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Procjena mikropropusnosti Zirconomera®: staklenoionomernog cementa ojačanog cirkonijevim oksidom

Evaluation of Microleakage in Zirconomer®: A Zirconia Reinforced Glass Ionomer Cement

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Sažetak
Cilj: Procijeniti mikropropusnost četiriju materijala za direktne restauracije. **Materijali i metode:** Odabrano je šesnaest govеđih sjekutića i nasumično su podijeljeni u četiri skupine: skupina I – Zirconomer, skupina II – Ketac™ Silver, skupina III – Filtek™ Z500 (kompozit), skupina IV – Dispersalloy® (amalgam). Za svaki materijal preparirano je sedam aproksimalnih (mezijalnih i distalnih) kaviteta koji su zatim restaurirani. Svi uzorci bili su pohranjeni 24 sata u destiliranoj vodi na 37° C, a zatim su podvrgnuti termocikličkom procesu na temperaturama od 5 i 55 °C. Nakon toga uronjeni su u 0,5-postotnu otopinu metilenskog modrila tijekom 24 sata. Svaki ispunjeni kavitet prerezan je kroz sredinu restauracije, a zatim je proučavan pod stereomikroskopom kako bi se procijenilo rubno propuštanje. Dobiveni rezultati mikropropusnoti statistički su analizirani. **Rezultati:** Najveća srednja vrijednost propuštanja zabilježena je u skupini II – Ketac™ Silver, nakon čega je slijedila skupina I – Zirconomer i skupina III – Filtek™ Z500 (kompozit). Najniža srednja vrijednost prodiranja boje ustanovljena je u skupini IV – Dispersalloy® (amalgam). Statistički su postojale značajne razlike između Zirconomera i skupina KetacTM Silver i amalgama, a nije bilo značajne razlike između Zirconomera i kompozita. Sve ispitane skupine pokazale su statistički značajne razlike u usporedbi s amalgamom. **Zaključak:** Rubna propusnost utvrđena je za sve restaurativne materijale. Potrebna su daljnja klinička istraživanja.

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Ključne riječi
stakleno-ionomerni cementi; cermet cementi; rubno prijanjanje materijala za punjenje; amalgam

Uvod

Mikropropusnost je fenomen u restaurativnoj stomatologiji koji nastaje zbog difuzije bakterija, tekućina, ostataka hrane te drugih iona i molekula duž spoja ispuna i zuba (1). Uzrokuje rekurentni karijes, diskoloracije, propadanje ispuna i/ili patologiju pulpe (1, 2). Zato su kontrola i sprječavanje rubnog mikropropuštanja itekako važni u suvremenoj restaurativnoj stomatologiji.

U istraživanjima su korištene različite metode *in vitro* ispitivanja mikropropusnosti. One uključuju penetraciju boje, filtraciju tekućina (3, 4), električnu vodljivost (5), aktivaciju neutrona (6), radioizotopni postupak (7) itd. No najčešće korištena metoda je uporaba obojenih agensa ili kemijskih tragova koji mogu lako prodrijeti u mikropukotine na spoju ispuna i zuba (8, 9).

Dobro je dokumentirano da su najčešći čimbenici koji utječu na integritet veze ispuna i zuba polimerizacijsko skupljanje, toplinska ekspanzija, svojstva vezivnog sredstva, hidrofilna priroda monomera, manipulacije i rukovanje materijalima (10, 11). Na nastanak mikropropusnosti mogu utjecati i drugi čimbenici kao što su oblik kaviteta, njegov

Introduction

Microleakage is a phenomenon in operative dentistry resulting from diffusion of bacteria, fluids, food debris, other ions and molecules along the tooth-restoration interfaces (1). It causes recurrent caries, discoloration, restorative failure and/or pulpal pathology (1, 2). Therefore, controlling and eliminating the marginal leakage is an important goal of modern restorative dentistry.

Studies have reported various methods for in vitro investigation of microleakage. These include dye penetration, fluid filtration (3, 4), electrical conductivity (5), neutron activation method (6), radioisotope method (7) and so on. The most commonly used method however, is by using coloured dye agents or chemical traces which are able to penetrate easily into the micro gaps between the tooth-restoration interfaces (8, 9).

It is well documented that the most common factors affecting the integrity of the tooth-restoration interface are polymerisation shrinkage, thermal expansion, properties of bonding agent, hydrophilic nature of the monomer, manipulations and handling of investigated materials (10, 11). Forpoložaj i dimenzije (10, 12). Nadalje, nastanku mikropropusnosti mogu pridonijeti i čimbenici kao što su struktura zuba, permeabilnost dentina i orijentacija dentinskih tubula (10).

Konvencionalni staklenoionomerni cement (SIC) razvili su Kent i Wilson 1960., a nastao je reakcijom lužine i kiseline (13, 14). Danas je SIC materijal izbora za različite indikacije u stomatologiji, uključujući brtvljenje korijenskog kanala, lijepljenje ortodontskih bravica, podloge u dubokim kavitetima, te pečaćenje fisura i ispune (15). Ti materijali imaju jedinstvena svojstva kao što su oslobađanje i apsorpcija fluorida, kemijsko prianjanje na zub, biološka kompatibilnost i minimalna toksičnost (15, 16). Međutim, malo je istraživanja provedeno o mikropropusnosti novih SIC-ova.

Potreba za poboljšanjem mehaničkih svojstava SIC-ova tema je mnogobrojnih istraživanja. Prije su se pokušavale dodavati metalne legure kao što su legura srebra s kositrom, zlato ili nehrđajući čelik kako bi se ojačala cementna jezgra konvencionalnog SIC-a. Radilo se o jednostavnom dodavanju metalnoga praha u prah cementa (17, 18). Druga istraživanja temeljila su se na modifikaciji SIC-a sinteriranjem srebrnih čestica i ionomernih stakala čime su dobiveni cementi (19). Williams i suradnici (20) uspoređivali su čvrstoću metalnih i nemetalnih ojačanih SIC-ova. Zaključili su da je dodavanje metala SIC-u značajno povećalo čvrstoću materijala.

Proizvođači i istraživači zainteresirani su za poboljšanje formulacije SIC-a i prevladavanje postojećih nedostataka. Novu generacija SIC-a (Zirconomer) razvila je tvrtka SHOFU iz Japana ugrađivanjem čestica cirkonijeva oksida kako bi se postigle veća tlačna i savojna čvrstoća te manje okluzalno trošenje uz bržu aplikaciju (21, 22). Loša rubna prilagodba restaurativnog materijala može rezultirati mikropropuštanjem, što potiče postoperativnu preosjetljivost, diskoloraciju i penetraciju bakterija (23, 24). Maksimalno brtvljenje na spoju ispuna i zuba bitan je čimbenik za trajnost i kvalitetu restaurativnih materijala. Stoga je svrha ovog istraživanja *in vitro* procijeniti mikropropusnost odnedavno dostupnog staklenoionomernog cementa (Zirconomer) i usporediti ga s postojećim restaurativnim materijalima.

Materijali i metode

Priprema uzoraka

Odabrano je šesnaest goveđih sjekutića bez karijesa koji su očišćeni i pohranjeni na 4 °C u 0,2-postotnoj otopini timola tijekom najviše dva tjedna. Zubi su slučajnim odabirom podijeljeni u četiri skupine (n = 4). Jedan istraživač preparirao je standardizirane kavitete na mezijalnim i distalnim plohama dimenzija 3,0 mm (duljina) x 2,0 mm (širina) x 2,0 mm dubina koristeći se dijamantnim svrdlom ISO #014 Straight Fissure (za preparaciju) i ISO #012 Inverted Cone (za završenu obradu) turbinom uz vodeno hlađenje. Rubovi preparacije nisu zakošeni. Potrebno vrijeme za obradu svakog kaviteta iznosilo je oko 10 minuta, a nakon sedme preparacije zamijenjena su svrdla. Kaviteti su ispunjeni jednim od materijala kako slijedi:

mation of marginal leakage can also be affected by other factors such as the cavity design, location and its dimensions (10, 12). Furthermore, factors such as; tooth structure, dentine permeability and orientation of dentinal tubules may contribute to microleakage formation (10).

The conventional glass ionomer cements (GICs) were developed in 1960s by Kent and Wilson and formed by an acid-base reaction (13, 14). Nowadays, GICs are the material of choice for various dental applications including; sealing for root canal treatment, bonding agent for orthodontic brackets, liner in deep cavities, fissure sealant and restorative filling for damaged tooth surfaces (15). These materials have unique properties such as; their ability to release and uptake fluoride, chemical adhesion to the tooth as well as biological compatibility and minimal toxicity (15, 16). However, few studies have been done on the microleakage of newly developed GICs.

The need to improve the mechanical properties of GICs is always a major concern. Earlier studies tried to fuse the metal alloy such as; silver-tin alloy, gold or stainless steel, in an attempt to reinforce the cement core of conventional GIC by simple addition of metal powder into glass powder (17, 18). Other research was based on modifying the GICs by sintered the silver particles and ionomer glasses to form cermet cements (19). Williams et al., (20) compared the strength of metal and non-metal reinforced GICs. They concluded that the addition of metal to GICs enhanced the strength of the material markedly.

There has been considerable interest by manufacturers and researchers to improve the formulation of GIC and also to overcome some of its drawbacks. A new generation of GICs (Zirconomer) was developed at SHOFU, Japan by incorporating particles of zirconia in order to achieve greater compressive and flexure strengths, as well to attain less occlusal wear and fast setting reaction (21, 22). The poor adaptation of restorative material can result in marginal leakage which leads to post-operative sensitivity, discoloration, and bacteria penetration (23, 24). The maximum sealing at the tooth-restoration interface is essential factor for an ideal performance of the restorative materials. Hence, this present in vitro study was intended to assess the microleakage of recently available glass ionomer (Zirconomer) and compare it with those previously existing restorative materials.

Materials and Methods

Specimen Preparation

Sixteen non-carious bovine incisors were selected, cleaned and stored at 4ºC in 0.2% thymol solution for a maximum of two weeks. The teeth were randomly divided into four groups (n = 4). One operator prepared standardized cavities on the mesial and distal surfaces with dimension of 3.0 mm length x 2.0 mm width x 2.0 mm depth using ISO #014 Straight Fissure diamond bur (for preparation) and ISO #012 Inverted Cone diamond bur (for finishing) with a high-speed handpiece under water spray. No bevels were added at the preparation margins. The required time for each cavity was about 10 min and burs were replaced after the seventh preparation. Every seven prepared cavities were restored with one material; as following:

skupina I: Zirconomer (konvencionalni SIC, SHOFU, Japan), ručno miješan na staklenoj ploči plastičnom lopaticom u specifičnom omjeru praha i tekućine od 2 : 1;

skupina II: Ketac™ Silver (konvencionalni SIC, 3M ESPE, Njemačka), ručno miješan na staklenoj ploči plastičnom lopaticom u specifičnom omjeru praha i tekućine od 1 : 1;

skupina III: FiltekTM Z500 (Composite, 3M ESPE, Njemačka);

skupina IV: Dispersalloy® (Amalgam, DENTSPLY, UK) u kapsulama.

Svi restaurativni materijali korišteni su prema preporukama proizvođača. Uzorci SIC-a restaurirani su debeloslojnom tehnikom te je uklonjen višak cementa. Površina ispuna odmah je premazana vazelinom kako bi se izbjegla apsorpcija vode i dehidracija. Za kompozitne ispune sve stijenke kaviteta jetkane su 37-postotnim gelom ortofosforne kiseline koji je nanesen malom četkicom i ostavljen da djeluje 15 sekunda, nakon čega je odmah slijedilo ispiranje i sušenje kaviteta. Adheziv (OptiBond Solo Plus, Kerr, serijska br. 3536355) nanesen je i osvijetljen na svim rubovima 20 sekunda LED lampom (3M ESPE Elipar™). Kompozitni materijal postavljen je u slojevima sve dok kavitet nije ispunjen. Svaki sloj polimeriziran je 20 sekunda. Amalgamski ispuni ručno su kondenzirani. Oštar ručni rezač upotrijebljen je za reprodukciju pravilne anatomije zuba.

Svi restaurirani zubi odmah su pohranjeni u destiliranoj vodi na 37 °C tijekom 24 sata. Nakon toga slijedilo je 500 termo ciklusa na 5 - 55 ± 2 °C, s vremenom zadržavanja od 15 sekunda i vremenom prijenosa od 2 do 3 sekunde kako bi se oponašao oralni milje.

Provjera i vizualizacija mikropropuštanja

Vrhovi korijena zapečaćeni su ljepljivim voskom nakon čega su površine zuba dvostruko premazane lakom za nokte s razmakom od 1 mm od rubova restauracije. Zubi su uronjeni u 0,5-postotno metilensko modrilo (Sigma-Aldrich, UK, P 101704491, LOT #BCBR1927V) tijekom 24 sata kako bi se omogućilo prodiranje boje u moguće praznine. Nakon uklanjanja zuba iz boja, premaz (lak za nokte) je uklonjen, a zubi su isprani vodom, osušeni na sobnoj temperaturi i ugrađeni u rastaljenu smjesu za otiskivanje kako bi se zub fiksirao. Zatim su kaviteti prerezani na dva dijela od središta s pomoću brzog dijamantnog mikrotoma uz vodeno hlađenje (Struers, M0D08). Svaki presjek fotografiran je stereomikroskopom (VWR VistaVision) pri povećanju od 2,5 puta da bi se procijenio prodor boje kroz spoj ispuna i zuba. Stupnjeve (0 – 4) penetracije boje, kako je navedeno u tablici 1., procjenjivala su dva kliničara neovisno jedan o drugome.

Group I: Zirconomer (Conventional GIC, SHOFU, Japan), hand mixed at specific powder to liquid ratio of 2:1 using glass slab and plastic spatula.

Group II: Ketac™ Silver (Conventional GIC, 3M ESPE, Germany), hand mixed at specific powder to liquid ratio of 1:1 using glass slab and plastic spatula.

Group III: Filtek™ Z500 (Composite, 3M ESPE, Germany).

Group IV: Dispersalloy® (Amalgam, DENTSPLY, UK), capsule mixed.

All restorative materials were used according to their manufacturer's recommendations. The GICs samples were restored by bulk placement and the excess cement was removed. Vaseline was used immediately for coating the restorations surface to avoid water absorption and dehydration. For composite restorations, acid etching with 37% phosphoric acid gel was applied to all cavity walls using a small brush for 15 s, followed immediately by rinsing and drying the cavities. Single bonding agent (OptiBond Solo Plus, Kerr, Batch #3536355) was applied and exposed to the light on all margins for 20 s using a LED light curing unit (3M ESPE Elipar™). Composite resin were placed and condensed incrementally until the cavities were completely filled. Each increment was light polymerised for 20 s. The amalgam restorations were hand condensed. Sharp hand carver was used to reproduce the proper tooth anatomy. Burnishing also was achieved by a small ball burnish.

All restored teeth were immediately stored in distilled water at 37°C for 24 hr. Afterwards, the specimens were subjected to 500 thermo-cycles at 5-55 ± 2°C with a dwell time of 15 s and a transfer time of 2-3 s in order to mimic the oral environment.

Microleakage Testing and Imaging

The roots apices were sealed with a sticky wax, thereafter the tooth surfaces were coated twice with a nail varnish up to 1 mm from the restoration margins. The teeth were immersed in 0.5% methylene blue dye (Sigma-Aldrich, UK; P 101704491, LOT #BCBR1927V) for 24 hr to allow dye penetration into the possible gaps. After removing teeth from dye, the coatings (nail varnish) were peeled then the teeth were washed in water, dried at room temperature and embedded in molten impression compound to fix whole tooth. Subsequently, the cavities were sectioned into two parts from the centre using a high-speed, water-cooled diamond cut-off wheel (Struers, M0D08). Each section was photographed under a stereomicroscope (VWR VistaVision) at 2.5x magnification to evaluate dye penetration along the tooth-restoration interface. The index scores (0-4) of dye penetration, as listed in Table 1, were assessed by two clinicians independently.

Tablica 1. Kriterij procjene stupnja penetracije boje duž spoja ispuna i zuba
Table 1 Score criteria for dye penetration along tooth-restoration interface

Stupanj • Scores	Spoj ispuna i zuba • Tooth-restoration interface
0	Nema prodiranja boje • No dye penetration
1	Prodiranje boje do 1/3 preparirane stijenke kaviteta • Dye penetration up to 1/3 of the prepared cavity wall
2	Prodiranje boje do 2/3 preparirane stijenke kaviteta • Dye penetration up to 2/3 of the prepared cavity wall
3	Prodiranje boje preko cijele preparirane stijenke kaviteta • Dye penetration onto the entire prepared cavity wall
4	Prodiranje boje na svim stijenkama kaviteta • Dye penetration onto the whole prepared cavity walls

Statistička analiza

Rezultati procjene stupnja penetracije boje statistički su analizirani neparametrijskim testom (Kruskal-Wallis) softverom IBM SPSS (verzija 24, SPSS Inc., Chicago, SAD). Parametrijska usporedba (Mann-Whitney U-test) provedena je na razini značajnosti $p < 0{,}05$.

Rezultati

U ovom istraživanju pri uporabi svih ispitanih materijala dogodilo se znatno prodiranje boje; slika 1. (a – d). Kruskal-Wallisov test pokazao je značajne razlike u srednjim vrijednostima mikropropusnosti između četiriju skupina ($p = 0{,}000$). Najveća srednja vrijednost mikropropuštanja utvrđena je za KetacTM Silver (3,71 ± 0,48). Srednja vrijednost penetracije boje za Zirconomer (2,86 ± 0,69) bila je slična kao srednja vrijednost za kompozitne restauracije (2,86 ± 1,06). Uzorci ispunjeni amalgamom imali su niže srednje vrijednost (0,57 ± 0,53) u usporedbi s drugim testiranim materijalima. Paralelna usporedba pokazala je statistički značajne varijacije između Zirconomera i drugih ispitivanih skupina, KetacTM Silvera i amalgama ($P < 0{,}05$), a nije bilo značajne razlike između Zirconomera i kompozitnih restauracija ($P > 0{,}05$). Razlike između svih ispitanih skupina i amalgamske skupine bile su statistički značajne ($P < 0{,}05$); tablice 2. i 3.

Rasprava

Testiranje mikropropusnosti naširoko se koristi kao glavni pokazatelj kojim kliničari i istraživači mogu predvidjeti kvalitetu vezivanja restaurativnih materijala. Klinički uspjeh uglavnom ovisi o svojstvu materijala da se čvrsto veže za površinu zuba i da se ta površina izolira od okoline te se tako spriječi pojava sekundarnog karijesa (25).

Statistical Analysis

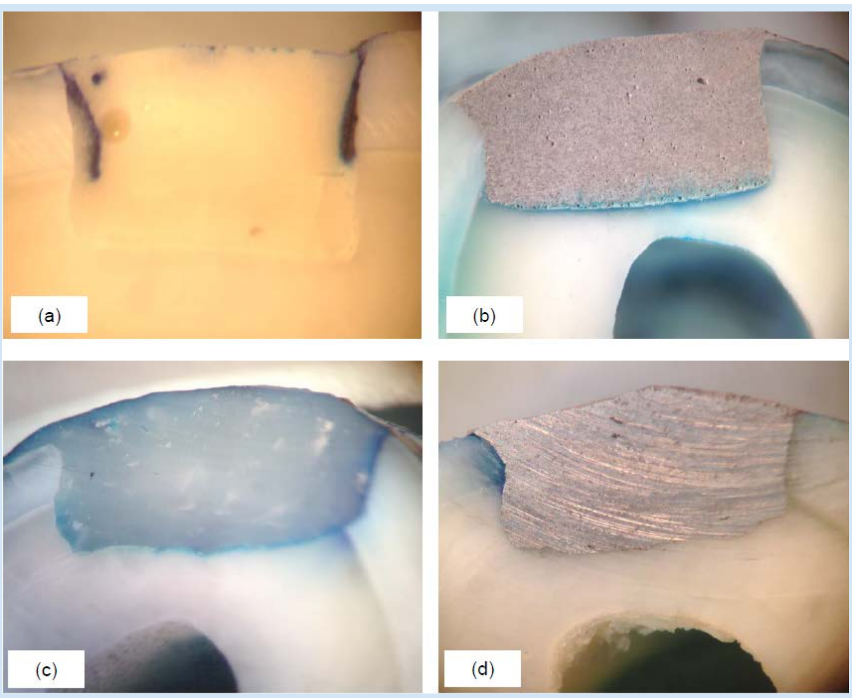
The evaluated scores of dye penetration were statistically analysed with non-parametric test (Kruskal-Wallis test) for comparison groups; using IBM SPSS software (version 24, SPSS Inc., Chicago, USA). The pairwise comparison (Mann-Whitney U Test) was performed at a significance level of ($p < 0.05$).

Results

In light of the current study, all tested materials showed a considerable amount of dye penetration; Figure 1 (a-d). The Kruskal-Wallis Test revealed significant differences in mean microleakage scores among the four groups ($p = 0.000$). The highest mean value of leakage was significantly assessed with Ketac™ Silver (3.71 ± 0.48). The mean value of dye penetration for Zirconomer (2.86 ± 0.69) was evaluated to be similar to the mean value of composite restorations (2.86 ± 1.06). It was evident that the samples filled with amalgam had significantly lower mean value (0.57 ± 0.53) compared to the other tested materials. The paired comparison showed considerable variation statistically between Zirconomer and other investigated groups of Ketac™ Silver and amalgam ($P < 0.05$), whilst there was no significant difference between Zirconomer and composite restorations ($P > 0.05$). Differences between all tested groups and amalgam group were highly significant ($P < 0.05$); Tables 2-3.

Discussion

Microleakage experiments are widely used as a major indicator by which both clinicians and researchers can predict the ideal performance of the restorative materials in terms of bonding characteristics. However, the clinical success mainly depends on the ability of the material to bond firmly to the tooth surfaces and to isolate these surfaces from the sur-



Slika 1. Mikropropusnost u (a) skupini I – Zirconomer, (b) skupini II – Ketac™ Silver, (c) skupini III – Filtek™ Z500 (kompozit) i (d) skupine IV – Dispersalloy® (amalgam)

Figure 1 Microleakage in (a) Group I-Zirconomer, (b) Group II-Ketac™ Silver, (c) Group III-Filtek™ Z500 (composite) and (d) Group IV-Dispersalloy® (amalgam)

Tablica 2. Srednje vrijednosti, standardne devijacije (SD), minimalne i maksimalne vrijednosti mikropropuštanja u različitim eksperimentalnim skupinama
Table 2 Mean, standard deviation (SD), minimum and maximum values of microleakage in different experimental groups

Skupina • Groups	Srednja vrijednost ± SD • Mean ± SD	min.-maks. • Min-Max
ZIRCONOMER	2.86 ± 0.69^{a}	2.00-4.00
KETAC™ SILVER	3.71 ± 0.48^{b}	3.00-4.00
FILTEK™ Z500	$2.86 \pm 1.06^{a,b}$	1.00-4.00
DISPERSALLOY®	0.57 ± 0.53^{c}	0.00-1.00

Različita slova u superskriptu predstavljaju statistički značajne razlike između ispitanih skupina (p < 0,05) •
Different superscript letters represent the significant differences between the tested groups (p < 0.05)

Tablica 3. Usporedba mikropropuštanja u različitim eksperimentalnim skupinama primjenom Mann-Whitneyeva U-testa
Table 3 Paired comparison of microleakage in different experimental groups using Mann-Whitney U test

Skupina • Groups	*P*-vrijednost • *P*-value
ZIRCONOMER vs KETAC™ SILVER	0.026**
ZIRCONOMER vs FILTEK™ Z500	0.836***
ZIRCONOMER vs DISPERSALLOY®	0.001*
KETAC™ SILVER vs FILTEK™ Z500	0.080***
KETAC™ SILVER vs DISPERSALLOY®	0.001*
FILTEK™ Z500 vs DISPERSALLOY®	0.002*

P-vrijednost* je vrlo statistički značajna; *P*-vrijednost** je statistički značajna; *P*-vrijednost*** nije statistički značajna •
P-value* is highly significant; *P*-value** is significant; *P*-value*** is insignificant

Velik stupanj propuštanja utvrđen je na restauracijama u skupini I – Zirconomer nakon 24 sata uranjanja u boju; kao što se vidi na slici 1. (a). Takav nalaz u skladu je s istraživanjem Patela i suradnika (22) koji su dobili slične rezultate kada su testirali penetraciju boje kroz Zirconomer na ljudskim zubima. To se može objasniti kemijskom strukturom Zirconomera koji kao punilo sadržava keramičke čestice (cirkonijev oksid). Moguće je da će to punilo uzrokovati smetnje u reakciji keliranja između karboksilne skupine (-COOH) poliakrilne kiseline i kalcijevih iona ($Ca2^{+}$) apatita zuba. Rubno propuštanje također je ustanovljeno za staklenoionomerni cement ojačan srebrom (skupina II – Ketac™ Silver); kao što je prikazano na slici 1. (b). To se također može pripisati prekidu poliakrilne matrice u cementu i nakon dodavanja cirkonij-oksidnog punila u Zirconomeru.

U nedavno provedenom istraživanju Asafarlal (26) je kvantitativno analizirao mikropropusnost triju različitih staklenoionomernih cemenata – Zirconomera, Fuji IX Extra i Ketac Molara. Nakon 24-satnog uranjanja zuba pojedinačno u 0,5-postotno metilensko uzorci su natopljeni u 2 ml 65-postotne dušične kiseline, otopine su centrifugirane, a zatim je korišten spektrofotometar za procjenu prodiranja boje. Rezultati koncentracije boje u eksperimentalnim otopinama pokazali su da je mikropropuštanje pri uporabi Zirconomera bilo veće u usporedbi s drugim SIC-ima. Smatra se da je to zbog velikih čestica punila cirkonijeva oksida, što rezultira slabom prilagodbom na spoju ispuna i zuba (26).

Nadalje, na kompozitnim ispunima (skupina III – FiltekTM Z500) u ovom istraživanju zabilježen je značajan stupanj mikropropuštanja na spoju ispuna i zuba – kao što je prikazano na slici 1. (c). Varijance u srednjim vrijednostima propuštanja statistički su analizirane između kompozitnih is-

rounding environment and hence to prevent the secondary caries occurrence (25).

A significant degree of leakage was exhibited in Group I-Zirconomer restorations after 24 hr of dye immersion; as seen in Figure 1 (a). This finding goes in a good agreement with the previous work by Patel et al., (22) who found almost similar outcomes when they tested the dye penetration of Zirconomer in human molar teeth. This could be explained due to the fact that the chemical structure of Zirconomer which comprises ceramic particles (zirconia) as fillers. It is possible that the zirconia fillers would cause interference in the chelating reaction between the carboxylic group (-COOH) of poly-acrylic acid and the calcium ions (Ca^{2+}) of tooth apatite. The marginal leakage was also observed with silver reinforced glass ionomer filling material (Group II-Ketac™ Silver); as displayed in Figure 1 (b). This can also be attributed to the disruption of polyacrylate matrix in the cement as noted with the addition of zirconia fillers in Zirconomer.

A recent study by Asafarlal (26) investigated the microleakage of three different GICs-Zirconomer, Fuji IX Extra and Ketac Molar quantitatively. After immersion the teeth individually in 0.5% methylene blue dye for 24 hr, the samples were dissolved in 2ml of 65% nitric acid, the solutions were centrifuged and then spectrophotometer was used to assess the dye penetration. The results of the dye concentration in the experimental solutions showed that the microleakage value of Zirconomer was higher compared to the other GICs. This was believed to be owing to large size of the filler particles of zirconia which leading to poor adaptation at the tooth-restoration interface (26).

In addition, the examined composite (Group III-Filtek™ Z500) restorations in this study showed a substantial degree puna i drugih skupina s konvencionalnim stakleonionomernim cementima (skupina I – Zirconomer i skupina II - KetacTM Silver), ali nisu utvrđene nikakve razlike ($p > 0,05$) – kao što je navedeno u tablicama 2. i 3. Mikropropusnost kompozitnih ispuna može se objasniti polimerizacijskim skupljanjem, što rezultira lošijim prianjanjem na površinu zuba. Zbog toga korištenje adhezivnih sustava ovdje nije pomoglo smanjiti pojavu mikropukotina na sučelju.

Dobiveni rezultati također su pokazali da je na amalgamskim ispunima (skupina IV – Dispersalloy®) zabilježeno minimalno propuštanje u usporedbi s drugim testiranim materijalima – kao što je prikazano na slici 1. (d). Statistički je postojala razlika između amalgamskih ispuna i ostalih triju testiranih materijala ($p < 0,05$) – kao što je navedeno u tablicama 2. i 3. To je zbog činjenice da amalgam ima svojstvo brtvljenja mikropukotina tijekom procesa kondenzacije i modelacije. Osim toga, ti pokusi izvedeni su u idealnom okružju bez izlaganja bilo kakvim onečišćenjima. Bez obzira na loša estetska svojstva, toksični učinak žive, postupak pripreme i ekspanziju što rezultira stvaranjem pukotina na površini zuba (27), amalgam je i dalje jedna od alternativa u stomatologiji zbog razmjerno niske cijene.

U prethodnom istraživanju u kojemu su se uspoređivala svojstva brtvljenja amalgama i kompozitnih materijala zaključeno je da je amalgamski ispun I. razreda imao manju mikropropusnost od kompozitnoga (28). U drugom istraživanju dokumentirano je da oblaganje stijenki lakom nije spriječilo rubno propuštanje na amalgamskim ispunima (29). Uz smanjenje popularnosti amalgama u posljednjih nekoliko godina i nedostatke materijala na bazi smola, postoji potreba za restaurativnim materijalom koji stvara jaku vezu i jednostavan je za primjenu. Zbog toga je Zirconomer (bijeli amalgam) novi razred staklenoionomernih cemenata i savršen izbor zbog svojih prednosti i svojstava da se koristi za stalne ispune na stražnjim zubima kod pacijenata s visokom incidencijom karijesa (30).

Važno je istaknuti da konvencionalni SIC-ovi imaju jedinstveno svojstvo formiranja jake kemijske veze s prirodnim zubnim tkivom (31), pa nije potrebno korištenje vezivnih sredstava ili kiselina za jetkanje jer poliakrilna kiselina (tekuća komponenta SIC-a) može najetkati površinu zuba i tako pospješiti vezu. Vjeruje se da se SIC veže kemijski stvaranjem ionske veze između karboksilnih skupina (COO-) cementa i kalcija (Ca2 +) iz zuba (32, 33).

SIC ima svojstvo remineralizacije i zaštite zuba jer otpušta ione kao što su fluor, kalcij i fosfat. Otpuštanje iona iz ispuna također se može promovirati u mediju koji sadržava fluor (34). SIC tako djeluje kao spremnij fluora kako bi se postigao dugoročni protukarijesni učinak u usporedbi s kompozitnim materijalima. Van Duinen i suradnici (35) i Van Duinen (36) utvrdili su da se spojevi slični apatitu stvaraju na površini materijala temeljenih na SIC-u. Zaključili su da se SIC može smatrati savršenim izborom u restaurativnoj stomatologiji - osobito za ART tehniku (Atraumatic Restorative Treatment) zbog sposobnosti remineralizacije. To svojstvo (remineralizacija) razlikuje ove pametne materijale (SIC-ove) od drugih materijala za ispune kao što su kompoziti i amalgami zbog njihova jedinstvenog sastava. To bi moglo biti ko-

of microleakage along tooth-restoration interface; as revealed in Figure 1 (c). The variances in the mean values of leakage were assessed statistically between composite fillings and other groups of conventional glass ionomer cements (Group I-Zirconomer & Group II-Ketac™ Silver), which did not show any differences ($p > 0.05$); as listed in Tables 2-3. Microleakage in composite restorations can be explained on the bases of polymerisation shrinkage which leads to a poor bonding ability to tooth surfaces. Consequently, using bonding system here did not aid in decreasing incidence of gaps at the interfaces.

The obtained results further showed that the minimum leakage scores were seen with amalgam (Group IV-Dispersalloy®) restorations compared to other tested materials; as illustrated in Figure 1 (d). Statistically, there were also dissimilarities between the amalgam restorations and other three tested dental materials ($p < 0.05$); as given in Tables 2-3. This is due to the fact that amalgam has ability to seal the micro-interfaces during the condensation and carving process. Besides of that these experiments were done in an ideal environment with no exposure to any contaminations. Regardless of poor aesthetic characteristics, toxic effect of mercury, its method of preparation and expansion process resulting in creating cracks in the tooth surfaces (27), amalgam is still one of the options in dental practices due to its relatively low costs.

A previous study comparing the sealing properties of amalgam to resin composite restorations concluded that Class-I amalgam restoration had lower microleakage than the composite (28). A different study documented that coating the walls with cavity varnish did not decline the marginal leakage in amalgam restorations (29). With decreasing in popularity of amalgam in recent years and drawbacks of resin-based materials, there is a special need for a strong bonding and easily handling restorative material. Therefore, Zirconomer (white amalgam) is a new class of glass ionomer restorative material and the perfect choice due to its advantages and capacity to be used for permanent posterior restorations in patients with high caries incidence (30).

It is important to highlight that the conventional GICs have unique ability to form a strong chemical bond naturally to the tooth surfaces (31) without the need of bonding agents or etching solutions as the poly-acrylic acid (liquid component of GIC) can etch the tooth surfaces thus the interlocking bonds would be improved. It is believed that GICs bond chemically via formation of an ionic bond between carboxyl groups (COO-) of the cements and calcium ions (Ca^{2+}) of the tooth surfaces (32, 33).

GICs have the ability to remineralise and protect the tooth through therapeutic ion release such as fluoride, calcium and phosphate. Ions release from the restoration that also could be promoted in fluoride-containing media (34). The GICs therefore act as reservoir of fluoride to obtain a long-term anti-cariogenic action compared to the composite resins. Van Duinen et al., (35) and Van Duinen (36) found that apatite-like species formed on the surface of GIC-based materials. They concluded that the GICs have to be considered as the perfect choice in the restorative dentistry; particularly for Atraumatic Restorative Treatment (ART) technique due risno za brtvljenje praznina uzrokovanih mikropropuštanjem između SIC-a i zuba.

Jedno od ograničenja SIC-a jest osjetljivost tek postavljenog ispuna na vlažnost i to je razlog zašto ga odmah treba zaštititi i obložiti lakom. SIC je također sklon apsorpciji boje zbog svoje hidrofilne prirode, što može dati lažno pozitivne rezultate. Tijekom pripreme uzoraka za ispitivanje mikropropusnosti u restauracijama je uočena dehidracija, što bi moglo rezultirati povećanom apsorpcijom boje.

Dobivanje zdravih izvađenih ljudskih zuba sve je teže zbog popularnosti minimalno invazivnih restaurativnih postupaka. U skladu s tim kao alternativa obično se koriste goveđi zubi zbog dostupnosti, veličine i ravnih površina (37). Dosadašnja istraživanja o mikropropusnosti pokazala su da nema značajnih razlika u rezultatima dobivenima na ljudskim i goveđim zubima (38, 39), što goveđe zube čini prikladnima za istraživanja *in vitro*.

Zaključak

Uzimajući u obzir ograničenja ovog istraživanja može se zaključiti: I. na svim testiranim restaurativnim materijalima pojavilo se mikropropuštanje; II. na kaviteima ispunjenima materijalom Ketac™ Silver zabilježena je najveća mikropropusnost, a slijede Zirconomer i kompozit; III. utvrđeno je da je noviji materijal (Zirconomer) pokazao statistički značajne razlike u odnosu na Ketac™ Silver i amalgam, ali ne i na kompozit; IV. amalgam je imao najmanju mikropropusnost te su postojale vrlo značajne razlike između amalgama i drugih testiranih restaurativnih materijala.

Potrebna su dodatna istraživanja kako bi se postigla maksimalna dugoročna kvaliteta vezivanja novih staklenoionomernih cemenata.

Sukob interesa

Autori nisu naveli sukob interesa.

to their capacity to remineralise. This property (remineralisation) distinguishes these smart materials (GICs) from the other filling material such as composite and amalgam restorations due to the unique composition of GICs. This could be beneficial in sealing any gaps produced due to microleakge between GIC and the tooth.

One of the limitations of GICs is the sensitivity of freshly set cement to moisture and this is the reason why it should be protected immediately and coated with varnish. GICs also have the tendency to absorb the dye due to their hydrophilic nature and this could give false-positive results. During specimen's preparation for microleakage investigation, some dehydration was noticed in the restorations which could result in increased dye absorption.

Obtaining sound extracted human teeth are becoming quite difficult due to popularity of minimally invasive restorative treatment. Accordingly, bovine teeth have been commonly used as an alternative for human teeth due to their availabilities, larger sizes and flat surfaces (37). Previous microleakage studies have demonstrated that there is no significant difference in the results obtained in human and bovine teeth (38, 39) which making bovine teeth suitable for in vitro studies.

Conclusions

Within the limitation of this study, it can be concluded that: I.None of the tested restorative materials was free from the microleakage; II. Cavities filled with Ketac™ Silver exhibited the highest microleakage scores followed by Zirconomer and composite; III. It found that the newer material (Zirconomer) had significant differences verses Ketac™ Silver and amalgam, but not with composite; IV. Amalgam showed the least microleakage scores and also there were highly significant variations between amalgam and other tested restorative materials.

Further experiments are required to be done in order to achieve the maximum enhancement of bonding characteristics in newly available GICs in the long-term.

Conflict of interest

None declared

Abstract
Objective: To evaluate the microleakage of four direct restorative materials. **Materials and Methods:** Sixteen sound bovine incisors were chosen and randomly divided into four groups; Group I-Zirconomer, Group II-Ketac™ Silver, Group III-Filtek™ Z500 (composite) and Group IV-Dispersalloy® (amalgam). Seven proximal (mesial & distal) cavities, for each material were prepared and restored. All restored samples were stored in 37°C distilled water for 24 hr and then subjected to thermo-cycling process at temperatures between 5-55°C. The samples were immersed in dye solution of 0.5% methylene blue for 24 hr. Each filled cavity was sectioned through the centre of restoration and then studied under a stereomicroscope to assess the marginal leakage. The obtained microleakage scores were statistically analysed. **Results:** The highest mean score of leakage was recorded in Group II-Ketac™ Silver followed by Group I-Zirconomer and Group III-Filtek™ Z500 (composite). The lowest mean score of dye penetration was verified in Group IV-Dispersalloy® (amalgam). Statistically, there were significant differences between Zirconomer and other groups of Ketac™ Silver and amalgam, whereas the Zirconomer groups had no significant differences with composites. All tested groups showed significant differences with amalgam restorations. **Conclusions:** The marginal leakage was evident in all restorative materials. Further studies with clinical trial have to be done.

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Microleakage; Glass Ionomer Cements; Zirconomer

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