i ravnog dna radi lakše kontrole preparacije. Dubina prodiranja brusila od 3 mm kontrolirala se jednostavnom oznakom na svrdlu, povučenom vodootpornim flomasterom. Dužina i širina kaviteta kontrolirale su se milimetarskom ljestvicom.

Uzorci su slučajnim odabirom podijeljeni u dvije skupine od po šest uzoraka. U objema je upravljen kompozitni materijal "Tetric Ceram" boje A1 (Lot. E58491, 10/2006). U prvoj skupini koristio se caklinsko-dentinski adhezijski sustav "AdheSE" (Lot. F25882, 09/2004), a u drugoj "Xeno III" (Lot.0308001472, 06/2005) (Tablica 1.). Postupak rada s adhezijskim sustavima proveden je pre-

to penetrate and color the surface where the adhesion of restorative material and cavity wall is insufficient (2, 14) making them appropriate for this research.

A fluid transport model is another quantitative method that can also be used to determine microleakage. Water pressure to the apical surface of the specimen causes moving of the fluid through the microspace of the marginal gap that was detected by the air bubble movement in the micro-pipe (15).

The purpose of this study was to evaluate the microleakage between the cavity wall and resin composite restorations made with the use of two different self-etch adhesive systems: two-step self-etch AdheSE (Vivadent, Schaan, Liechtenstein), and single-step self-etch Xeno III (DeTrey, Konstanz, Germany). The evaluation of microleakage was made by two techniques: fluid transport model and dye penetration technique.

### **Materials and Methods**

The experiment was conducted on 12 human third molars with completely developed roots, extracted for orthodontic reasons. Teeth were extracted at the Department of Oral Surgery, School of Dental Medicine, University of Zagreb. They were stored in normal saline on constant temperature of 37°C until they were used.

Teeth were mechanically cleaned and sectioned at the cemento-enamel junction with a diamond bur in order to separate the crowns from the roots. The coronal part of the pulp was removed with an excavator. The cavities were prepared into the occlusal surface using a diamond bur (ISO 545/018), with constant water cooling. The cavities had standardized dimensions and were conically shaped, with divergent opposite sides and flat bottoms, with dimensions 6 mm mesio-distally, 4 mm labio-orally and 3 mm in depth. The flat bottom was planned and executed because of an easier preparation control. The 3 mm depth was controlled with a simple mark on the bur made with a waterproof ink. The depth and the length of the cavity were controlled with a millimeter scale.

The specimens were randomly divided into two groups, six specimens in each. In both groups the resin composite restorative material "Tetric Ceram" colour A1 (Vivadent, Schaan, Liechtenstein, Lot. E58491, 10/2006) was used. In group I "AdheSE" adhesive system (Vivadent, Schaan, Liechtenstein, Lot. F25882, 09/2004) was used, and in group II "Xeno III" (Dentsply-DeTray, Konstanz, Germany, Lot.0308001472, 06/2005) (Table 1.). The adhesive

**Tablica 1.** Usporedba rabljenih adhezijskih sustava  
**Table 1** Comparison of used adhesive systems

AdheSE	Vivadent, Shaan, Lichtenstein	samojetkajući • self-etch	dvokomponentni • twocomponent	dvofazni • two-step	otapalo - voda • solvent - water	pH=1,4 “srednje jaki” • “intermediary strong”
Xeno III	Dentsply-DeTray, Konstanz, Njemačka	samojetkajući • self-etch	dvokomponentni • twocomponent	jednofazni • single-step	otapalo - voda i alkohol • solvent - water and alcohol	pH=1,4 “srednje jaki” • “intermediary strong”

ma naputku proizvođača. Kompozitni restaurativni materijal postavljen je u kavitet slojevito (u dva nasuprotna kosa sloja). Materijali su polimerizirani halogenim svjetlom uređaja “Astralis 7” (Vivadent, Schaan, Liechtenstein) u polimerizacijskom programu “High power” intenziteta  $750 \text{ mW/cm}^2$ . Svaki sloj kompozitnoga restaurativnog materijala postavljen u kavitet polimeriziran je 40 sekundi.

Nakon polimerizacije i završne obrade poliranjem gumicama, uzorci su osušeni mlazom zraka i izolirani dvama premazima laka za nokte. Lak je nanesen oko ispuna na površinu cakline na udaljenosti 1-2 mm od ruba ispuna, a uzorci su zatim ostavljeni 24 sata na zraku kako bi se osušili. Površina dentina prema pulpi nije bila izolirana.

Na tako pripremljenim uzorcima izmjerena je mikropropusnost konstrukcijom za prijenos tekućine (Slika 1.). Uzorci su cijanoakrilatnim ljepilom učvršćeni u cjevčicu povezanu s mikropipetom i stavljeni u posudu s tekućinom. Rezna površina tlačena je sa 120 kPa. Propusnost se mjerila pomakom zračnog mjehurića u mikropipeti. Za svaki uzorak mjerenje je ponovljeno tri puta. Rezultati su izraženi u  $\mu\text{L}$  (mikrolitrama) i statistički obrađeni neparametrijskom Kruskal-Wallisovom analizom varijance.

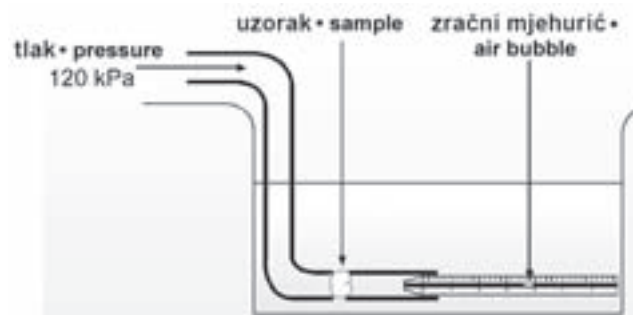
Nakon mjerenja konstrukcijom, uzorci su pripremljeni za mjerenje mikropropusnosti tehnikom prodora boje. Izolacijskim lakom premazane su i

systems were applied following the manufacturer’s instructions. The placement of resin composite in the cavity was incremental (two inclined layers opposite to each other). Materials were polymerized with halogen light from “Astralis 7” (Vivadent, Schaan, Liechtenstein) machine, set on “High power” polymerization program with intensity of  $750 \text{ mW/cm}^2$ . Every layer of resin composite in the cavity was polymerized for 40 seconds.

After polymerization and final polishing, the specimens were dried with compressed air and isolated with two layers of nail varnish. The varnish was put on enamel surfaces of the specimens up to 1-2 mm from the restoration margin and left to air dry for 24 hours. The dentin surface toward the pulp was not isolated.

Microleakage was measured on specimens prepared by the stated method using the fluid transport model (Figure 1.). The specimens were sealed with cyanoacrylate into the tube connected with micro-pipe and placed in the container with the fluid. The sectioned surface of the samples was pressurized with 120 kPa. Leakage was detected by movements of the air bubble in the micro-pipe. The measurement was repeated three times for each sample. The results are shown in  $\mu\text{L}$  and statistically analyzed using non-parametrical Kruskal-Wallis analysis of variance.

Following these measurements, the specimens were prepared to measure microleakage using dye



**Slika 1.** Konstrukcija za prijenos tekućine  
**Figure 1** Fluid transport model

dentinske površine uzoraka. Sušili su se 24 sata, a zatim su uronjeni u kontrastno sredstvo-tintu "Rotring Ink" (Stanford, GmbH, Hamburg, SR Njemačka). Nakon 48 sati isprani su mlazom vode i uronjeni u kiselinu (5-% dušična kiselina, HNO<sub>3</sub>), koja se mijenjala svaka dva dana. Za 10 dana tvrdo zubno tkivo dovoljno je omekšalo, pa su se ispuni vadili eskavatorom iz kaviteta.

Tako pripremljeni ispuni-uzorci promatrani su stereomikroskopom Olympus SZX-12 povezanim s digitalnom kamerom Olympus DP-12 (Olympus Optical Co., GmbH, Hamburg, SR Njemačka). Svaki uzorak snimljen je iz četiri profilna položaja pod povećanjem od 7x. Prije toga su površine ispuna-uzoraka označene drugom bojom kako bi se kasnije znalo koja je površina na pojedinoj snimci. Ukupno ih je snimljeno 48. Na kontaktnim površinama ispuna-uzoraka s površinom kaviteta promatrala se dubina prodora kontrastnog sredstva te je stupnjevana prema shemi prodora boje (Slika 2.). Rezultati su obrađeni Studentovim *t*-testom.



**Slika 2.** Shematski prikaz dubine prodora boje  
**Figure 2** The scheme of dye penetration depth

penetration technique. The dentin surfaces of specimens were isolated with isolating varnish. Samples were air dried for 24 hours and placed in contrast dye "Rotring Ink" (Stanford GmbH, Hamburg, Germany). After 48 hours the samples were rinsed with water and placed in 5% solution of HNO<sub>3</sub>. The acid was replaced every second day. During the ten days, the dental hard tissue softened enough to remove the fillings out from the cavities with an excavator.

The fillings were analyzed with stereomicroscope Olympus SZX-12 connected to digital camera Olympus DP-12 (Olympus Optical Co. GmbH, Hamburg, Germany). Fillings were photographed in four profile marked positions under magnification (7x). Previously, the surfaces of samples were marked with different colours in order to identify them on photographs. In total 48 photographs images were taken. On contact surfaces of the samples, facing the cavity wall, the dye penetration was analyzed using the scheme in Figure 2.). Statistic analysis was performed by Student *t*-test.

- 0 nema prodora boje / no dye penetration
- I prodor boje do 1/3 zida ispuna / dye penetration covers 1/3 of the cavity wall
- II prodor boje do 2/3 zida ispuna / dye penetration covers 2/3 of the cavity wall
- III prodor boje cijelom dužinom zida ispuna / dye penetration covers the cavity wall
- IV prodor boje cijelom dužinom zida ispuna, a zahvaćeno je i dno ispuna / dye penetration covers the cavity wall and the cavity bottom

## Rezultati

Ovim istraživanjem ustanovljeno je rubno propuštanje u oba postupka mjerenja. Rezultati propusnosti dobiveni konstrukcijom za prijenos tekućine predstavljani su na Slici 3. Između dvaju adhezivskih sustava ( $p=0,7488$ ) konstrukcijom za prijenos tekućine nije utvrđena statistički znatna razlika u propusnosti.

Rezultati dubine prodora boje za obje skupine prikazani su u Tablicama 2 i 3. Kod dvaju ispuna obojene su tri površine, a u ostalim uzorcima sve četiri (Slike 4., 5. i 6.). Na svakom ispunu određena je najveća dubina prodora boje. Skupina II. (Xeno III) pokazala je statistički znatno veći stupanj srednje vrijednosti najdubljih prodora boje ( $p=0,002$ ) od skupine I. (AdheSE).

## Results

Both methods in the study detected microleakage. Microleakage measured by the fluid transport model is shown in figure 3. There was no statistically significant difference between microleakage of two adhesive systems ( $p=0,7488$ ) measured by the fluid transport model.

The results of dye penetration in both groups are shown in Tables 2 and 3. Two fillings had three surfaces coloured, while other specimens had all four of their surfaces coloured (Figures 4, 5 and 6). The deepest point of dye penetration was determined for each specimen. Group II. (Xeno II) showed statistically significant higher degree of mean values in extent of dye penetration ( $p=0,002$ ) than group I. (AdheSE).

**Tablica 2.** Rezultati dubine prodora boje za I. skupinu (AdheSE)

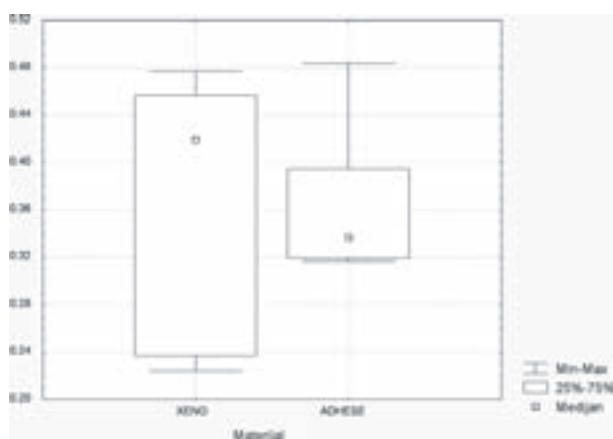
**Table 2** Results of dye penetration in group I (AdheSE)

Ispun • Filling	Dubina prodora boje • Dye penetration depth				Površina s najdubljim prodorom boje • Dye penetration surfaces
	I	I	I	O	
1.	I	I	I	O	I
2.	II	I	II	II	II
3.	I	I	II	O	II
4.	II	I	II	I	II
5.	I	I	II	I	II
6.	II	II	II	I	II

**Tablica 3.** Rezultati dubine prodora boje za II. skupinu (Xeno III)

**Table 3** Results of dye penetration in group II (XenoIII)

Ispun • Filling	Dubina prodora boje • Dye penetration depth				Površina s najdubljim prodorom boje • Dye penetration surfaces
	II	II	II	III	
1.	II	II	II	III	III
2.	III	III	III	II	III
3.	I	III	II	III	III
4.	III	III	III	III	III
5.	III	III	II	III	III
6.	II	II	II	II	II

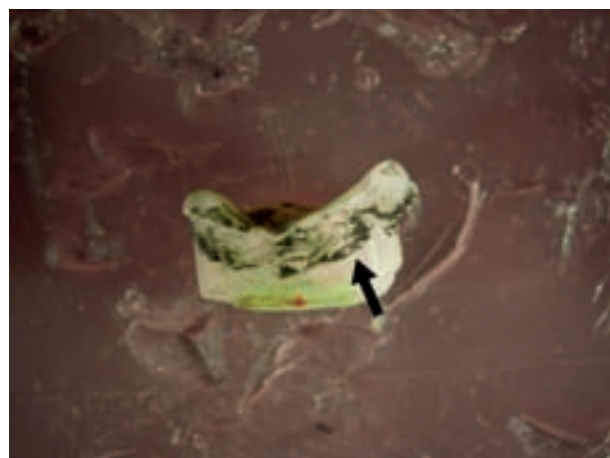


**Slika 3.** Rezultati mjerenja propusnosti konstrukcijom za prijenos tekućine (rezultati u µL)

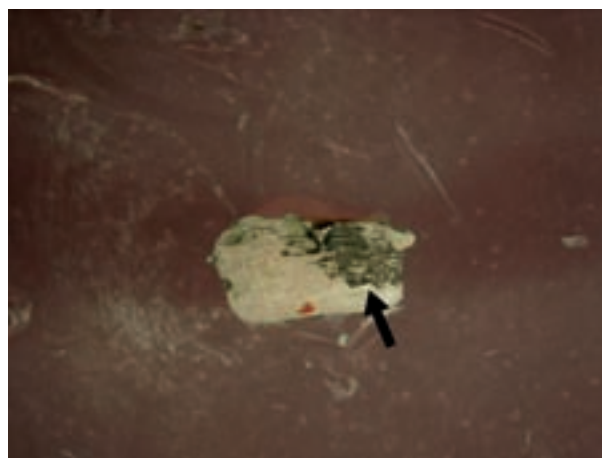
**Figure 3** Results in microleakage measured by fluid transport model (results are shown in µL)



**Slika 4.** Dubina prodora boje (prodor boje I)  
**Figure 4** Dye penetration depth (dye penetration I)



**Slika 5.** Dubina prodora boje (prodor boje II)  
**Figure 5** Dye penetration depth (dye penetration II)



**Slika 6.** Dubina prodora boje (prodor boje III)  
**Figure 6** Dye penetration depth (dye penetration III)

## Rasprava

Ispitivanja mikropropusnosti kompozitnih ispuna adheriranih pomoću adhezijskih sustava temelje se na kvantitativnim podacima o propuštanju (1, 2). Skupljanjem podataka o mikropropuštanju zaključuje se o kvaliteti pojedinih restaurativnih materijala, što ta istraživanja čini esencijalnim za proizvođače i korisnike. Međusobnim kombiniranjem više postupaka očitavanja rubnog propuštanja točnije se mogu donositi zaključci o kvaliteti kombiniranja pojedinih adhezijskih sustava i kompozitnih materijala (1, 2). Ovo istraživanje nastalo je na temelju prijašnjih istraživanja (1, 2) u kojima su kombinirana dva različita načina očitavanja rubnog propuštanja kako bi se dobila jasnija slika o kvaliteti adhezije restaurativnih sustava (1, 2).

Za ispitivanje rubne propusnosti koristila se tehnika prodora boje. U stereomikroskopskom pregledu vide se kontaktne površine ispuna i zida kaviteta obojene bojom, pa se tako može odrediti dubina njezina prodora. U ovom je istraživanju mikropropusnost ispitana i konstrukcijom za prijenos tekućine. Takav je način prikladan jer omogućuje dugoročno praćenje stupnja vezivanja materijala za razliku od ranijih istraživanja (1, 4, 16). Tijekom mjerenja uzorci se ne oštećuju, kao kod "trodimenzionalnih postupka" i tehnike prodora boje, kada se uzorci - kako bi se očitali rezultati - moraju demineralizirati (1, 2, 16). Iz tog je razloga postupak mjerenja konstrukcijom za prijenos tekućine obavljen na istim uzorcima prije ispitivanja prodorom kontrastnog sredstva. Dva su kvantitativna postupka istraživanja obavljena na istim uzorcima s istim ciljem - da bi se sa što većom vjerodostojnošću, na temelju kvantitativnih podataka o rubnom prijanjanju, moglo govoriti o kvaliteti samih adhezijskih sustava.

Kako su neka istraživanja pokazala da pojednostavljuvanje kliničkog postupka smanjuje kvalitetu vezivanja (17-19), ovim se istraživanjem to željelo provjeriti kod samojetkajućih caklinsko-dentinskih adhezijskih sustava. Tako su se rabila dva samojetkajuća adhezijska sustava - dvofazni AdheSE i jednofazni Xenon III. U oba načina provjere rubnog propuštanja ono je i ustanovljeno. I u jednom i drugom načinu mjerenja zapažena je nešto veća propusnost kada se koristio adhezijski sustav Xenon III, iako je ta razlika bila statistički znatna samo za ispitivanje tehnikom prodora boje. Na grafičkom prikazu rezultata dobivenih konstrukcijom za prijenos tekućine (Slika 3.) veći je raspon srednjih vrijednosti rubnog propuštanja u skupini u kojoj je rabljen Xenon III, nego u onoj u kojoj se koristio AdheSE.

## Discussion

Composite fillings and adhesive systems microleakage measurements are based on quantitative data on leakage (1, 2). The gathered data on microleakage can contribute to the conclusions about the quality of certain restorative materials, making such research essential for manufacturers and users. By combining different microleakage measurements techniques the conclusions concerning the quality of certain adhesive systems and composite materials may be more reliable (1, 2). This research was based on earlier articles (1, 2) where two different microleakage measurements techniques were combined with the purpose of clarifying the data on quality of restorative materials adhesion (1, 2).

A dye penetration technique was used to detect marginal leakage. The dyed restoration surfaces can be visualized using a stereomicroscope; this enables determination of the depth of dye penetration. Additionally, in this study the microleakage was determined using a fluid transport model. This technique of microleakage measurement enables follow-up of the degree of material bonding, different from earlier research (1, 4, 16). During this procedure the specimens are not damaged, as they are in three-dimensional procedures and dye penetration technique, where specimens need to be demineralized in order to observe the results (1, 2, 16). Due to that fact, the measurement by the fluid transport model was performed before the dye penetration technique measurement, on the same samples. Two quantitative procedures were performed on the same samples with the same aim - to be able to discuss the quality of adhesive systems with increased credibility.

Due to the fact that some studies have shown that the simplification of clinical procedure decreases the bonding quality (17-19), this study was performed in order to test this hypothesis with the self-etching adhesive systems. Therefore, we used two different self-etch adhesive systems, two-step AdheSE and single-step Xenon III. Both methods in this study confirmed marginal leakage, greater when Xenon III was used, although the difference was statistically significant only with the dye penetration technique. The results measured by fluid transport model (Figure 3) showed higher range of mean values in group II using Xenon III, than in group I using AdheSE. It can be concluded that this study determined better marginal quality of two-step self-etch adhesive systems when compared to one-step self-etch systems which is in accordance with the results of Frankenberg et al. (18).

Može se reći da je ovim istraživanjem ustanovljeno bolje rubno prijanjanje dvofaznih samojetkajućih sustava nego jednofaznih, što se podudara s istraživanjima Frankenbergera i suradnika (18).

Adhezijski sustavi u kojima se najprije jetka, pa se odvojeno primjenjuju primer i bond, pokazuju bolje rezultate u propusnosti i snazi vezivanja od adhezijskih sustava sa sjedinjenim komponentama, od kojih su najučinkovitiji dvofazni samojetkajući sustavi (12, 17, 18, 20, 21). Peumans i suradnici (17) također ističu dvofazne samojetkajuće adhezijske sustave kao klinički pouzdanije od jednofaznih.

Ovim istraživanjem pokazana je mogućnost prodora mjehurića zraka pod utjecajem tlaka i, osim što upozorava na prodor intraoralne tekućine (medija) kroz rubnu pukotinu, može upućivati i na difundiranje intratubulusne tekućine kroz tubuluse nakon završenog adhezijskog postupka. To valja potvrditi daljnjim istraživanjima, na što nas navodi i činjenica da su rezultati ovog rada u skladu s rezultatima Frankenbergera i suradnika (18) te Taya i suradnika (19). Frankenberger i suradnici (18) dokazali su bolje rubno vezivanje klasičnih adhezijskih sustava u kojima se primer primjenjuje odvojeno, nego kod pojednostavljenih u kojima se sjedinjuju priming i bonding. Ističu i znatnu razliku između dvofaznih i jednofaznih samojetkajućih adheziva te smatraju da tomu najviše pridonosi nakupljanje vode između sloja adheziva i kompozita, što se slaže i s rezultatima istraživanja Taya i suradnika (19). Dokazali su da se pojednostavljeni adhezivi, posebice jednofazni samojetkajući, ponašaju kao polupropusna membrana nakon polimerizacije te je dokazana difuzija vode kroz sloj adheziva koja ostaje zarobljena uzduž sloja adheziva i kompozita i utječe na slabljenje veze adhezijskog sustava i restaurativnoga kompozitnog materijala (18, 19, 22, 23).

## Zaključci

Oba adhezijska sustava (AdheSE i Xeno III) ispitana u istraživanju pokazuju rubno propuštanje. Pomakom mjehurića zraka pomoću konstrukcije za prijenos tekućine, u rubnom propuštanju između dvaju adhezijskih sustava ( $p=0,7488$ ) nije ustanovljena statistički znatna razlika.

Rubno propuštanje, prikazano "trodimenzionalnim postupkom" tehnikom prodora boje, pokazalo je statistički znatno veći stupanj rubnog propuštanja prodora boje ( $p=0,002$ ) u skupini uzoraka u kojoj je rabljen adhezijski sustav Xeno III.

Potrebna su daljnja istraživanja kako bi se ispitao utjecaj stupnja rubnog propuštanja na kvalitetu caklinsko-dentinskog adhezijskog sustava.

Adhesive systems in which etching is performed prior to priming and bonding show better results of leakage and bond strength than adhesive systems with combined components, two-step self-etch adhesive systems being the most efficient (12, 17, 18, 20, 21). Peumans et al. (17) reported two-step self-etch adhesive systems to be clinically more reliable, while inefficient clinical performance was noted for the single-step self-etch adhesive systems.

This study showed the possibility of pressurized air bubble penetration, pointing out not only to the penetration of intraoral fluid through the marginal gap, but also to the diffusion of intratubular fluid through the tubules after the completion of adhesion process. This must be confirmed in further studies, indicated by the fact that our results are comparable to the results of Frankenberger et al. (18) and Tay et al. (19). Frankenberger et al. (18) showed better marginal quality of conventional adhesive systems with separate bonding agents when compared to the simplified ones that combine priming and bonding. They also stated that the significant difference between two-step and single-step self-etch systems is mostly attributed to water adsorption between adhesive layer and resin composite. That is comparable to the report by Tay et al. (19) who stated that simplified adhesives, the single-step self-etch systems in particular, after polymerization behave as semi-permeable membrane. They confirmed that water, diffusing through the adhesive layer, stays trapped along the adhesive-composite interface, decreasing bonding of adhesive systems and resin composites (18, 19, 20, 23).

## Conclusion

Both adhesive systems (AdheSE, Xeno III) tested show marginal leakage. Microleakage measurement by fluid transport model did not determine statistically significant difference ( $p=0,7488$ ) between two adhesive systems (AdheSE and Xeno III). Measuring marginal leakage using a "three-dimensional" method of dye penetration showed statistically significant higher degree of marginal leakage ( $p=0,002$ ) in the group of samples using Xeno III.

Further research, that will show the influence of the leakage on the quality of the adhesive system, is necessary.



**Abstract**

The purpose of this study was to evaluate microleakage of two different adhesive systems between resin composite restorations and cavity walls. The experiment was conducted on 12 intact human third molars, extracted for orthodontic reasons, sectioned at the cemento-enamel junction to provide crown segments. The cavities were prepared in the occlusal surfaces. The samples were divided into two groups, six samples in each. Restorations were made with the resin composite "Tetric Ceram" (Vivadent, Schaan, Liechtenstein) and two different self-etch adhesive systems: in one group two-step self-etch AdheSE (Vivadent, Schaan, Liechtenstein), and in the other single-step self-etch Xeno III (DeTrey, Konstanz, Germany). Microleakage was measured by two different methods, fluid transport model and dye penetration. The results using fluid transport model showed no statistically significant difference between two groups of samples ( $p=0,7488$ ). Marginal leakage measured using dye penetration technique method showed statistically significant higher degree of marginal leakage ( $p=0,002$ ) in the group where Xeno III was used.

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