

## ISPITIVANJE DEMINERALIZACIJE OKO KOMPOZITNIH RESTAURACIJA ZUBA

### EXAMINATION OF DEMINERALIZATION AROUND COMPOSITE RESTORATIONS OF TEETH

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#### Sažetak

*Cilj ovog rada bio je da se u in vitro uslovima proceni demineralizacija marginalne gleđi oko restauracija od kompozitnih smola nakon delovanja kariogenog rastvora.*

*Materijal i metod. Kao materijal u istraživanjima korišćeno je 56 ekstrahovanih humanih molara. Preparisani su kaviteti V klase sa ivicama u gleđi. Kaviteti su restaurisani različitim kompozitnim materijalima i odgovarajućim adhezivnim sistemima: (1) Single Bond/Z 250, (2) Prompt-L-Pop/Z 250, (3) Single Bond/ Filek flow i (4) Prompt-L-Pop/ Filek flow. Posle restaurativne procedure restauracije su izlagane demineralizaciji tokom 1 i 4 nedelje. Uzorci su držani u demineralizujućem- kariogenom rastvoru ( mlečna kiselina, pH 4.5, 0,1M) na 37°C ili u destilovanoj vodi (kontrolna grupa). Perimarginalna gleđ je ispitivana skening elektronskim mikroskopom (SEM). Širine demineralizovane zone i marginalne pukotine oko restauracija merene su cervikalno, okluzalno i na aproksimalnim stranama.*

*Rezultati. Dobijeni rezultati su pokazali da je gleđ blizu ivica kompozitnih restauracija bila jače oštećena dejstvom kiselog kariogenog rastvora i označavana je kao perimarginalna demineralizovana gleđna zona. Tipični ultrastrukturni znaci perimarginalne zone bili su erozija intraprizmatične gleđi, porozitet, naprsline i frakture. Periferna zona gleđi pokazivala je neznatna oštećenja pod dejstvom kariogenog rastvora. Nije nađena statistički značajna razlika u prosečnoj širini demineralizovanih perimarginalnih zona gleđi između restaurativnih materijala ( $p > 0,05$ ).*

*Zaključak. Oštećenje perimarginalne gleđne zone izazvano kariogenim rastvorom uočeno je oko svih kompozitnih ispuna i nije zavisilo od primenjenog adhezivnog postupka i restaurativnog materijala.*

**Ključne reči:** Gleđ, Kompozitne smole, Demineralizacija, Skening elektronska mikroskopija

#### Summary

*Objective. The aim of this study was to perform an in vitro evaluation of the demineralization on perimarginal enamel of composite resin restorations after storage in cariogenic solutions.*

*Method. The material used in the research were 56 extracted human molars. Class V cavities were prepared with margins in enamel. Cavities were restored with various composite materials and appropriate adhesive systems: (1) Single Bond/Z 250, (2) Prompt-L-Pop/Z 250, (3) Single Bond/Filek flow and (4) Prompt-L-Pop/Filek flow. After the restorative procedure, the restorations were submitted to demineralization during the first and the fourth week. Samples were stored in a demineralizing- cariogenic solution (lactic acid, pH 4.5, 0,1M) at 37°C or in distilled water (control group). The margins of restorations and perimarginal enamel were examined by scanning electron microscope (SEM). The width of demineralized zone and marginal fissures around restorations were measured at cervical, occlusal and approximal margin.*

*Results. The enamel close to composite restoration margins was strongly affected by acidic cariogenic solution and was marked as perimarginal demineralized enamel zone. The typical ultrastructural patterns of the perimarginal enamel zone were erosions of intraprismatic enamel, porosities, fissures and fractures. The peripheral zone showed only minimal alteration by cariogenic solution. There was no statistically significant difference ( $p < 0,05$ ) between restorative materials concerning the approximate width of demineralized perimarginal enamel zones.*

*Conclusion. Deterioration of perimarginal enamel zone caused by cariogenic solution could be seen around all composite fillings and did not depend on adhesive treatment and restorative materials.*

**Keywords:** Enamel, Composite resins, Demineralization, Scanning electron microscopy

#### Uvod

Morfologija gleđne površine blizu ivica kompozitnih restauracija je često povezana sa pojavom sekundarnog karijesa. Pukotine, naprsline i frakture se mogu uočiti uz gleđne ivice mnogih restauracija. Ova oštećenja oslabljuju integritet perimarginalne gleđi i uzrok su nastanka sekundarne demineralizacije. Istraživanja su pokazala da se gleđ blizu ivica restauracija (označena kao perimarginalna zona) jače oštećuje delovanjem kariogenog rastvora u odnosu na gleđ udaljeniju od restauracije. <sup>1</sup>

#### Introduction

The morphology of enamel surface close to the margins of the composite restorations is often connected to secondary caries. Gaps, fissures and fractures could be seen adjacent to the enamel margins of many restorations. These failures weaken the perimarginal enamel integrity and may be responsible for the formation of secondary demineralization. Previous studies have demonstrated that enamel adjacent to the margin of the restoration (defined as the perimarginal zone) was more severely affected by

Marginalna oštećenja gleđi su najčešće posledica stresova usled polimerizacione kontrakcije kompozita ali mogu biti izazvana i postupkom kondicioniranja kaviteta. Međutim, nagrizanje (kondicioniranje) tvrdih zubnih tkiva kiselinom povećava retenciju površinu koja učestvuje u vezivanju kompozita za gleđ i dentin, a ova mikroretencija u zubnom tkivu obezbeđuje dobru, klinički zadovoljavajuću adheziju.<sup>2,3</sup>

Danas se koriste dva protokola pripreme (kondicioniranja) kaviteta u sklopu dva adhezivna restaurativna sistema.

Adhezivni sistem sa totalnim nagrizanjem podrazumeva primenu fosforne kiseline za nagrizanje i gleđi i dentina, pri čemu se kiselina obavezno ispira vodom pa se postupak naziva „nagrizanje i ispiranje“ ili „etch and rinse“. Veruje se da ovaj sistem može da oslabi integritet perimarginalne gleđi ali i da dovede do jače demineralizacije duž ivica restauracije.<sup>4</sup>

Izveštaji o mogućoj postoperativnoj osetljivosti zuba nakon primene totalno nagrizajuće tehnike doveli su do uvođenja samonagrizajućih adheziva koji čuvaju integritet čepova debrisa unutar dentinskih kanalića i vezu ostvaruju preko modifikovanog razmaznog sloja..

Samonagrizajući adhezivni sistem ne uključuje nagrizanje fosforom kiselinom već se za kondicioniranje zidova kaviteta koristi samonagrizajući prajmer (hemijski blaža kiselina), koji se ne ispira. Samonagrizajući postupak je uz to i kraći i zato i klinički prihvatljiviji.<sup>2,5,6,7,8.</sup>

Cilj ovog rada je bio da se u in vitro uslovi- ma proceni uticaj kariogenog rastvora (mlečne kiseline) na gleđnu površinu oko kompozitnih restauracija, metodom SEM-a

## ***Materijal i metodi***

### ***Preparacije kaviteta***

Kao materijal u istraživanju poslužili su hu- mani treći molari mlađih pacijenata, ekstrahira- ni iz različitih razloga (ukupno 56 zuba). Do eksperimenta zubi su čuvani na 4C° u fiziološkom rastvoru. Pre preparacije kaviteta zubima su odsecani korenovi. Sa vestibular- ne strane svakog zuba napravljena je adheziv- na preparacija V klase ( 3mm x 2mm obima i 2mm dubine) primenom turbinskog kolenjaka i kruškastog dijamantskog svrdla. Svi kaviteti su preparisani iznad gleđno-cementne granice kako bi sve ivice kaviteta bile u gleđi. Rubovi kaviteta zakošavani su plamičastim, najfinijim dijamantskim svrdlom uz primenu mikroko- lenjaka i smanjene brzine rotacije svrdla.

cariogenic solution. In comparison to enamel distant from restoration. 1

Marginal enamel alterations are very of- ten the result of stress induced by a composite polymerizing contraction but could, also, be induced by the cavity conditioning treatment. However, etching (conditioning) of hard tooth tissues with acid enlarges the surface which takes part in bonding the composite to the enamel and dentine. The forming of micro retention in the tooth tissue by acid application provides a good, clinically satisfying adhesion.<sup>2,3</sup>

Today, two conditioning protocols for the preparation of cavity are used enclosed in two restorative adhesive systems.

Total etch adhesive system includes the ap- plication of a strong phosphoric acid for enamel and dentine etching, where the acid is rinsed away with water, and therefore the procedure is called “etch and rinse”. It is believed that this system can weaken the perimarginal enamel in- tegrity as well as cause a stronger demineraliza- tion along the restoration edges.

Reports on post-operative sensitivity of teeth after using the total etch technique have led to a greater popularity of self etch adhesives which keep the integrity of stopper debris in- side dental canals.

Self etch adhesive system does not include phosphoric acid etching- the cavity wall condi- tioning is done by a self etch primer, which is not rinsed away.

Moreover, the self etch treatment is shorter and has better clinical application.<sup>2, 5,6,7,8</sup>

The aim of this study was to perform an in vitro evaluation of the effects of cariogenic so- lution (lactic acid) on enamel surface around composite restoration.

## ***Materials and methods***

SEM study was performed at the Institute of Biochemical Research at the Faculty of Medi- cine, University of Nis.

### ***Cavity preparation***

We used human third molars of younger pa- tients extracted out of various reasons (total of 56 teeth) as the research material. Before the experiment, the teeth were stored in the physi- ological solution at 4°C. Prior to the cavity preparation, the roots were cut off. On each vestibular side of the teeth an adhesive Class V (3mm x 2mm scope and 2mm depth) prepa- ration was done with handpiece and a pear- shaped diamond bur. All cavities were prepared above the enamel-cement line so that all cavity

Uzorci zuba su potom podeljeni u 4 grupe, po 12 zuba za dva opservaciona perioda (48 zuba).

Osam zuba, po dva za svaki od četiri metoda restauracije, je služilo kao kontrola (zubi su nakon postavljanja ispuna držani u destilovanoj vodi).

### **Metodi restaurisanja zuba**

Uzorci prve dve grupe zuba su restaurisane hibridnim a druge dve tečnim kompozitom. Kaviteti I i III grupe zuba su pripremani totalnom tehnikom nagrizanja a II i IV grupe primenom samonagrizajućih prajmera.

*I grupa: Kiselina + Adper Single Bond 2 + Filtek Z250 (totalno nagrizajuća tehnika- total etch + hidrofилni adheziv + hibridni kompozit)*

*II grupa: Adper Prompt-L-Pop (APLP) + Filtek Z 250 (samonagrizajući- self etch adheziv + hibridni kompozit)*

*III grupa: Kiselina + Adper Single Bond 2 + Filtek Flow (Filtek Supreme XT Flowable) (totalno nagrizajuća tehnika + hidrofилni adheziv + tečni kompozit)*

*IV grupa: Adper Prompt-L-Pop (APLP) + Filtek Flow (Filtek Supreme XT Flowable) (samonagrizajući adheziv + tečni kompozit)*

Svi materijali su postavljeni po uputstvu proizvođača a kompozitni materijali su nanošeni u dva sloja i polimerizovani.

Vezujući agensi i kompoziti su fotopolimerizovani, sa prethodno testiranom, Visilux Command 2 lampom (3M) jačine 400 mW/cm<sup>2</sup>. Svaki sloj materijala osvetljavan je po 20 s.

Svi uzorci su polirani dijamantskim finirima uz, obavezno, hlađenje vodom, Sof-Lex diskovima različite finoće zrna i gumicama (3M ESPE)

### **Procedura u mlečnoj kiselini**

Uzorci četiri grupe zuba (po šest zuba za svaki metod restauracije) uranjani su u rastvor mlečne kiseline pH 4,5 0,1M, na 37°C, u tube od 20 ml. Po šest uzoraka zuba iz svake grupe, držano je u kariogenom rastvoru 1 nedelju, odnosno 4 nedelje. Rastvor mlečne kiseline je menjan svaka 24 časa. Kontrolni uzorci, po dva za svaku grupu (ukupno 8 zuba), držani su u destilovanoj vodi - pH 6,7 na 37°C, tokom 1 nedelje u tubama od 20ml. Posle ovog perioda

edges were in the enamel. The cavity rims were slanted with a flame-like, superfine diamond bur with the use of handpiece and a decreased speed of bur rotation.

The teeth specimens were divided into 4 groups: 12 teeth for two observation periods (48 teeth).

Eight teeth, two for each of 4 methods of restoration, served as control (these teeth were stored in deionized water after being filled).

### **Toot restoration methods**

Four restoration methods were used (four groups of teeth). The materials were placed according to manufacturers' instructions. The samples from the first two groups were restored with a hybrid, and the other two with a liquid composite. The first and third group cavities were prepared using the total etch procedure and the second and the fourth groups were prepared using the self etch procedure.

*1<sup>st</sup> group: Acid + Adper Single Bond 2 + Filtek Z250 (total etch + hydrophilic adhesive + hybrid composite)*

*2<sup>nd</sup> group: Adper Prompt-L-Pop (APLP) + Filtek Z250 (self etch adhesive + hybrid composite)*

*3<sup>rd</sup> group: Acid + Adper Single Bond 2 + Filtek Flow (Filtek Supreme XT Flowable) (total etch + Hydrophilic adhesive + liquid composite)*

*4<sup>th</sup> group: Adper Prompt-L-P (APLP) + Filtek Flow (Filtek Supreme XT Flowable) (self etch adhesive + liquid composite)*

All materials were applied following the manufacturer's instructions. Composite materials were put in two layers and polymerized.

The bonding agents and composites were photopolymerized, with a previously tested, Visilux Command 2 lamp (3M) at 400 mW/cm<sup>2</sup>. Each material layer was light-cured for 20s. All specimens were polished with diamond finishing Sof-Lex discs of different granule fineness and rubbers (3M ESPE), with obligatory water cooling.

### **Lactic acid procedure**

Tooth specimens from the four groups (six teeth per each restoration method) were immersed into 20ml tubes containing lactic acid solution whose pH value was 4.5 0.1 M at 37°C. Six specimen teeth from each group were immersed into the cariogenic solution for 1 and for 4 weeks (all together 48 teeth). The lactic acid solution was regularly replaced after

Tabela 1.

Materijali	Kiseli rastvor		Destilovana voda
	1 nedelja	4 nedelje	1 nedelja
S.Bond/ Z 250	n=6	n=6	n=2
APLP/ Z 250	n=6	n=6	n=2
S.Bond/ Filtek flow	n=6	n=6	n=2
APLP/ Filtek flow	n=6	n=6	n=2

Korišćeni materijali i broj uzoraka u svakoj grupi

Tabela 1.

Materials	Acid solution		Deionized water
	1 week	4 weeks	1 week
S.Bond/ Z 250	n=6	n=6	n=2
APLP/ Z 250	n=6	n=6	n=2
S.Bond/ Filtek flow	n=6	n=6	n=2
APLP/ Filtek flow	n=6	n=6	n=2

Used materials and the number of specimens in each group.

svaki uzorak je ispiran vodom i čuvan 24 časa u destilisanju vodi, pre SEM ispitivanja.

### **SEM analiza perimarginalne zone gleđi**

Za skening elektron mikroskopsko ispitivanje korišćen je SEM JEOL-JSM 530

Kvalitativna analiza perimarginalne gleđi vršena je korišćenjem metode Prati i sar.<sup>1</sup>

### **Merenje širine demineralizovane zone**

Širina demineralizovane zone merena je pomoću skalera na monitoru mikroskopa. Merenje je vršeno na četiri strane restauracije (mezijalnoj, distalnoj, okluzalnoj i gingivalnoj) i to na mestima najšireg pružanja zone. Na osnovu četiri izmerene vrednosti izračunavana je prosečna širina zone demineralizacije.

### **Statističke metode**

Poređenje vrednosti prosečnih širina demineralizovanih polja vršeno je jednostranom analizom varijanse (One-way ANOVA) i sledbenim Danetovim testom (Post-hoc Dunnett test).

### **Rezultati istraživanja**

Dobijeni rezultati prikazani su na slikama 1-5 i grafikonima 1 i 2.

24 hours. The control specimens, two for each group (all together 8 teeth), were stored in deionized water of pH value 6.7 at 37°C, in 20ml tubes. After 1 week, each specimen was rinsed with water and before SEM examination, it was stored in deionized water for 24 hours.

### **SEM analysis of the perimarginal enamel zone**

Scanning electron microscope SEM JEOL-JSM 530 was used for this study.

A qualitative analysis of composite adhesion and perimarginal enamel was performed according to Prati<sup>1</sup>

### **Measuring demineralized zone width**

The width of the demineralized zone was measured with scales on the microscope screen. It was performed on four sides of the restoration (mesial, distal, occlusal and gingival) in places of the widest zone spreading. The average zone width was calculated according to four measured values.

### **Statistical methods**

The following statistical parameters were presented: average values (X<sub>av</sub>), standard deviations (SD), median (Med), minimum (Min) and maximum (Max). Comparison of average width of demineralized areas was performed by one way analysis of variance (One-way ANOVA) followed by Post-hoc Dunnett test. The data analysis was performed using SPS 10.0 statistical program.

## SEM nalaz

### Morfologija marginalne zone gleđi

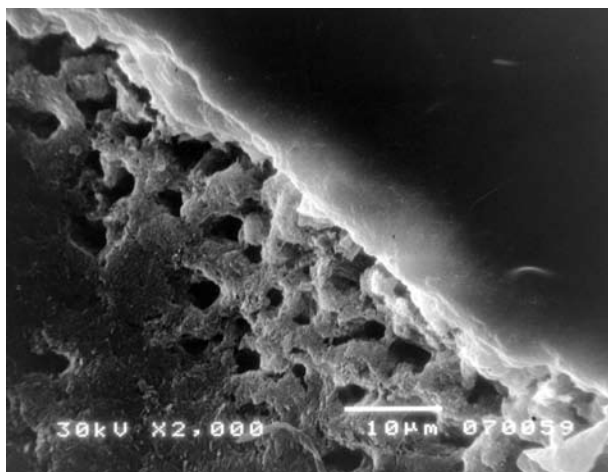
SEM analizom je utvrđeno jače rastvaranje gleđi oko ivica ispunja pod dejstvom mlečne kiseline, kod svih restauracija bez obzira na metod postavljanja kompozita. Udaljenija gleđ od ivica restauracija je bila sa neznatnim promenama. Polje gleđi sa izraženijom demineralizacijom označavano je kao demineralizovano perimarginalno polje ili demineralizovana zona gleđi. Ultrastrukturne promene ove ivične zone gleđi bile su izraženije nakon dužeg delovanja mlečne kiseline. Tako je, nakon delovanja kiselog rastvora u trajanju od četiri nedelje, na perimarginalnoj demineralizovanoj površini gleđi često uočavana konfiguracija pčelinjeg saća, što je bilo posledica rastvaranja gleđnih prizmi. To se jasno uočava oko restauracija postavljenih i po totalno nagrizujućem kao i po samonagrizujućem postupku. (Slike 1,2,3)

Tela gleđnih prizmi su bila jako oštećena i često kompletno uklonjena. (Slika 4.)

Često je gubitak tkiva između i unutar gleđnih prizmi davao porozni izgled perimarginalnoj gleđnoj zoni.

Pored jače demineralizacije ivične zone gleđi blizu kompozitnih ispunja, često su uočavane erozije i frakture gleđi u perimarginalnoj zoni. (Slika 5)

Nisu pronađene značajne razlike ultrastrukture površine perimarginalne gleđi povezane sa vrstom kompozita (hibridni ili tečni) kao i sa adhezivnim postupkom primenjenim kod postavljanja ispunja.



Sl. 1. SEM fotomikrografija spoja ispun/gleđ posle nedelju dana izlaganja dejstvu mlečne kiseline. Širina demineralizacione gleđne zone je oko 20 mikrona. Single Bond/Z250 (x1000)

Picture 1. SEM photomicrography of enamel/restoration connection after 1 week of applying the lactic acid. The width of the demineralized enamel zone is about 20 microns. Single Bond/Z250 (x1000)

## Study results

The achieved results were shown in pictures 1-5 and charts 1 and 2.

## SEM results

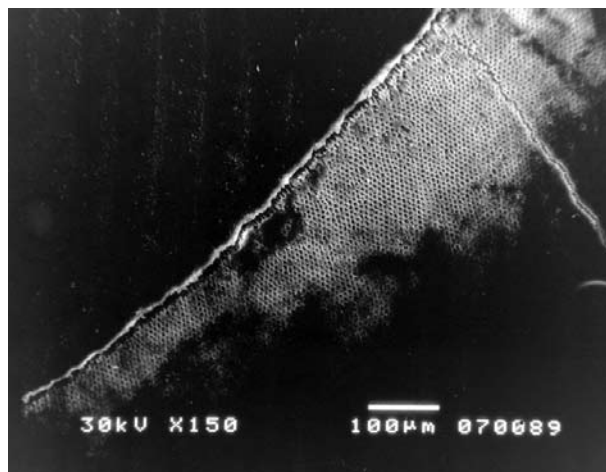
### Marginal enamel zone morphology

SEM analysis showed increased enamel dissolving around the margins of the fillings, after lactic acid effect, with all restorative methods. There were little changes on more distant enamel. The enamel area with a distinct demineralization was marked as a demineralized perimarginal area or demineralized enamel zone. Ultrastructural changes of this marginal enamel were greater after a longer exposure to lactic acid. The typical honeycomb configuration was observed on perimarginal demineralized surface after exposure to acidic solution for four weeks which was a consequence of dissolution of enamel prisms. This is clearly observed around both total etch and self-etch restorations. (Pictures 1,2,3)

The enamel prism core was severely damaged and sometimes completely removed. (Picture 4)

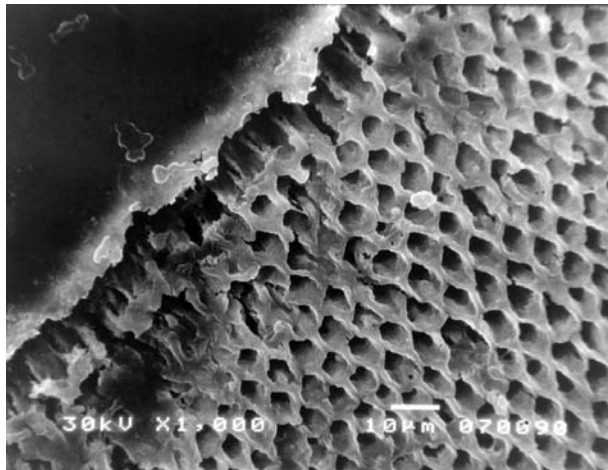
Loss of tissue between and within enamel prisms gave the perimarginal enamel zone a porous appearance.

Besides stronger demineralization of marginal enamel zones near composite fillings, there were often erosions and enamel fractures in the perimarginal zone (Picture 5)



Sl. 2. SEM fotomikrografija perimarginalne gleđne zone nakon 4 nedelje izlaganja dejstvu mlečne kiseline. Širina demineralizovane gleđi je oko 200 mikrona. APLP/Filtec flow (x150)

Picture 2. SEM photomicrography of the perimarginal enamel zone after 4 weeks of applying the lactic acid. The width of the demineralized zone is about 200 microns. . APLP/Filtec flow (x150)



Sl.3. Uvećanje prethodne slike. Omotači i tela gleđnih prizmi su kompletno uklonjeni dok je interprizmatska gleđ na mestu. Demineralizovana gleđ je u obliku pčelinjeg saća. Uočava se i marginalna pukotina između kompozita i gleđi. APLP/Filtec flow. SEM foto mikrofografija x1000

Picture 3. Higher magnification of previous picture. The core and body of enamel prisms completely removed but interprismatic enamel was still in place. The honeycomb structure of demineralized enamel was evident. A marginal gap could be seen between the composite and enamel. APLP/Filtec flow.

SEM photo micrograph x1000

### Širina demineralizovane zone

Najveća izmerena širina demineralizovane perimarginalne zone, oko kompozitnih ispuna nakon 1 nedelje delovanja kiselog rastvora, iznosila je 20 μm i vidana je mestimično po obodu ispuna (S.Bond/ Z 250)

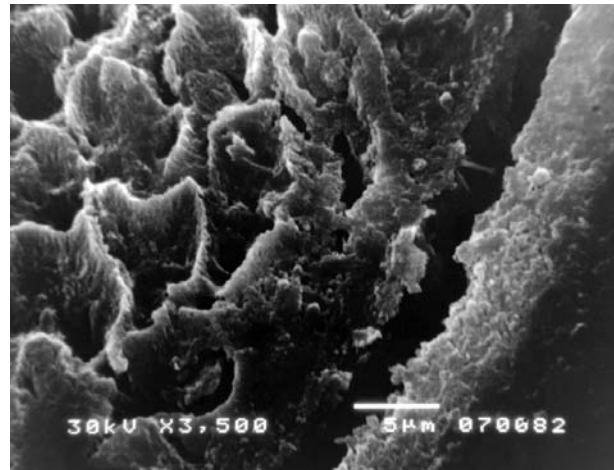
Prosečne širine demineralizovanih zona se nisu značajno razlikovale između grupa, za ovaj opservacioni period.

Nakon delovanja kariogenog rastvora u trajanju od 4 nedelje utvrđeno je postojanje šireg polja još jače demineralizovane gleđi, oko svih kompozitnih restauracija. Najveća izmerena širina demineralizovane perimarginalne zone, nakon 4 nedelje, iznosila je 500 μm (APLP/ Filtec flow).

Iako je prosečna širina demineralizovane zone iznosila u prvoj grupi 84.2±49.7 μm a u četvrtoj 100.6±62.9 μm, nisu utvrđene statistički značajne razlike među grupama, u ispitivanom uzorku i za period delovanja kiselog rastvora od 4 nedelje.

### Diskusija

Nagrizanje gleđne površine zuba rastvorima različitih kiselina, predstavlja postupak kojim se povećava aktivna površina za vezu sa kompozitnim materijalom i značajno smanjuje marginalna mikropukotina oko restauracije. Preparacija kao i koncentracija i tip kiseline imaju značajnu ulogu u obezbeđivanju dobre adhezije između gleđi i kompozitnih materijala. Najče-



Sl. 4. SEM foto izgled perimarginalne gleđi, nakon 4 nedelje u kariogenoj soluciji. Prizme nisu prisutne na površini gleđi dok se interprizmatska supstanca jasno uočava. APLP/Filtec flow. (x3500)

Picture 4. SEM photo outlook of the perimarginal enamel after 4 weeks in the cariogenic solution. The prisms cannot be seen on the enamel surface while interprismatic substance is clearly defined. APLP/Filtec flow. (x3500).

There were no significant differences in the ultrastructure of the surface of perimarginal enamel connected to the type of composite (hybrid or liquid) or the adhesive techniques used for the application of fillings.

### Demineralized zone width

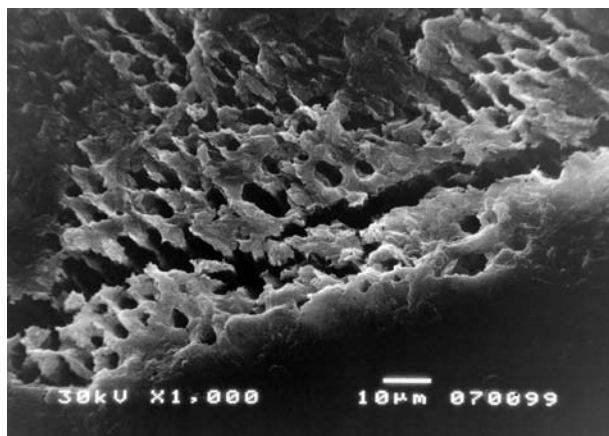
The largest measured demineralized perimarginal zone width, around composite fillings, after 1 week in acidic solution, was 20μm and it was sporadically seen around the edge of the filling (S. Bond/Z 250). (Chart 1)

During this observation period, the average demineralized zone widths did not differ significantly among groups.

After a 4-week cariogenic solution application, an even wider area of demineralized enamel was found, around all composite restorations. The biggest demineralized perimarginal zone width, after 4 weeks, was 500μm (APLP/Filtec flow). (Chart 2)

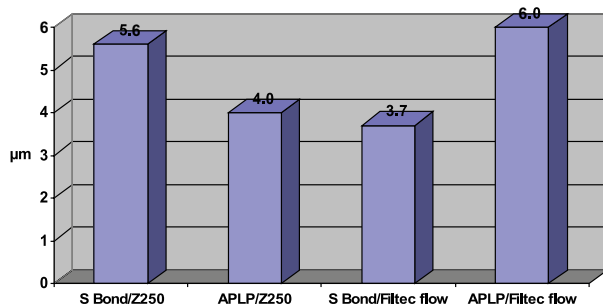
Although average width was 84.2±49.7 μm in the first group and 100.6±62.9 μm in the fourth group, no statistically significant differences between groups were observed for the tested sample as well as for the four week exposure to acidic solution.

Approximate width of demineralized zone is the biggest with the fourth (IV) method of restoration but there are no statistically important differences among the applied methods, in the specimen. Approximate width of demineralized zones in the first group, total erosion (84.2±49.7μm) and the fourth group — self erosion (100.6±62.9μm) do not differ statistically.



Sl. 5. SEM fotomikrografija nakon 4 nedelje dejstva kiselog rastvora. Vide se frakture i krhotine perimarginalne gleđi. Single Bond/Z250 (x1000).

Picture 5. SEM photomicrography after 4 weeks of acid solution effect. You can see fractures and fissures along the perimarginal enamel. Single Bond/Z250 (x1000).



Grafikon 1. Prosečna širina demineralizovanih zona (µm) kod testiranih grupa posle 1 nedelje; Prosečne širine demineralizacionih polja se ne razlikuju značajno između grupa.

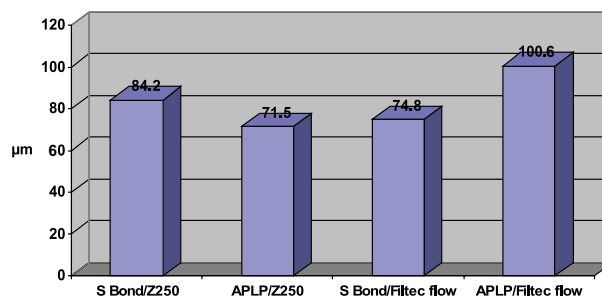
Chart 1. Approximate width of demineralized zones (µm) with tested groups after 1 week; Approximate width of demineralized fields do not significantly differ among the groups.

šće korišćene su fosforna (37%, 36% i 35% rastvor) i limunska kiselina.<sup>7,16,19,20,21,25,27</sup>

Postupkom nagrizanja gleđi povećava se površina za vezu i povećava mogućnost vlaženja površine gleđi, a samim tim i kvalitet adhezije. Na uspešnost nagrizanja utiču: orijentacija gleđnih prizmi prema površinskoj ravni, kiselina i njena koncentracija i vreme nagrizanja.

Sveže nagrižena gleđ koja ostaje nepokrivena ispunom posle završene restauracije je podložnija sekundarnoj demineralizaciji i prebojavanju. Zbog toga se i preporučuje lokalna fluorizacija nagriženih zona i dnevna primena fluorid gela, dve nedelje posle postavljanja kompozitnog ispuna. Ipak, pošto je utvrđeno da je za stvarnu remineralizaciju potrebno dva i više meseci, neophodno je da se prilikom nagrizanja obuhvate samo one površine koje će biti prekrivene ispunom.<sup>7</sup>

Otpornost ivične gleđi oko kompozitnih restauracija, na rastvaranje u kariogenim rastvori-



Grafikon 2. Prosečna širina demineralizovanih zona (µm) kod testiranih grupa posle 4 nedelje; Prosečna širina demineralizovane zone je najveća kod IV metoda restauracije ali nema statistički značajnih razlika između primenjenih metoda, u ispitivanom uzorku. Prosečne širine demineralizovanih zona u prvoj grupi, totalno nagrizanje (84.2±49.7µm) i četvrtoj grupi – samo nagrizanje (100.6±62.9µm) se ne razlikuju statistički značajno.

Chart 2. Approximate width of demineralized zones (µm) with tested groups after weeks; Approximate width of demineralized zone is the biggest with the fourth (IV) method of restoration but there are no statistically important differences among the applied methods, in the specimen. Approximate width of demineralized zones in the first group, total erosion (84.2±49.7µm) and the fourth group – self erosion (100.6±62.9µm) do not differ statistically.

## Discussion

Erosion of enamel surface of the teeth caused by solutions of different acids represents a procedure used to obtain a larger active area for the bonding of composite material and to reduce the micro fissure around restoration. The preparation as well as both the concentration and type of acid have a significant role in providing a solid adhesion between the enamel and composite materials. The most frequently used are phosphoric acid (37%, 36% and 35% solution) and citric acid.<sup>7,16,19,20,21,25,27</sup>

The procedure of enamel erosion increases the bonding surface and the possibility of moisturizing the enamel surface whereby the quality of adhesion is improved. The effectiveness of erosion is influenced by the following: orientation of enamel prisms on the surface plane, type of acid and its concentration and etching time. Freshly etched enamel which is not covered with filling after a restoration is more prone to secondary demineralization and coloration. Therefore, local fluorization of etched zones and daily application of fluoride gel is recommended during a period of two weeks following composite fillings. However, as it has been confirmed that remineralization takes two or more months, it is necessary that etching includes only the areas that will be covered by filling.<sup>7</sup>

The resistance of marginal enamel around composite restorations to dissolving in cariogenic solutions depends, among other things,

ma zavisi, pored ostalog, od načina nagrizanja i adhezivne tehnike, tokom restaurativne procedure. <sup>1,13,14</sup>

Nakon delovanja kariogenog rastvora (mlečne kiseline), SEM ispitivanje je jasno pokazalo jače rastvaranje perimarginalne gleđi i jaku eroziju površine gleđi uz kompozitne ispune. Gledna površina, udaljenija od ivica restauracija, bila je sa manje izraženom demineralizacijom.

Jača demineralizacija gleđi oko restauracija, u odnosu na udaljeniju gleđ može biti posledica različitih faktora: ne sprovođenja principa preparacije adhezivnih kaviteta; nepravilnog kondicioniranja kaviteta; nepravilne tehnike unošenja adheziva i kompozita; prisutnih pukotina, fraktura ili ranije demineralizacije gleđi; nepravilnog finiranja i poliranja ispuna. <sup>4,5</sup>

Primena adhezivnih kompozitnih sistema menja ultrastrukturu perimarginalne gleđi. Agresivni sistemi, koji sadrže fosforu kiselinu mogu da u većij meri oslabe integritet perimarginalne gleđi (primarna demineralizacija) i dovedu do njene jače demineralizacije (sekundarna) nakon postavljanja ispuna i delovanja kariogenih rastvora.

Novi način vezivanja za gleđ i dentin, bez primene fosforne kiseline podrazumeva korišćenje manje kiselih rastvora u vidu samo-nagrizajućih prajmera. <sup>2,3,22,23,24,26</sup>

Adper Prompt- L- Pop je samonagrizajući adheziv (sve u jednom „all in one“) jer sadrži dve tečnosti koje se nanose istovremeno na zubni supstrat. U jednoj tečnosti su metakrilatni fosfati a druga sadrži, pored ostalog, i fluoride. Neposredno pre nanošenja, dve tečnosti se pomešaju u jedinstven blago kiseli rastvor. Adper Prompt dovodi do blažeg nagrizanja gleđnih prizmi i obezbeđuje slabiju retenciju kompozita zbog plićeg prodora u gleđ i to samo u formi interkristalne infiltracije smole. Adheziv može biti i mestimično odvojen od hibridnog sloja gleđne i dentinske površine uz formiranje pukotine između restauracije i zubnog tkiva. <sup>3,2</sup>

Nasuprot tome, utvrđeno je znatno dublje nagrizanje gleđne površine primenom fosforne kiseline uz obezbeđivanje jače retencije kompozita. Fosforna kiselina formira, kako pliću interkristalnu poroznost tako i dublju interprizmatsku retenciju koja omogućava dublju infiltraciju adhezivne smole u gleđ. <sup>2,3,4,5,9,10,11,12,15,18</sup>

Procedura demineralizacije in vitro (primenom kariogenog rastvora u formi mlečne kiseline), korišćena u ovom proučavanju, dovela je do erozije gleđne površine posebno duž ivica restauracija

on the type of etching and adhesive technique used during restoration. <sup>1,13,14</sup>

After lactic acid effect, the SEM examination clearly showed perimarginal enamel zones with strong erosion. There was evidence of a stronger dissolving of the marginal enamel area in an acid solution, in comparison to the rest of the enamel surface.

A more severe demineralization, compared to more distant enamel, can be a consequence of various factors: not carrying out the principles of adhesive cavity preparation; improper cavity conditioning; improper technique of adhesive and composite application; presence of marginal enamel gaps and fractures or previous enamel demineralization; improper finishing and filling polishing. <sup>4,5</sup>

The application of adhesive composite systems can significantly alter the perimarginal enamel ultrastructure. Aggressive systems which include phosphoric acid may affect perimarginal enamel integrity (primary demineralization) and cause stronger (secondary) demineralization around fillings after acting of cariogenic solutions.

A new way of bonding to the enamel and dentine, without applying the phosphoric acid, recommended by manufacturers is the use of less acidic solutions in form of self etch adhesives. <sup>2,3,22,23,24,26</sup>

Adper Prompt-L-Pop is a self etch adhesive (“all in one”) because it contains two liquids which are simultaneously applied to the tooth substrate. One liquid contains metacrylic phosphates whereas the other, among other things, contains fluorides. Before the application, two liquids blend into a single mildly acidic solution.

Adper Prompt (self etch two component system) leads to a mild enamel prisms etching and provides a mild retention of composites due to a shallow penetration into the enamel in the form of inter crystal infiltration of resin. The adhesive may be sporadically detached from the hybrid layer of enamel and dentine surface creating gaps between the restoration and tooth tissue. <sup>3,2</sup>

Opposite to this, a significant enamel prisms etching was identified after the phosphoric acid application providing stronger composite retention. The phosphoric acid forms, not only shallow intercrystal porosity but also interprismatic retention which enables a deeper infiltration of adhesive resin into enamel. <sup>2,3,4,5,9,10,11,12,15,18</sup>

In vitro demineralization (the use of cariogenic solution in the form of lactic acid), used in this study, led to early enamel surface erosion, particularly along the restoration edges.



Demineralizacija i poroznost gleđi oko ivica restauracija, bile su izraženije nakon delovanja kariogenog rastvora nakon četiri nedelje u odnosu na kraće izlaganje dejstvu rastvora, u trajanju od jedne nedelje.

Razlike u širini demineralizovanih ivičnih zona gleđi, u odnosu na korišćene opservacione periode su značajne. Značajno je veća širina polja demineralizacije kod dužeg delovanja kiselog rastvora.

Nisu utvrđene statistički značajne razlike u širini demineralizovanih zona u zavisnosti od adhezivnog postupka i restaurativnih materijala, u okviru istog opservacionog perioda.

I pored korišćenja jače fosforne kiseline kod totalno nagrizajućeg adhezivnog postupka (I i III grupa ispuna) pronađena je manja prosečna širina zone demineralizacije ivične gleđi oko kompozitnih ispuna, u poređenju sa samonagrizajućim postupkom. To se može objasniti boljim kvalitetom veze kompozita za gleđ u odnosu na vezu koju obezbeđuju samo nagrizajući prajmeri

Kod samo nagrizajućeg adhezivnog postupka (II i IV grupa ispuna) usled formiranja slabije retencije kompozita za gleđ, češće je uočavana mikropukotina oko ispuna koja se i može smatrati glavnim putem prodora mlečne kiseline duž zidova ispuna. Zato je utvrđena i šira zona demineralizacije oko kompozitnih plombi postavljenih pomoću samonagrizajućih prajmera.

Ipak, nije utvrđena statistički značajna razlika u širini ivičnih demineralizacija gleđi a vezano za adhezivni postupak, na ispitivanom uzorku. Samo nagrizajući postupak je prihvatljiviji za svakodnevnu kliničku praksu zato što je kraći a kiseli prajmer kao blaža kiselina često sadrži i fluoride. Zato treba ova istraživanja ponoviti na većem broju uzoraka, sa različitim robnim markama adheziva i kompozita i sa dužim opservacionim periodom.

Ultrastrukturapovršineperimarginalnegleđi, utvrđena u ovom istraživanju, je komparabilna sa prethodnim istraživanjima demineralizacije izazvane ranim karijesnim procesom.<sup>17, 26.</sup>

## ZAKLJUČAK

1 Demineralizacija perimarginalne gleđne zone, izazvana kariogenim rastvorom uočena je oko svih kompozitnih ispuna.

2. Morfologija i širina perimarginalne gleđne zone nisu zavisile od adhezivnog restaurativnog postupka.

Demineralization and enamel porosity around restoration margins were more evident after a four-week exposure to cariogenic solution, as opposed to a shorter, one week, period of exposure to the solution.

The differences in the width of demineralized marginal enamel zones, compared to the used observation periods, are significant. The width of demineralization field is significantly greater with longer exposure to acidic solution.

There were no statistically significant differences in the width of demineralized zones depending on adhesive procedure and restoration materials within the same observation period.

Despite using a stronger phosphoric acid in total etching adhesive procedure (1<sup>st</sup> and 3<sup>rd</sup> group of fillings), there was a smaller average width of marginal enamel demineralization zone around composite fillings, compared to self etch procedure. This can be explained by a better binding quality between composite and enamel, compared to the binding provided by etching primers.

Due to formation of weaker retention of composite to enamel in self etch adhesive procedure (2<sup>nd</sup> and 4<sup>th</sup> group), there were more frequent micro-fissures around the fillings which may be considered to be the major penetration route of lactic acid along fillings walls. Therefore, a wider zone of demineralization was determined around composite fillings applied by the use of self etch primers.

However, there was no statistically significant difference in the width of marginal enamel demineralization in relation to adhesive procedure on a tested sample. Self etch procedure is more acceptable in everyday clinical practice because it is shorter and the acidic primer used as mild acid often contains fluorides. Therefore, the researches should be repeated on a greater number of samples, using different brands of adhesives and composites and for longer observation periods.

The ultrastructure of perimarginal enamel surface, determined in this research, is compatible with previous studies of demineralization caused by early caries process.<sup>17, 26</sup>

## CONCLUSION

1. Demineralization of perimarginal enamel zone influenced by cariogenic solution was observed around all composite fillings.

2. Morphology and perimarginal enamel zone width did not depend on the adhesive restorative procedure.

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